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Acoustical and thermodynamic study of binary mixture cyclohexane-methanol using ultrasonic interferometer at different temperatures

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The ultrasonic velocity (U) mass density (ρ) and shear viscosity (η) of the binary liquid mixtures of cyclohexane-methanol have been determined experimentally in the single-phase region and over the whole composition range at temperature range (T = 321.15 to 325.15 K). The experimental measurements of these properties have been carried out at atmospheric pressure, for a constant frequency 2 MHz. From these experimental data values, various acoustic and thermodynamic parameters namely adiabatic compressibility (βs), acoustic impedance (Z), intermolecular free length (L_f), relaxation time (τ), molar volume (V_m), free volume (V_f), internal pressure (π_i), attenuation (α/f^2), Gibb's energy (ΔG), and cohesive energy (CE) have been calculated. Also their excess values have been calculated. All these parameters and their excess values have been interpreted in terms of molecular interaction such as dipole-dipole and dipole induced dipole interactions through hydrogen bonding between components of binary liquid mixture.

Keywords: Cyclohexane-methanol binary liquid, Molecular interactions, Ultrasonic velocity, Shear viscosity, Acoustic parameters, Excess values

1 Introduction

The present work is part of a research program whose objective is a systematic study of the thermodynamic and transport properties of binary mixtures. In previous papers[1-6], we have reported experimental data on macroscopic properties such as densities, viscosities, refractive indexes and specific electrical conductivities for water-isobutyric acid, water-triethylamine and water-1,4-dioxane binary liquids. From these results, it was observed that the existing specific interactions between the components play an important role in intermolecular association in the medium. As an extension of our studies, we have undertaken a new program for studying the ultrasonic velocity measurement for binary liquid mixtures in order to understand possible associations existing between the various species in the solutions.

The ultrasonic velocity of a liquid is deeply connected to the binding forces between atoms or molecules and has been fruitfully employed in understanding the nature of molecular interactions not only in pure liquids but also in binary and ternary mixtures[7-9]. The measurement of ultrasonic speed in liquid mixture enables us to determine some thermodynamically parameters and their excess values. Knowledge of acoustic and thermodynamic properties in liquid mixture provides valuable insight in to the physical nature and strength of molecular interaction in the studied medium. Excess parameters play a significant role in assessing the compactness due to molecular arrangement and the extent of molecular interactions in the liquid mixtures through charge transfer, dipole-dipole and dipole induced dipole interactions[10]. The sign of these functions depend on the strength of interaction between different molecules[11]. Important physico-chemical parameters such as adiabatic compressibility (βs), intermolecular free length (L_f), acoustic impedance (Z), relaxation time (τ), free volume (V_f), internal pressure (π_i), attenuation (α/f^2), Gibb's energy (ΔG), and cohesive energy (CE), and their excess parameters can be calculated from acoustic and thermodynamic data.

In the present study, we report ultrasonic velocity, densities and shear viscosities of binary mixture cyclohexane-methanol system known as typical, partly miscible system that has an upper critical solution temperature. Critical temperature that was

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*Corresponding author (E-mail: adel.toumi@fst.rnu.tn) identified visually in our laboratory to be T_c = 319.608±0.234 K at the critical composition Cc =27.5 % of methanol mass fraction[12]. Since, methanol is known to be very hygroscopic and cyclohexane as a non-polar, unassociated molecule, inert hydrocarbon possessing globular structure[13]. We have measured densities, shear viscosities and ultrasonic velocity at different temperatures in the single phase region($T > T_c$), under atmospheric pressure, along the coexistence curve for methanol mole fraction in the temperature range (321.15–325.15 K). The dependence of these quantities on mole fraction of the binary mixture is discussed.

2 Materials and Method

The cyclohexane and methanol were purchased from prolabo without any further purification. All mixtures were prepared from weighed amounts of the pure components. The weight was performed by using an electronic balance with a resolution of 10^{-3} g. The cell, in which the cyclohexane and methanol were mixed together, was immersed in a thermally stabilized water bath with thermal regulation in the order of 10^{-3} K over hours. The temperature was measured by using a quartz thermometer giving a resolution of ± 10^{-3} K. (Table 1)

Table 1 — A brief summary of the purity of the used materials.

