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Refractive Index and Excess Volume for Diisopropylamine + Isomeric Butanol Mixtures in terms of Nakata and Sakurai model

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Abstract – Alkyl amines are widely used in various industries. Nowadays these are also used in CO_2 capture technology because amines react with CO_2 and remove it from the flue gas. To make the amines more compatible towards this technology, physico chemical properties may be altered by mixing with other solvents. In the present report, we measured the refractive properties of pure diisopropylamine (DIPA) (1) + isomeric butanol (2) at 298.15 K to 308.15 K. The Δn values were positive for DIPA + n-butanol or sec-butanol or isobutanol or tert-butanol mixtures. The measured data was correlated with Redlich-Kister equation. The excess molar volume data were predicted from refractive index data using Nakata and Sakurai model. The experimental data were also predicted by various correlations, and the prediction capability of these correlations was reported through standard deviation. Further, the deviation in refractive index (Δn) data was interpreted by the consideration of specific molecular interactions between DIPA and isomeric butanol.

Key words: Isomeric butanol, Diisopropylamine, Refractive index, Molecular interactions, Mixing rules, Excess molar volume

1. Introduction

Thermophysical properties are of great importance in the design of engineering equipment. Among various properties, the most important and frequently applied properties are density, viscosity, enthalpy, speed of sound and refractive index [1-6]. For the complete understanding of molecular interactions between the components, the thermophysical properties of pure or binary or multicomponent mixtures are to be determined which illustrate the deviation from ideality [7-12]. Thermophysical properties are highly influenced by the size, shape and polarity of the molecules [13].

Alkanolamine aqueous solution has more advantages than amines as novel solvent due to higher loading capacity, faster reaction rate and low energy need for the process of regeneration. The mixing of alkanol to diisopropylamine (DIPA) leads to increase in solubility of alkanolamine due its H-bonding capability with alkanols, which results in association of the components. Thus DIPA + alkanol binary mixtures can be considered as a good mixture for removal of acid gas in plants [14-23]. Above discussion indicates that determination of thermophysical of liquid mixtures applicable in various industries processes is an area of keen interest for the researchers.

The refractive indexes of pure liquids and mixtures have been reported for a long time to understand the optical properties of the materials [24-27]. This property gives valuable information about the intermolecular interactions between the components of binary liquid mixtures. The refractive index is influenced by density and temperature of the medium. Therefore, determinations of the refractive index at different temperatures are required for the complete understanding of the properties and molecular interactions [26,28]. Accurate estimation of the refractive index can be correlated to the concentration, temperature and pressure of the liquid mixtures. Among various solvents (water, ether, alcohols, amines, etc.) used to alter the thermo-physical properties, alcohols are widely used. Alcohols are associated molecules which are associated with the presence of polar functional groups. The excess properties are highly influenced by the variability of carbon chain, geometry of the molecule and position of the functional group. In branching alcohols, the geometric area is highly occupied by the hydrophobic organic part, which affects the properties in liquid mixtures when they are added. Further, alkyl amines and alkanolamines are the components which are used in various industries like post-combustion carbon capture, pharmaceuticals, and fertilizers [29-32]. For a long time, alcohols or amines were used in this process to alter these properties. Amines are widely applied in the production of agrochemicals used for crop protection. Alkanols are polar molecules and used in pharmaceutical industries in various synthesis processes. Therefore, determination of various thermophysical properties for binary mixtures containing amines and alkanols is useful for various industries.

In this view, it is interesting to study the physical properties of binary liquid mixtures containing alkyl amines and alcohols. In the continuation of our research work on the determination of physical properties of liquid mixtures, the present investigation reports the refractive indices data of DIPA + isomeric butanol mixtures at 298.15 K to 308.15 K and fitted to Redlich-Kister polynomial equation. Further various mixing rules were applied to predict the experimental

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data. The deviation in refractive index (Δn) was used to analyze to interpret the molecular interaction between DIPA and isomeric butanol molecules. The excess molar volumes were predicted from the measured n_D data of pure components and their binary mixtures by Nakata and Sakurai model [33,34].

