Excess Volume, Viscosity, Heat of Mixing and IR Studies of Ternary Liquid Mixtures involving H-Bond

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Abstract - Densities and viscosities of ternary liquid mixtures of isopropanol +1,2-propanediol + cyclohexanone, were measured at 308.15 K and 318.15 K over entire range of composition. From the density (ρ) and viscosity (η) data, the excess thermodynamic properties, viz., the excess volume (V*), molar volume (V), deviation in viscosity (Δη), interaction parameter (d) and excess Gibbs free energy of activation of viscous flow (ΔG*η) were calculated. The values of excess parameters namely V*, Δη and ΔG*η were fitted to Redlich Kister type polynomial equation. Further heat of mixing and IR spectra were studied at equimolar concentration. From these results, the nature of interaction have been discussed in terms of intermolecular interaction between the mixing components.

Keywords – Excess volume, Deviation in viscosity, Excess Gibbs free energy of activation of viscous flow. Heat of mixing and IR-Study.

I. INTRODUCTION

Physico-chemical and thermodynamic investigations play an important role in helping to understand the nature and extent of patterns of molecular aggregation that exists in binary and ternary liquid mixtures and their sensitivities to variation in composition and molecular structure of pure components [1]. A full understanding of thermodynamic and transport properties of binary and ternary liquid mixtures is essential in many chemical engineering processes such as separation process, design calculation, heat transfer, mass transfer, fluid flow and so forth [2].

Isopropanol is self-associated liquid molecule through hydrogen bonding and it is used as solvent for coating in pharmaceutical applications. 1,2-propanediol is also self-associated through intermolecular and intramolecular hydrogen bonding. It is used as medical lubricant, moisturizer in medicines, tobacco products and cosmetics. cyclohexanone exhibit dipole-dipole interaction in pure state and its derivatives are used for the synthesis of pharmaceuticals, dyes, herbicides, pesticides, plasticizers and rubber chemicals. Keeping in view of the importance, the liquids, measurement of density and viscosity of isopropanol (1) + 1,2-propanediol (2) + cyclohexanone (3) ternary liquid mixtures were reported over the entire range of composition at 308.15K and 318.15K. A survey of literatures has shown that no experimental physico-chemical studies for the above ternary liquid mixtures were reported earlier.

II. EXPERIMENTAL METHODS

The chemicals used in the present study are analytical grade (Merck) and further purified by standard methods [3, 4]. Before use, the chemicals were dried by suitable drying agents to remove the water content, if any. The mixtures were prepared by knowing mass and were kept in special air tight glass stoppered conical type bottles to avoid evaporation. The weighing measurements were performed on a Shimadzu Auy 220 Japan electronic digital balance with precision of ± 0.0001 g.

a. Density and Viscosity measurements

The density of pure liquids and their liquid mixtures were measured by using special type specific gravity bottle of 10 ml capacity with an accuracy ± 0.0001 g. The specific gravity bottle containing solution was immersed in a constant temperature water bath (Guna company) measured at 308.15 and 318.15 K. Each reported values are the average of at least three measurements.

The viscosity of pure liquids and their liquid mixtures were measured by using in ostwald’s viscometer of 10 ml capacity. The viscosities at temperature 308.15 and 318.15 K were measured. The time given to attain thermal equilibrium for the content of viscometer was 15 min. The time of flow was measured with an accurate stop watch which is capable of measuring time to within 0.01 s. Three sets of reading for the flow time were taken and the average values were taken for each pure liquids and liquid mixtures.

b. Heat of Mixing

Heat of mixing of ternary liquid mixtures were measured with calorimeter (Dewar flask) at equimolar concentration.

c. IR study

IR spectra for pure liquids and equimolar liquid mixtures were recorded by Perkin Elmer FT-IR.