Compound	CAS registry number	Formula	Supplier Mass fraction purity	Purification method
Cyclohexane	110-82-7	C_6H_{12}	Prolabo 0.998	none
Methanol	67-56-1	CH_4O	Prolabo 0.998	none

2.1 Density measurement

The mass density of the samples were measured by means of an Anton Paar digital oscillating U-tube densitometer (model DMA 4500) and samples were observed with a reproducibility of $\pm 2\times10^{-5}$ g.cm^{-3}. The apparatus was calibrated by using the published density values in references[14,15].

2.2 Viscosity measurement

The kinematic viscosities of the cyclohexane - methanol mixtures were measured with an Ubbelhode viscometer suspended in a thermostatically controlled water bath at the required temperature. The viscometer was calibrated using viscosity values taken from published values[16]. An average of four or five sets of flow times were taken for each liquid and liquid mixture. The measured values of kinematic viscosities were converted into dynamic viscosities η after multiplication by the density. The flow–time measurements were made with an electronic stop watch having a precision of the order of 0.01 s.

2.3 Ultrasonic velocity measurement

The velocity of ultrasonic waves in the liquid mixture was measured using an ultrasonic interferometer (Model-81, supplied by Mittal Enterprises) operating at a frequency of 2 MHz. The temperature around this device was maintained within 0.01 K in electronically controlled thermostatic water bath. The measuring cell is a specially designed double walled vessel with provision of temperature constancy. The high frequency generator excites a quartz crystal fixed at the bottom of the measuring cell, at its resonant frequency. The capacity of the measuring cell is 12 cc. A fine micrometer screw, with a least count of 0.001 mm at the top of the cell, can raise (or) drop the reflector plate in the liquid through a known distance. The measuring cell is connected to the output terminals of the high frequency generator through a shielded cable. Ultrasonic waves, normal to quartz crystal, are reflected from the reflector plate. Stationary waves are formed in the region between reflector plate and the quartz crystal. The micrometer was slowly moving till a number of maximum readings (n) of the anode current went through. The total distance (d) moved by the micrometer was noted. The wavelength of the ultrasonic waves in the liquid is $\lambda = 2d/n$. The velocity of ultrasonic waves in the liquid $U = \lambda f$, where f is the generator frequency.

Purity of each compound was ascertained by measuring their densities and viscosities at different temperatures, which agreed with those published in the literature[17-23], as given in Table 2.

3 Theory and Calculation

The ultrasonic velocity (U), density (ρ) and viscosity (η) in pure liquids and liquid mixtures of various concentrations were measured at 321.15, 322.15, 323.15 and 325.15 K. Thermodynamic and acoustical parameters such as adiabatic compressibility (β), intermolecular free length (L_f), viscous relaxation time (τ), Gibbs free energy (ΔG), internal pressure (π_i), free volume (V_f), specific acoustic impedance (Z) and attenuation coefficient (α/f^2) were determined using the observed values of velocity, density and viscosity by means of the equations.

3.1 Adiabatic compressibility

The adiabatic compressibility is the fractional decrease of volume per unit increase of pressure,

Table 2 — Comparison of experimental ultrasonic velocity (U) of pure components with available literature values at 303.15 K, 308.15 K, 313.15 K and 318.15 K and atmospheric pressure P= 101.32 kPa[a].

Compounds	T (K)	U (m.s^{-1})	
		Experimental	Literature
Cyclohexane	303.15	1227.29	1230 [17,18] 1228 [19]
	308.1	1206.00	1208[19]
	313.15	1183.29	1188.00 [19] 1181.90 [20]
	318.15	1158.18	1160.90 [21]
Methanol	303.15	1090.00	1085.80 [22] 1086.37 [23]
	308.15	1076.53	1070.11 [23]
	313.15	1054.70	1053.20 [22] 1053.96 [23]
	318.15	1040.96	1037.91 [23]