2. Experimental

DIPA from Merck and butanol isomers (n-butanol, sec-butanol, isobutanol, tert-butanol) from CDH were purchased having purity >99%. All chemicals were stored over molecular sieve (4A) for more than one week before analysis. Purity of the samples was checked by measurement of their density and refractive index. The density was measured by vibrating tube densimeter (Anton Paar-5000) with a precision of 0.05 kg m^{-3} at T = (298.15 to 318.15) K and p = 0.1 MPa. The n_D data were measured at similar condition with a refractometer (Abbemat-200) with a precision of 0.0001and having temperature controlled within ±0.01 K with accuracy up to ±1×10^{-4}. The binary mixtures were made using a balance of precision ±0.1 mg (OHAUS, AR224CN).

The experimental uncertainty in the measured density (ρ), refractive index (n_D) and mole fraction (x) was 10^{-3} kg m^{-3}, 0.0002 and 0.0001, respectively. The uncertainty in the measurement of these properties, $u(\bar{X})$, was calculated by the following equation [23]:

$$u(\bar{X}) = \left[\frac{\sum_{i=1}^{N} \left(X_i - \bar{X} \right)^2}{N\left(N-1 \right)} \right]^{1/2}$$

Table 1. Measured densities (ρ) and refractive indices (n_D) of the pure compounds

Compound	ρ/g cm^{-3}		n_D	
	Experimental	Literature	Experimental	Literature
diisopropylamine				
298.15 K	0.712174	0.7148 [35]	1.3910	1.3921 [35]
		0.7100 [35]		
308.15 K	0.702393	0.70518 [35]	1.3853	1.3857 [35]
318.15 K	0.692496	0.69559 [35]	1.3794	1.3799 [35]
n-Butanol				
298.15 K	0.805918	0.805907 [36]	1.3970	1.39728 [37]
		0.80589 [36]		
		0.805806 [38]		
308.15 K	0.798201	0.79829 [39]	1.3929	1.39324 [37]
		0.79825 [40]		
		0.798101[38]		
318.15 K	0.790333	0.790212[37]	1.3887	1.38911[37]
		0.79018 [41]		
sec-Butanol		0.790248 [38]		
298.15 K	0.802750	0.802847 [42]	1.3949	1.3952 [43]
		0.80242 [44]		1.395 [45]
		0.802938 [38]		
308.15 K	0.794245	0.794363 [42]	1.3903	
		0.79421 [46]		
		0.794435 [38]		
318.15 K	0.785348	0.78521[39]	1.3856	
		0.785546 [38]		
isobutanol				
298.15 K	0.798146	0.7982 [47]	1.3928	1.3939 [48]
		0.7982 [49]		
308.15 K	0.790296	0.790629 [37]	1.3886	
		0.79026 [35]		
318.15 K	0.782227	0.781982 [37]	1.3844	
tert-butanol				
298.15 K	0.780511	0.780720 [37]	1.3846	1.38488 [37]
		0.78068 [50]		
308.15 K	0.770347	0.770212 [37]	1.3795	1.37985 [37]
		0.77024 [49]		1.37947 [51]
		0.770296 [38]		
318.15 K	0.760157	0.759502 [37]	1.3742	1.37402 [37]
		0.75987 [52]		1.37384 [51]

Table 2. Refractive index (n_D) and deviation in refractive index (Δn)