III. RESULTS AND DISCUSSION

Comparison of experimental density (ρ) and viscosity (η) values with literature values for pure isopropanol, 1,2-propanediol and cyclohexanone, are presented in Table-1.
There is a good agreement with literature values at 308.15 and 318.15 K temperature for both density ($\rho$) and viscosity ($\eta$) data.

### Table 3.1 Comparison of density ($\rho$) and viscosity ($\eta$) values with literature values of pure liquids at 308.15 K and 318.15 K

<table>
<thead>
<tr>
<th>Name of the Components</th>
<th>Temperature K</th>
<th>Density $\rho$/g cm$^{-3}$</th>
<th>Viscosity $\eta$/m Pa s</th>
</tr>
</thead>
<tbody>
<tr>
<td>Isopropanol</td>
<td>308.15</td>
<td>0.7720</td>
<td>1.1282</td>
</tr>
<tr>
<td></td>
<td>318.15</td>
<td>0.7668</td>
<td>1.1890</td>
</tr>
<tr>
<td>1,2-propanediol</td>
<td>308.15</td>
<td>1.0256</td>
<td>21.3250</td>
</tr>
<tr>
<td></td>
<td>318.15</td>
<td>1.0199</td>
<td>25.2200</td>
</tr>
<tr>
<td>Cyclohexane</td>
<td>308.15</td>
<td>0.9290</td>
<td>1.3140</td>
</tr>
<tr>
<td></td>
<td>318.15</td>
<td>0.9237</td>
<td>1.6562</td>
</tr>
</tbody>
</table>

#### a. Calculation of Excess Thermodynamic Parameters

1. **Excess volume ($V^\Delta$)** [17]

\[
V^\Delta = \sum_{i} \frac{x_i M_i}{\rho_i} - \frac{x_i M_i}{\rho_{mix}}
\]

where $x_i$, $x_2$ and $x_3$ are mole fraction of component 1, 2 and 3, $\rho_i$, $\rho_2$ and $\rho_{mix}$ are densities of pure liquids and mixtures, $M_i$, $M_2$ and $M_{mix}$ are molecular weight of components 1, 2 and 3 respectively.

2. **Molar volume ($V$)** [18]

\[
V = \sum_{i} \frac{x_i M_i}{\rho}
\]

$V$ refers to molar volume that can be calculated from the density of mixtures, Here $\rho$ is density of mixtures.

3. **Viscosity ($\eta$)** [19]

\[
\eta = (\Delta T - \frac{B}{t}) \rho
\]

$A$ and $B$ are characteristic constants of viscometer calculated using standard liquids water and nitrobenzene, $t$ is time of flow.

4. **Deviation in viscosity ($\Delta\eta$)** [20]

\[
\Delta\eta = \eta_{mix} - (x_1 \eta_1 + x_2 \eta_2 + x_3 \eta_3)
\]

5. **Excess molar volume ($V^\Delta$)** [17]

\[
\Delta V = \Delta V_{mix} - (\Delta V_1 + \Delta V_2 + \Delta V_3)
\]

6. **Interaction parameter ($d$)** [21]

\[
d = \frac{\ln \eta_{mix} - (x_1 \ln \eta_1 + x_2 \ln \eta_2 + x_3 \ln \eta_3)}{x_1 x_2 x_3}
\]

7. **Excess Gibbs free energy of activation of viscous flow ($\Delta G^E$)** [21]

\[
\Delta G^E = RT \ln(\eta_{mix}) - (x_1 \ln(\eta_{1}) + x_2 \ln(\eta_{2}) + x_3 \ln(\eta_{3}))
\]

8. **Heat of mixing ($\Delta H^m$)** [22]

\[
q = m s \Delta T + w \Delta T
\]

where $m$ is mass of liquid solution, $s$ is specific heat of solution, $w$ is water equivalent calorimeter, $q$ is the heat loss or gained, $\Delta T$ is the change in Temperature.

Excess values of other parameter are calculated using the relation

\[
A^E_{mix} = A_{mix} - A_{id}
\]

\[
A_{mix} = \sum_{i} x_i A_i, x_i and A_i are mole fraction and parameters of i$th$ component liquid.