[a]Standard uncertainties u (U) = 0.2 m.s^{-1}, u(T) = 0.01 K and u (P)= 0.05 kPa

when no heat flows in or out. It also determines the orientation of the solvent molecules around the liquid molecules. The structural arrangement of the molecule affecting the adiabatic compressibility has been calculated from sound velocity (U) and the density (ρ) of the medium using the Newton - Laplace equation[24]:

$$\beta = \frac{1}{\rho\, U^2} \qquad \dots (1)$$

3.2 Impedance acoustic

Acoustic impedance is important in the determination of acoustic transmission and reflexion of two materials having different acoustic impedance, design of ultrasonic transducers. It is also useful in the design of ultrasonic transducers and for assessing absorption of sound in a medium. The specific acoustic impedance is given by:

$$Z = U\rho \qquad \dots (2)$$

where U and ρ are the ultrasonic velocity and density of the mixture, respectively.

3.3 Intermolecular free length (L_f)

The intermolecular free length is the distance between the surfaces of the neighboring molecules. It is a predominant factor in determining the variation of the ultrasonic velocity of solution. It is calculated by using the relation[25]:

$$L_f = K_T \beta^{1/2} \qquad \dots (3)$$

Where K_T is the temperature dependent constant and whose numerical value, K_T = (93.875 + 0.375 ×T) ×10^{-8}, is valued from literature[26] (T is the absolute temperature and β is the adiabatic compressibility).

3.4 Relaxation time (τ)

Relaxation time is the time taken for the excitation energy to appear as translational energy, and it depends on temperature and impurities. The relaxation time can be calculated from the following relation:

$$\tau = \frac{4\eta}{3\rho U^2} \qquad \dots (4)$$

3.5 Molar volume

$$V_m = \frac{\bar{M}}{\rho} \qquad \dots (5)$$

Where$\bar{M} = x_1M_1 + x_2M_2$ is the effective molecular weight given as $\bar{M} = \sum m_i x_i$ (m_i is the molecular weights and mole fraction of individual constituents)

3.6 Free volume (V_f)

On the basis of dimensional analysis, Suryanarayana and Kuppusamy[27] have obtained an expression for free volume in terms of experimentally measurable parameters of ultrasonic velocity and viscosity, and are given by:

$$V_f = \left(\frac{MU}{K\eta}\right)^{\frac{3}{2}} \qquad \dots (8)$$

where M is the effective mass of the mixture, K is a dimensionless constant independent of temperature and liquid. Its value is 4.281 × 10^{9}.

3.7 Attenuation α/f2

The absorption coefficient is characteristic of the medium and it also depends on the external conditions like temperature, pressure and the frequency of measurement. It is a measure of spatial rate of decrease in the intensity level on ultrasonic waves. Treating the propagation of sound as an adiabatic phenomenon and following Stoke's theory, absorption may be considered proportional to the viscosity of the medium and the square of the frequency. It can be calculated from the viscosity (η) using the relation:

$$\alpha/f^2 = \frac{8\pi^2\eta}{3\rho U^3} \qquad \dots (10)$$

Where α is absorption coefficient, f is frequency, η is shear viscosity, ρ is density and U is ultrasonic velocity in dispersion[28].

3.8 Internal pressure (π_i)

The measurement of internal pressure (π_i) is important in the study of the thermodynamic properties of liquids. The internal pressure is the cohesive force, which is a resultant force of attraction and repulsion forces of between the interacting components. The attractive forces mainly consist of hydrogen bonding, dipole-dipole and dispersion interaction. Repulsive forces are acting over very small intermolecular distances. It is calculated by using the relation[29]:

$$\pi_{\mathrm{i}} = bRT\left(\frac{K\eta}{U}\right)^{1/2}\frac{\rho^{2/3}}{M^{7/6}} \qquad (11)$$

where, b stands for cubic packing, which is assumed to be 2 for all liquids; K is a dimensionless constant independent of temperature and nature of liquids. Its value is 4.281×10^9. T is the absolute temperature expressed in Kelvin, M is the effective molecular weight, R is the universal gas constant, η is the viscosity and U is the ultrasonic velocity.