x_1	n_D	Δn	n_D	Δn	n_D	Δn
	298.15 K		308.15 K		318.15 K	
DIPA (1) + n-butanol (2)						
0	1.3970	0.0000	1.3929	0.0000	1.3887	0.0000
0.0712	1.3997	0.0031	1.3953	0.0029	1.3910	0.0029
0.1377	1.4016	0.0054	1.3969	0.0051	1.3924	0.0050
0.1796	1.4025	0.0066	1.3977	0.0062	1.3930	0.0060
0.2502	1.4034	0.0079	1.3986	0.0076	1.3937	0.0073
0.3032	1.4037	0.0085	1.3988	0.0083	1.3938	0.0080
0.3664	1.4038	0.0090	1.3988	0.0086	1.3936	0.0083
0.4975	1.4025	0.0085	1.3974	0.0083	1.3922	0.0081
0.5516	1.4016	0.0079	1.3964	0.0077	1.3911	0.0076
0.6264	1.4000	0.0068	1.3947	0.0066	1.3894	0.0065
0.6576	1.3993	0.0062	1.3939	0.0060	1.3885	0.0059
0.7415	1.3971	0.0046	1.3916	0.0043	1.3861	0.0043
0.7846	1.3959	0.0036	1.3904	0.0034	1.3847	0.0033
0.8348	1.3944	0.0024	1.3889	0.0024	1.3832	0.0023
0.8867	1.3932	0.0015	1.3875	0.0014	1.3817	0.0013
0.9150	1.3925	0.0009	1.3868	0.0009	1.3810	0.0008
1	1.3910	0.0000	1.3853	0.0000	1.3794	0.0000
DIPA (1) + sec-butanol (2)						
0	1.3949	0.0000	1.3903	0.0000	1.3856	0.0000
0.0844	1.3965	0.0020	1.3918	0.0019	1.3869	0.0018
0.1445	1.3976	0.0032	1.3927	0.0031	1.3877	0.0030
0.1738	1.3980	0.0037	1.3931	0.0036	1.3880	0.0035
0.2182	1.3985	0.0044	1.3935	0.0043	1.3884	0.0041
0.2787	1.3989	0.0051	1.3938	0.0049	1.3886	0.0048
0.3190	1.3991	0.0054	1.3939	0.0052	1.3886	0.0050
0.3562	1.3991	0.0056	1.3938	0.0053	1.3885	0.0052
0.4180	1.3988	0.0056	1.3936	0.0054	1.3882	0.0052
0.4916	1.3983	0.0053	1.3929	0.0051	1.3875	0.0049
0.5359	1.3978	0.0050	1.3925	0.0048	1.3869	0.0046
0.6035	1.3970	0.0044	1.3916	0.0043	1.3860	0.0041
0.6690	1.3961	0.0038	1.3906	0.0037	1.3849	0.0035
0.7401	1.3951	0.0031	1.3896	0.0030	1.3838	0.0028
0.7887	1.3944	0.0026	1.3888	0.0025	1.3830	0.0023
0.8536	1.3935	0.0019	1.3878	0.0018	1.3820	0.0017
0.9119	1.3926	0.0013	1.3869	0.0011	1.3811	0.0011
1	1.3910	0.0000	1.3853	0.0000	1.3794	0.0000
DIPA (1) + isobutanol (2)						
0	1.3928	0.0000	1.3886	0.0000	1.3844	0.0000
0.1082	1.3970	0.0044	1.3925	0.0043	1.3879	0.0041
0.1366	1.3977	0.0052	1.3931	0.0049	1.3884	0.0047
0.1782	1.3987	0.0062	1.3939	0.0059	1.3891	0.0056
0.2206	1.3994	0.0070	1.3946	0.0068	1.3897	0.0064
0.2688	1.4000	0.0077	1.3951	0.0074	1.3901	0.0071
0.3922	1.4006	0.0086	1.3955	0.0082	1.3904	0.0080
0.4965	1.4004	0.0085	1.3952	0.0082	1.3899	0.0080
0.5782	1.3996	0.0078	1.3943	0.0076	1.3890	0.0074
0.6063	1.3993	0.0076	1.3939	0.0073	1.3885	0.0071
0.6608	1.3984	0.0068	1.3930	0.0066	1.3875	0.0064
0.6898	1.3979	0.0063	1.3925	0.0062	1.3870	0.0060
0.7858	1.3958	0.0045	1.3903	0.0043	1.3846	0.0041
0.8022	1.3954	0.0041	1.3899	0.0040	1.3842	0.0038
0.8396	1.3945	0.0032	1.3890	0.0032	1.3833	0.0031
0.9185	1.3927	0.0015	1.3870	0.0015	1.3812	0.0014
1	1.3910	0.0000	1.3853	0.0000	1.3794	0.0000