The calculated values of $V^E$, $\Delta\eta$ and $\Delta G^E$ were fitted to Redlich-Kister type polynomial equation

\[
A^E_{mix} = x_1 x_2 x_3 [(a + b(x_1 - x_2) + cx_1 x_2(x_1 - x_2)^2 ]...
\]

The method of least squares was used to derive the adjustable parameters $a$, $b$ and $c$. The standard deviation [25] $\sigma$ values were calculated using the relation.

\[
\sigma = \sqrt{\frac{(A^E_{mix} - A^E_{cal})^2}{(n-m)}}
\]

where $n$ is number of experimental data, ‘m’ is number of adjustable parameters.

The experimental values of the density ($\rho$) and viscosity ($\eta$) in case the ternary liquid mixtures under study over the entire mole fraction range at 308.15K and 318.15K are given in Table 3.2 and 3.3. From these available values of density and viscosity, the values of excess volume ($V^E$), molar volume ($V$), deviation in viscosity ($\Delta\eta$), interaction parameter ($d$) and excess Gibbs free energy of activation of viscous flow ($\Delta G^E$) were calculated. The excess volumes were plotted against with molefractions at 308.15K and 318.15K over the entire composition range. The excess parameters namely, $V^E$, $\Delta\eta$ and $\Delta G^E$ are fitted to the Redlich Kister type polynomial equation and the adjustable parameters $a$, $b$ and $c$ evaluated by the method of least squares, along with standard deviation ($\sigma$) are presented in Table 3.4.

The deviations observed in the excess parameters indicate the strength of interactions present between the unlike molecules of the liquid mixtures under study [23]. The variation in these excess parameters may be result of contributions from several effects such as (i) breaking up of hydrogen bond. (ii) The specific forces that exist between the molecules like dipole-dipole interaction and hydrogen bonding interaction. (iii) Geometrical fitting of one molecule into other molecule because of difference in their molar volumes.

The densities and viscosities of given ternary liquid mixtures containing namely, isopropanol+1,2-propanediol+ cyclohexane at 308.15K and 318.15K over entire composition range are presented in Table 2 and 3. It can be seen that variation of density and viscosity with composition of the mixture is non-linear which indicates the presence of molecular interactions.
Table 3.2 Densities, Viscosities and Excess Properties of Ternary Liquid Mixtures of Isopropanol (1) + 1,2-Propanediol (2) + Cyclohexane (3) at 308.15 K

<table>
<thead>
<tr>
<th>x₁</th>
<th>x₂</th>
<th>ρ  (g·cm⁻³)</th>
<th>V°  (cm³·mol⁻¹)</th>
<th>η   (mPa·s)</th>
<th>Δη  (mPa·s)</th>
<th>Δη/E *  (mPa·s)</th>
<th>d</th>
<th>V  (cm³·mol⁻¹)</th>
<th>ΔG°  (kJ·mol⁻¹)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.1150</td>
<td>0.2134</td>
<td>0.9251</td>
<td>-0.0138</td>
<td>1.9532</td>
<td>-1.5886</td>
<td>0.0913</td>
<td>-34.4112</td>
<td>96.1759</td>
<td>0.2726</td>
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<td>0.2079</td>
<td>0.2980</td>
<td>0.9197</td>
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<tr>
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<td>0.3568</td>
<td>-65.2045</td>
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<td>0.9567</td>
</tr>
</tbody>
</table>
The excess volume data for the ternary liquid mixtures of isopropanol + 1,2-propanediol + cyclohexanone are reported in Table 3.2 and 3.3 over the entire concentration range at 308.15K and 318.15K. It is found to be positive at 308.15K. The sign of excess volume of a system depends on the relative magnitude of expansion or contraction on mixing of two or more liquids. The positive contribution to $V^E$ cause to expansion in volume by breaking up of H-bond. This may due to (a) loss of dipolar association in isopropanol molecule which is relatively cleavable in H-bond. (b) steric hindrance of isopropanol molecules a secondary alcohol. The variation of excess volume with respect to molefractions $(x_1, x_2$ and $x_3)$ are graphically represented by 3D contour diagram in figure 3.1. This will indicate that the weaker forces are dominating over the attractive forces.