4 Results and Discussion

The experimental values of density (ρ), viscosity (η) and ultrasonic velocity (U) for pure cyclohexane and methanol liquids and their mixtures at 321.15, 322.15, 323.15 and 325.15 K are listed in Table 3. The ultrasonic velocity behavior is shown in Fig. 1. The dependences of the thermodynamic parameters such as adiabatic compressibility (β), acoustic impedance (Z), free length (L_f), relaxation time (τ) and absorption constant (α/f^2) are displayed in Figs 2-6. The excess variations of ultrasonic velocity U^E, adiabatic compressibility β^E and molar volume (V_m^E) are shown in Figs 7-9.

Ultrasonic velocity (U) represents an acoustical parameter that can provide good information about the molecular interactions between liquid mixtures. The existence of interaction between liquid mixtures is ascertained by the non-linear variation of U with respect to the mole fraction. In fact, the ultrasonic velocity variation through the mixture depends on the increase or decrease of the free length L_f. It is observed that the U and Z, values reached a minimum with a methanol mole fraction near 0.8 and decreased with increase the temperature, while those of β and L_f, decrease with increasing temperature and reach a maximum near 0.8 of mole fraction of methanol. The geometry of molecules and the structure arrangement have a role in deciding on the ultrasonic velocity of a liquid.

The adiabatic compressibility is a powerful thermodynamic parameter in sensing the molecular interactions in liquid mixtures[30]. In addition, Eyring and Kincaid[31] have proposed that the intermolecular free length (L_f) which is the distance between the surfaces of two molecules[32] is a predominant factor in determining the variation of the ultrasonic velocity of solution. This free length depends on intermolecular as well as intermolecular interaction existing in and

Table 3 — Experimental values of ultrasonic velocity (U),density (ρ), viscosity(η), as a function of mole fraction (x) of methanol at 321.15 K, 322.15 K, 323.15 K and 325.15 K and atmospheric pressure P = 101.32 kPa[a].

$x_{\mathrm{Méthanol}}$	U ($\mathrm{m.s^{-1}}$)	ρ (10^3 $\mathrm{kg.m^{-3}}$)	η (10^{-3}Pa.s)	U ($\mathrm{m.s^{-1}}$)	ρ (10^3 $\mathrm{kg.m^{-3}}$)	η (10^{-3}Pa.s)	U ($\mathrm{m.s^{-1}}$)	ρ (10^3 $\mathrm{kg.m^{-3}}$)	η (10^{-3}Pa.s)	U ($\mathrm{m.s^{-1}}$)	ρ (10^3 $\mathrm{kg.m^{-3}}$)	η (10^{-3}Pa.s)
		321.15 K			322.15 K			323.15 K			325.15 K	
1.000	1040.45	0.76518	0.4101	1037.70	0.76421	0.4068	1034.96	0.76324	0.4010	1029.46	0.76142	0.3925
0.959	1034.49	0.76090	0.4053	1031.95	0.75992	0.4003	1030.91	0.75894	0.3956	1026.49	0.75717	0.3862
0.913	1029.65	0.75679	0.4122	1026.81	0.75580	0.4074	1025.52	0.75481	0.4024	1020.53	0.75305	0.3931
0.860	1029.98	0.75362	0.4249	1026.56	0.75262	0.4198	1023.15	0.75161	0.4148	1016.32	0.74982	0.4048
0.830	1028.19	0.75241	0.4338	1024.51	0.75140	0.4284	1020.82	0.75037	0.4233	1013.46	0.74852	0.4130
0.798	1028.61	0.75202	0.4443	1024.73	0.75100	0.4363	1020.85	0.74969	0.4338	1013.09	0.74808	0.4234
0.762	1033.13	0.75122	0.4569	1028.70	0.75020	0.4514	1024.26	0.74878	0.4459	1015.4	0.74725	0.4352
0.724	1041.28	0.75076	0.4688	1036.58	0.74974	0.4624	1031.95	0.74873	0.4564	1023.71	0.74682	0.4438
0.637	1052.91	0.74961	0.5116	1047.99	0.74855	0.5017	1041.53	0.74751	0.4925	1031.45	0.74515	0.4760
0.527	1073.69	0.74902	0.5771	1067.69	0.74795	0.5464	1059.47	0.74689	0.5300	1048.75	0.74484	0.5060
0.500	1078.75	0.74858	0.5933	1075.11	0.74754	0.5559	1067.42	0.74649	0.5379	1057.29	0.74511	0.5133
0.467	1082.18	0.74853	0.5629	1074.53	0.74749	0.5508	1068.98	0.74644	0.5356	1058.15	0.74447	0.5129
0.396	1094.32	0.74875	0.5423	1088.91	0.74773	0.5334	1084.13	0.74671	0.5239	1074.8	0.74454	0.5071
0.226	1116.18	0.74895	0.5465	1111.57	0.74792	0.5386	1107.08	0.74688	0.5465	1098.1	0.74479	0.5153
0.121	1136.49	0.74962	0.5549	1132.96	0.74862	0.5469	1129.44	0.74761	0.5390	1122.38	0.74568	0.5240
0.000	1144.02	0.75170	0.5964	1139.30	0.75073	0.5901	1134.58	0.74976	0.5842	1125.13	0.74795	0.5734