Table 2. Continued

x_1	n_D	Δn	n_D	Δn	n_D	Δn
	298.15 K		308.15 K		318.15 K	
DIPA (1) + tert-butanol (2)						
0.0000	1.3846	0.0000	1.3795	0.0000	1.3742	0.0000
0.0617	1.3865	0.0015	1.3812	0.0013	1.3757	0.0012
0.0940	1.3872	0.0020	1.3819	0.0018	1.3764	0.0017
0.1420	1.3882	0.0027	1.3828	0.0025	1.3771	0.0022
0.2234	1.3895	0.0034	1.3840	0.0032	1.3783	0.0029
0.2613	1.3899	0.0036	1.3844	0.0034	1.3787	0.0031
0.3127	1.3904	0.0038	1.3849	0.0036	1.3792	0.0033
0.3835	1.3909	0.0038	1.3854	0.0037	1.3796	0.0034
0.5058	1.3914	0.0036	1.3858	0.0034	1.3800	0.0032
0.6703	1.3915	0.0026	1.3858	0.0024	1.3800	0.0023
0.6079	1.3915	0.0030	1.3859	0.0029	1.3801	0.0027
0.7401	1.3913	0.0020	1.3856	0.0018	1.3798	0.0017
0.8004	1.3912	0.0014	1.3854	0.0012	1.3796	0.0012
0.8665	1.3910	0.0008	1.3852	0.0007	1.3794	0.0007
0.9025	1.3909	0.0006	1.3851	0.0004	1.3793	0.0004
0.9383	1.3909	0.0003	1.3851	0.0002	1.3793	0.0002
1	1.3910	0.0000	1.3853	0.0000	1.3794	0.0000

where X_i is the measured property of the compound, $\overline{X}$ is the average of multiple measured data and N is the number of experimental points.

The densities (ρ) and refractive indices (n_D) of pure compounds and their binary mixtures are given in Table 1 and Table 2, respectively.

3. Results and Discussion

The deviation in refractive index Δn was calculated from the measured refractive index data for binary DIPA (1) + isomeric butanol (2) mixtures using Eq. (1):

$$\Delta n = n_D - \sum_{i=1}^{2} x_i n_{Di} \tag{1}$$

where n_{Di} and n_D are the refractive index of ith components and their binary mixture, respectively. The n_D and Δn for the binary mixtures in whole range of mole fractions are given in Table 2. The Δn values were also fitted to Redlich-Kister equation:

$$\Delta n = x_1(1-x_1)\left[\sum_{n=1}^{4} A^{(j)}\left(2x_1-1\right)^{(j-1)}\right] \tag{2}$$

where $A^{(j)}$ are the adjustable parameters and calculated by fitting Δn data in Eq. (2) and were recorded in Table 3 along with respective standard deviations $\sigma(\Delta n)$. The experimental Δn data as well as values calculated by Redlich-Kister equation are shown in Fig. 1.

The n_D values were further predicted in terms of various correlations like Arago-Biot (A-B), Gladston-Dale (G-D), Lorentz (L-L), Heller (H), Weiner (W), Newton (Nw), and Erying-John (E-J) (Eqs. (3)-(9)).

Table 3. Redlich-Kister equation parameters ($A^{(j)}$) and standard deviation (σ)

T/K	$A^{(1)}$	$A^{(2)}$	$A^{(3)}$	$A^{(4)}$	σ
DIPA (1) + n-butanol (2)					
298.15	0.0340	-0.0179	-0.0059	-0.0044	3×10^{-05}
308.15	0.0331	-0.0174	-0.0077	-0.0028	2×10^{-05}
318.15	0.0323	-0.0157	-0.0079	-0.0063	3×10^{-05}
DIPA (1) + sec-butanol (2)					
298.15	0.0210	-0.0118	-0.0006	0.0086	9×10^{-06}
308.15	0.0202	-0.0111	-0.0008	0.0065	5×10^{-06}
318.15	0.0195	-0.0116	-0.0013	0.0085	1×10^{-05}
DIPA (1) + iso-butanol (2)					
298.15	0.0337	-0.0097	-0.0003	-0.0093	3×10^{-05}
308.15	0.0326	-0.0088	-0.0008	-0.0093	4×10^{-05}
318.15	0.0318	-0.008	-0.0023	-0.0091	5×10^{-05}
DIPA (1) + tert-butanol (2)					
298.15	0.0143	-0.0075	0.0012	-0.0054	2×10^{-05}
308.15	0.0137	-0.0074	-0.0011	-0.0049	2×10^{-05}
318.15	0.0129	-0.0064	-0.0014	-0.0043	2×10^{-05}