Krishna Rao et al., [24] were observed negative excess volume for the binary mixture of isopropanol + 1,2-propanediol at 308.15K and positive excess volume value was obtained for the binary mixtures of isopropanol + cyclohexanone at 308.15K investigated by Mohammed alsmasi et al., [25].

In the present study, the constituent binary mixtures of isopropanol + 1,2-propanediol are negative magnitude of $V^E$ values and positive magnitude of $V^E$ values were observed in for isopropanol + cyclohexanone at 308.15K. These are good agreement.

When temperature is increased from 308.15K to 318.15K. The positive $V^E$ values changes to negative $V^E$ values. These are clearly represented in Fig 3.2. This may due to increase in the rate of association of unlike molecules and enhance in the dipolar association between the unlike molecules. Hence the interaction increase with increasing temperature. Therefore the given ternary liquid mixture are more compact than that of corresponding pure liquids at 318.15K.

Cyclohexanone is polar molecules, since the oxygen atom with its non-bonding electron pairs makes the cyclohexanone molecules H-bond acceptor. When isopropanol is mixed with 1,2-propanediol and cyclohexanone, the strength of interaction between the participating molecule depends on the dipolemoment, polarisability and geometry of the interacting molecules. The dipolemoment values fallow isopropanol $\mu = 1.66D; 1,2$-propanediol $\mu = 2.27D$ and cyclohexanone $\mu = 3.01D$.

The contraction in volume when increasing temperature to 318K results the negative $V^E$ values in the mixtures investigated. This fact is clearly evident from the values of molar volume for the molecules in study. The molar volumes of isopropanol, 1,2-propanediol and cyclohexanone at 318.15K are 78.3777 cm$^3$, mol$^{-1}$, 74.6151 cm$^3$.mol$^{-1}$ and 106.0950 cm$^3$.mol$^{-1}$ respectively. The above differences in their molar volumes of pure liquids reveal the geometrically more favorable fitting of one molecule into another molecule at higher temperature. From the $V^E$ values and the above observation conclude the presence of a hetero association like weak dipole – dipole interaction and formation of hydrogen bond between the unlike molecules.

The deviation in viscosity ($\Delta\eta$), $\Delta\ln\eta$ and interaction parameter ($d$) are recorded in Table 2 and 3 over all through whole concentration range at 308.15K and 318.15K. The values of $\Delta\eta$, $d$ are negative and $\Delta\ln\eta$ values are negative and positive at 308.15K. As temperature is increase to

<table>
<thead>
<tr>
<th>Temperature</th>
<th>Parameters</th>
<th>Isopropanol + 1,2-Propanediol + Cyclohexanone</th>
</tr>
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<tbody>
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<td></td>
<td>$V^E$(cm$^3$.mol$^{-1}$)</td>
<td>$\Delta\eta$(mPa.s)</td>
</tr>
<tr>
<td>308.15K</td>
<td>-167.1370</td>
<td>-0.0001</td>
</tr>
<tr>
<td>318.15K</td>
<td>-77.2645</td>
<td>-0.0134</td>
</tr>
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</table>
318.15K. The negative values $\Delta \eta$ and $d$ become less negative and $\Delta n\eta$ values become completely positive. The viscosity of a mixture depends on the molecular interactions between the components: mixtures with strong interactions between different molecules show positive deviations; while for mixtures without strong specific interactions, viscosity deviations are negative [26]. In the present study, the negative deviation in viscosity ($\Delta \eta$) values of this system will imply that the mixture is less viscous and can flow more easily than that of corresponding pure liquids.