[a] Standard uncertainties $u(U) = 0.2\ \mathrm{m.s^{-1}}$, $u(\rho) = 0.00005\ \mathrm{g.cm^{-1}}$, $u(T) = 0.01$ K and $u(P) = 0.05$ kPa

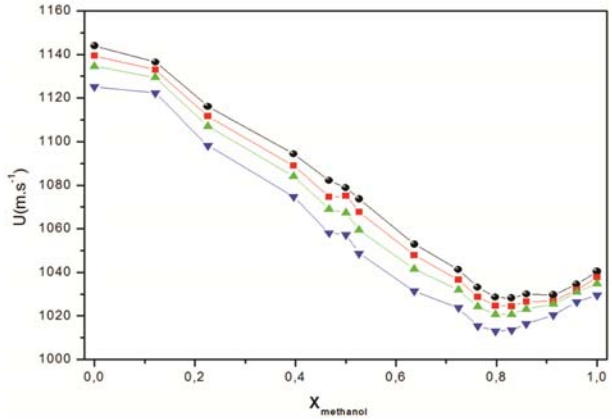

Fig. 1 — Plot of ultrasonic velocity (U) with temperature of mole fraction of methanol at temperatures 321.15 K (•), 322.15 K (■), 323.15 K (▲) and 325.15 K (▼).

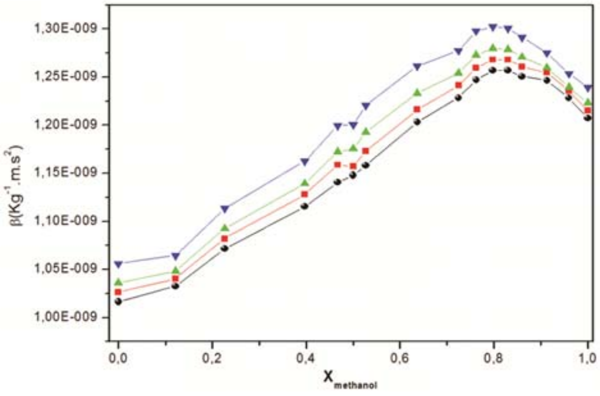

Fig. 2 — Compressibility adiabatic (β) variation against mole fraction for methanol at temperatures 321.15 K (•), 322.15 K (■), 323.15 K (▲) and 325.15 K (▼).

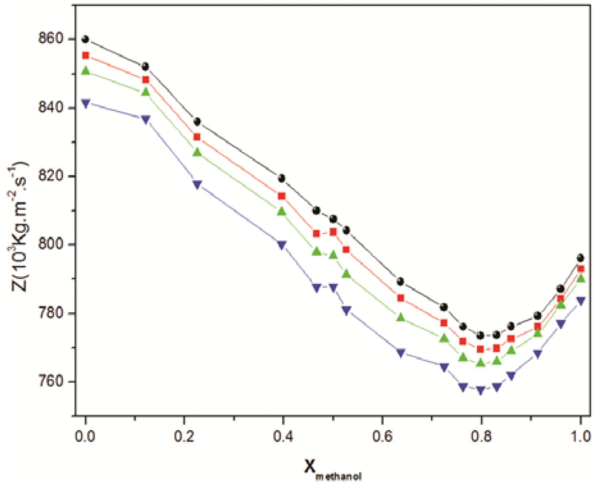

Fig. 3 — Acoustic impedance (Z) of mixture binary cyclohexane-methanol as a function of mole fraction of methanol at temperatures 321.15 K (•), 322.15 K (■), 323.15 K (▲) and 325.15 K (▼).