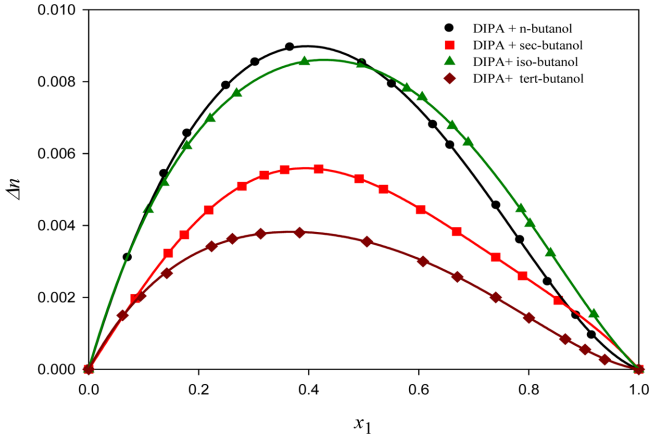

Fig. 1. Deviation in refractive index (Δn) with mole fraction of DIPA (x_1) at 298.15 K. Experimental values (Symbols) and Redlich-Kister Values (Solid lines).

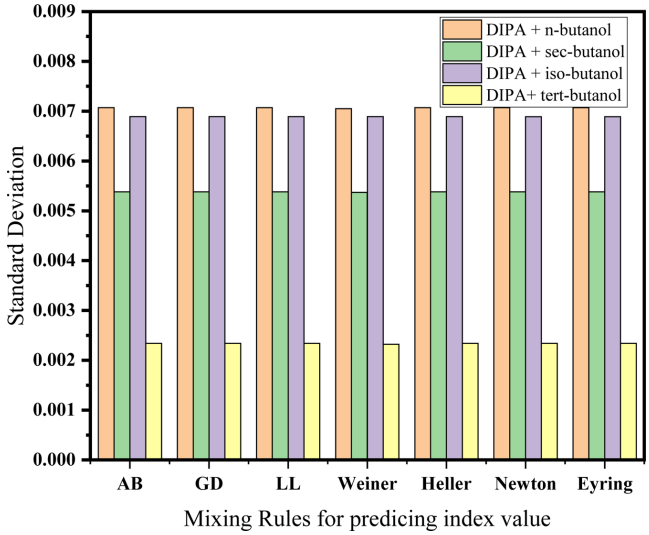

Fig. 2. Standard deviation in n_D calculated by various correlations Eqs. (3-9) for binary DIPA (1) + butanol (2) at 298.15 K.

The details of these mixing rules are given in our earlier paper [53].

Arago-Biot (A-B) $n_D = n_{D1}\phi_1 + n_{D2}\phi_2$ (3)

Gladstone-Dale (G-D) $n_D - 1 = (n_{D1} - 1)\phi_1 + (n_{D2} - 1)\phi_2$ (4)

Lorentz-Lorenz (L-L) $\frac{n_D^2 - 1}{n_D^2 + 2} = \left(\frac{n_{D1}^2 - 1}{n_D^2 + 2}\right)\phi_1 + \left(\frac{n_{D2}^2 - 1}{n_D^2 + 2}\right)\phi_2$ (5)

Heller (H) $\frac{n_D - 1}{n_D} = \frac{3}{2}\left(\frac{(n_{D2}/n_{D1}) - 1}{(n_{D2}/n_{D1}) + 1}\right)\phi_2$ (6)