The values of Gibbs free energy of activation of viscous flow ($\Delta G^E$) are listed in Table 2 and 3 over entire range of composition at 308.15K and 318.15K. It is found to be positive and negative values at 308.15K. Positive values of $\Delta G^E$ are due to presence of strong specific interaction between the component molecule, whereas their negative values suggest the existence of weaker intermolecular interaction [27]. When temperature is increased to 318.15K the negative values of $\Delta G^E$ changes to positive and the positive values of $\Delta G^E$ become more positive. This will indicate the presence of specific interaction between the unlike molecules at 318.15K.

b. Heat of mixing ($\Delta H$)

The heat of mixing ($\Delta H$) was measured by using dewar flask as calorimeter at equimolar concentration. Positive $\Delta H$ value was observed for the ternary mixtures of isopropanol + 1,2-propanediol + cyclohexanone. These positive value suggest that the dominant effect in the mixing process is the presumably endothermic dissociation of hydrogen bonds in isopropanol molecule [28]. The heat of mixing values is 1114.73 J. mol$^{-1}$. This less positive $\Delta H$ value reveals the presence of weaker intermolecular interaction between the unlike molecules of the component in the liquid mixture. This will also support the presence of weaker intermolecular hydrogen bonding in the isopropanol + 1,2-propanediol + cyclohexanone liquid mixtures.

c. FT-IR Spectra

FT-IR spectra for pure monomeric isopropanol, 1,2-propanediol and cyclohexanone along with ternary liquid mixture at equimolar concentration are recorded. The pure isopropanol molecule exhibit $-\text{OH}$ peak at 3315.26 cm$^{-1}$ and 1,2-propanediol exhibit $-\text{OH}$ peak at 3339.85 cm$^{-1}$, no $-\text{OH}$ peak can be observed for cyclohexanone. When isopropanol is mixed with 1,2-propanediol and cyclohexanone mixture, the absorption is shifted to longer wavenumber caused by weaker interaction like hydrogen bonding between $-\text{OH}$ group of isopropanol with $-\text{OH}$ group of 1,2-propanediol and C = O group of cyclohexanone. The mixture peak is observed at 3348.59 cm$^{-1}$ and the spectrum is broad. These are clearly seen in Fig. 3.4-3.7. Intermolecular hydrogen bonding involves association of two or more molecules of same or different compounds. The bonds that results from intermolecular hydrogen bonding appear at lower energies [29]. This supports the conclusion drawn from $V^E$, $\Delta \eta$ and $\Delta G^E$ data.

IV CONCLUSION

The densities and viscosities for ternary liquid mixtures of isopropanol + 1,2-propanediol + cyclohexanone are determined experimentally, at 308.15 and 318.15 K over whole composition range. The value of $V^E$, $\Delta \eta$ and $\Delta G^E$ are calculated from experimental results at both temperatures. The excess deviation function are fitted to Redlich-Kister type
polynomial equation and corresponding standard deviations ($\sigma$) are calculated. The observed positive and negative values of $V^E$, $\Delta\eta$ and $\Delta G^E$ for the given analysed ternary mixtures having the following conclusions may be drawn.

1. The positive and negative magnitude of $V^E$ values suggest that weaker intermolecular hydrogen bonding and dipole-dipole interaction between the molecules. This kind of interaction increasing with increasing temperature.
2. The negative $\Delta\eta$ values predict the given mixtures is less viscous and greater fluidity than that of corresponding pure liquids.
3. $\Delta G^E$ values reveal that there would be weaker specific interaction between the component molecules.
4. The less positive values of $\Delta H$ indicate weaker interaction among the unlike molecules.
5. FT-IR spectra reveals the formation of weaker intermolecular hydrogen bonding between the component liquids.

ACKNOWLEDGEMENTS

The authors express, sincere gratitude to management of St. Joseph’s College (Autonomous), Tiruchirappalli for providing necessary lab facilities.

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