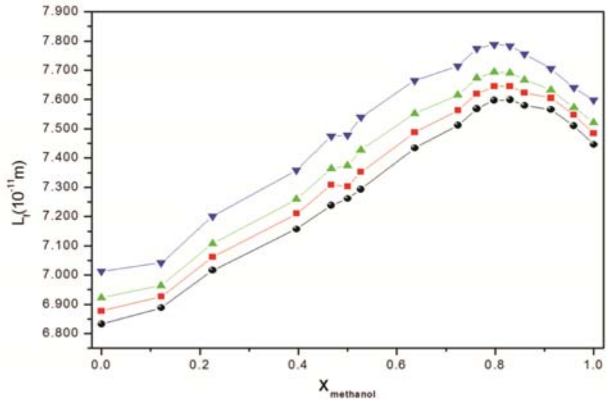

Fig. 4 — Free length (L_f) of mixture binary of cyclohexane with methanol as a function of mole fraction of methanol at temperatures 321.15 K (•), 322.15 K (■), 323.15 K (▲) and 325.15 K (▼).

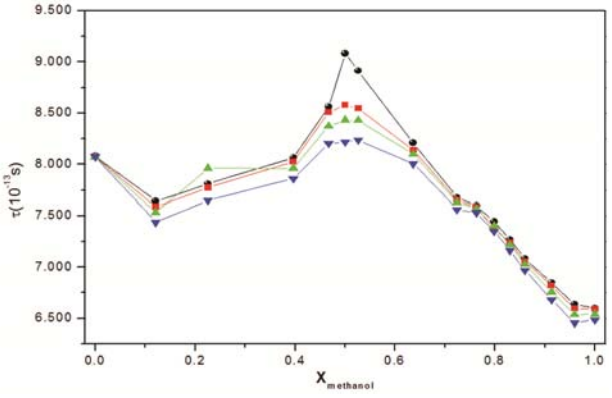

Fig. 5 – Relaxation time (τ) of mixture binary cyclohexane-methanol as a function of mole fraction of methanol at temperatures 321.15 K (•), 322.15 K (■), 323.15 K (▲) and 325.15 K (▼).

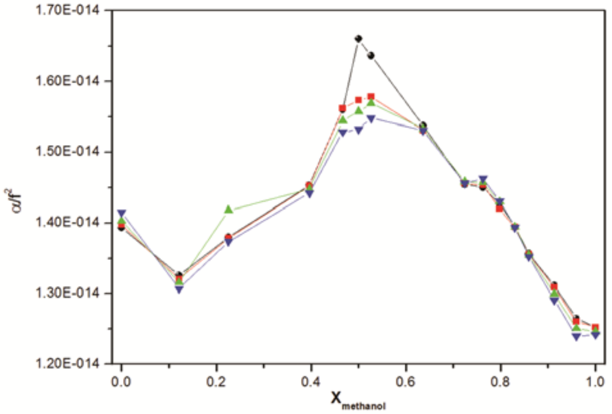

Fig. 6 — Absorption coefficient (α/f^2) versus mole fraction of methanol for the binary cyclohexane-methanol mixture at different temperatures 321.15 K (•), 322.15 K (■), 323.15 K (▲) and 325.15 K (▼).

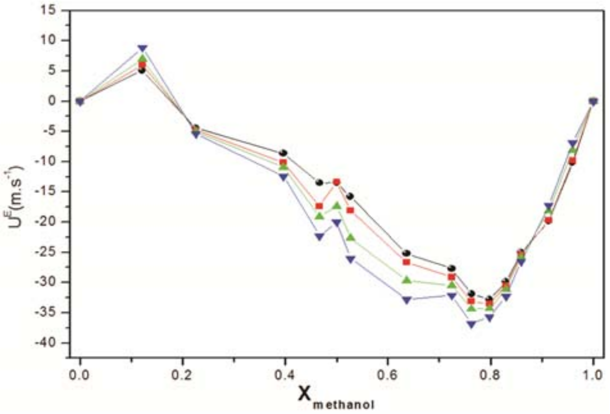

Fig. 7 — Excess ultrasonic velocity for cyclohexane-methanol mixture at temperatures 321.15 K (●), 322.15 K (■), 323.15 K (▲) and 325.15 K (▼) as a function of methanol mole fraction *x*.