Weiner (W) $\frac{n_D^2 - n_{D1}^2}{n_D^2 + 2n_{D2}^2} = \left(\frac{n_D^2 - n_{D1}^2}{n_{D2}^2 + 2n_{D2}^2}\right)\phi_2$ (7)

Newton (Nw) $n_D^2 - 1 = (n_{D1}^2 - 1)\phi_1 + (n_{D2}^2 - 1)\phi_2$ (8)

Eyring and John (E-J) $n_D = n_{D1}\phi_1^2 + 2(n_{D1}n_{D2})^{1/2}\phi_1\phi_2 + n_{D2}\phi_2^2$ (9)

In all the above mixing correlations n_{Di} (where $i = 1$ or 2) and n_D are the refractive index of pure components binary mixture. Here ϕ_1 and ϕ_2 represent the volume fraction of pure components 1 and 2 and can be given as

$$\phi_1 = x_1V_1/\sum x_1V_1 \text{ and } \phi_2 = x_2V_2/\sum x_iV_i \quad (10)$$

The details of these mixing rules are given elsewhere [53]. The results from various refractive index correlations were compared and found to agree well with the experimental data.

The deviations in the experimental and theoretical results from various correlations are presented in the form of standard deviation and also in Fig. 2 (only at 298.15 K). A good agreement was observed for all the mixing relations.

The Δn for DIPA (1) + n-butanol (2) or + sec-butanol (2) or + isobutanol (2) or + tert-butanol (2) are positive (Fig. 1). The positive value of Δn denotes the interactive interaction between DIPA and isomeric butanol molecules. In the present binary mixtures, the presence of polar function group (-NH and –OH) also addresses the presence of stronger H-bonding between unlike molecules which leads to positive deviation in Δn. Further, negative V_m^E values for all the binary mixture also support the strong H-bonding between unlike molecules (Fig. 3). DIPA and isomeric butanol have hydrogen bonding. The sign and magnitude of V_m^E is the result of unlike intermolecular interaction interactions as well as molecular packing in liquid mixture. The first factor is associated with disruption of forces between the components of like molecules and formation of interactions between unlike molecule of liquid mixtures. Presence of different functional groups alters the magnitude of interactions in the mixture. On the other hand, molecular arrangements in the liquid mixtures are influenced by structure and shape of molecule. The cumulative effect of these two factors decides the sign and magnitude of V_m^E. In the present systems, negative values V_m^E over the whole composition predict the stronger intermolecular attractive forces between amine group (-NH) of DIPA and -OH group of butanol and also somewhat better packing in mixed state.

The Δn values for equimolar mixtures of DIPA (1) + isomeric butanol (2) follow the sequence: n-butanol > iso-butanol > sec-butanol > tert-butanol. The branching at the carbon atom attached to the hydroxyl group obstructs the approach/interaction of hydroxyl hydrogen (OH^-) of butanol and -NH group of amines [50]. Thus Δn value decreases for with increase in branching of butyl group and was found to be maximum for n-butanol and minimum for *tert*-butanol.

3-1. The excess volume from refractive index: Nakata and Sakurai model

For the prediction of V_m^E values from n_D data, the Nakata and Sakurai model (Model I) is used [34]. According to this model,

$$V_m^E = \sum_{i=1}^{2}\left[(f(n_{Di}) - f(n_D))\left(\frac{x_i V_i}{f(n_D)}\right)\right] \quad (11)$$

In the above equation, the specific refraction is presented as

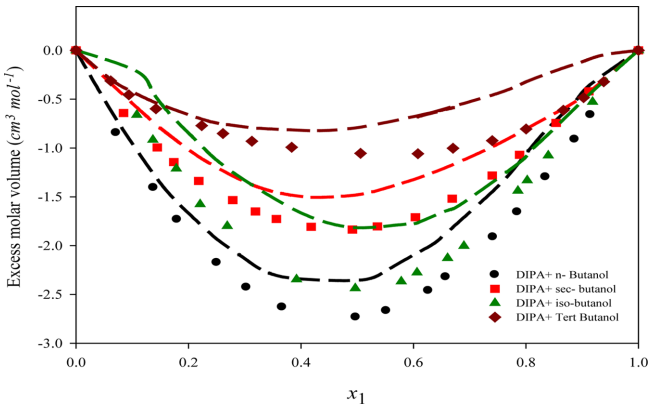

Fig. 3. Excess molar volume (V_m^E) versus mole fraction of DIPA (x_1) at 298.15 K. Experimental values (Symbols) and Nakata and Sakurai model [34] (dashed lines).

$$\frac{f(n_D)}{\rho} = \sum_{i=1}^{2} w_i \frac{f(n_{Di})}{\rho_i} \tag{12}$$

In Eqs. (11) and (12), subscript '*i*' represents the pure component DIPA (1) or butanol (2) and absence of subscript represents a binary mixture. In these equations, w, ρ, n_D, x, V represent the mass fraction, density, refractive index, mole fraction and molar volume, respectively, and $f(n_D)$ is a function of refractive index.

The V_m^E values were predicted from the experimental n_D data at 298.15 K for the DIPA (1) + butanol isomers (2) with the incorporation of L-L mixing rules, and the results were compared with the experimental excess volume data in Fig. 3. This equation predicts the sign and shape of excess volume curves only except for isobutanol mixtures in the mole fraction below 0.2 (Fig. 3) but unable to predict the magnitude.

The Δn values for DIPA (1) + isomeric butanols (2) at equimolar mixtures follow the sequence: n-butanol > iso-butanol > sec-butanol > tert-butanol. The refractive index is an index of speed of light, varied with density of the medium. With increase in density of the medium, n_D also increases. As in the binary mixtures of DIPA + n-butanol, n-butanol is highly approachable due to straight chain molecule and forms strong H-bonding with DIPA, causing maximum increase in density and hence the refractive index. Consequently, Δn is highly positive and maximum in this case. As *tert*-butanol has maximum branching at the carbon atom attached to hydroxyl group among all the isomers of butanol, therefore offers maximum steric hindrance to hydroxyl hydrogen to interact with –NH group. This weakens the H-bonding between both components to the minimum. Consequently, density as well as Δn should be minimum for *tert*-butanol mixture. This is what we have observed (Fig. 1). The least interaction of DIPA and tert-butanol may also be attributed to the higher exposed hydrophobic part due to its geometry, which makes it less approachable towards DIPA causing less specific interactions.

Temperature also affects Δn values. As temperature increases from 298.15 K to 308.15 K, the Δn values decrease, which is due to decrease in density of the medium owing to weakening of intermolecular H-bonding. The effect of temperature on Δn values for DIPA+ tert-butanol is higher in comparison to DIPA+ n-butanol or sec-butanol or iso-butanol. It is because of the lesser interaction of DIPA + tert-butanol molecule, which are further more affected by the increase in temperature.

4. Conclusion

The refractive index for the binary mixtures of diisopropylamine (DIPA) + isomeric butanol was measured and Δn values were determined from 298.15 K to 308.15 K. The Δn values for all the binary mixtures were found positive and magnitude of Δn for DIPA (1) + isomeric butanol (2) at equimolar mixtures follow the sequence: n-butanol > iso-butanol > sec-butanol > tert-butanol. This trend is observed as a result of cumulative effect of molecular interactions between unlike molecules and packing of the molecules in binary mixtures. The negative values of Δn represent the specific molecular interactions. Moreover, highest value of Δn in DIPA + n-butanol shows higher magnitude of specific interactions, which is least in case of DIPA + tert-butanol. The results were well explained on the basis of size, shape and geometry of the molecules. The refractive index data were also analyzed in terms of various mixing correlations and found to be in good agreement with experimental results. Nakata and Sakurai model [34] was applied to predict the V_m^E values from refractive index data of pure as well as their binary mixtures, and it predicted the sign and shape of excess molar volume (V_m^E) versus mole fraction (x_1) curves for the present binary systems.

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