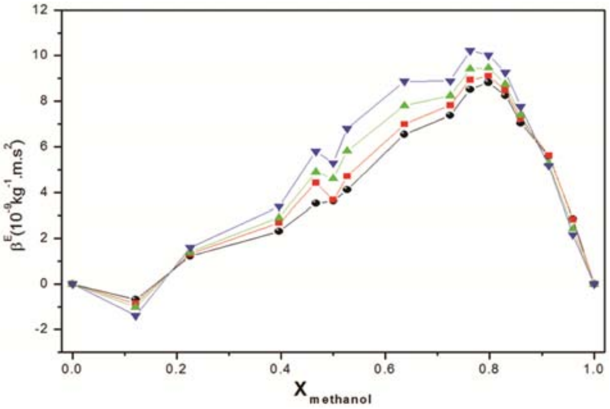

Fig. 8 – Plot of excess adiabatic compressibility (β^E) with temperature of mole fraction of methanol at temperatures 321.15 K (●), 322.15 K (■), 323.15 K (▲) and 325.15 K (▼).

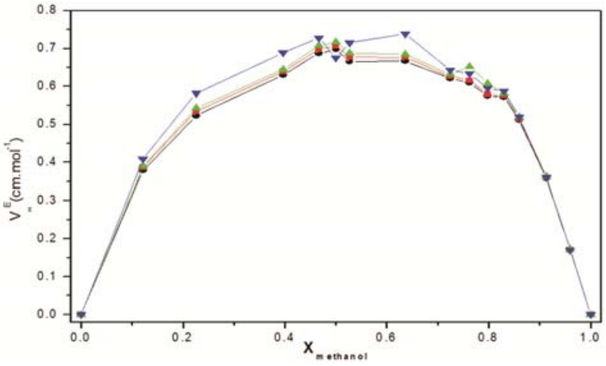

Fig. 9 – Excess molar volume (V_f^E) variation against mole fraction for methanol at temperatures 321.15 K (●), 322.15 K (■), 323.15 K (▲) and 325.15 K (▼).

between components of the mixture. These intermolecular interactions decompose in attractive and repulsive forces. The repulsive forces are dependent on the distance between the surfaces of the molecules, while the attractive forces are depending on the distance between what are named the molecule attraction centers. Molecular association is thus responsible for the observed decrease of free length. As depicted in Fig. 4, the general free length increases with methanol concentration increase.

The absorption coefficient (α/f^2) and the relaxation time (τ) have reached a maximum at the value of 0.5 mole fraction of methanol and decrease as the temperature increases. This is due to strong intermolecular attraction between the molecules of the components. Variation in the attenuation coefficient is a measure of spatial rate of decrease in the intensity level of the ultrasonic wave. The relaxation time increase is due to structural relaxation process, which shows the presence of molecular interaction between the component molecules. As the temperature increases, excitation energy increases, and therefore, relaxation time decreases, so longer time is taken for the rearrangement of molecules (Table 4).

The internal pressure (π_i) in a binary mixture is a measure of intermolecular attraction between the components. As shown in Table 4, when the methanol concentration increases internal pressure increases, suggesting strong intermolecular hydrogen bonding which may be due to the polarity of methanol. However, the free volume decreases with increasing the methanol concentration. This decrease of free volume indicates an increase in molecular attraction since the free volume is the average volume in which the center of a molecule can move due to the repulsion of the surrounding molecules. As the temperature is increased, the free volume increases. Thermal energy of the molecules increases with temperature, hence available free volume should be more.

The excess thermodynamic parameters helps us to understand the nature of molecular interaction between the components of mixtures which were studied by several workers[33-35], these parameters are defined as the difference between the experimental and ideal mixture values. It gives a measure of the non-ideality of the system as a consequence of associative or of other interactions[36]. The excess values of the parameters have been calculated using the following expression[37]:

$$Y^{\mathrm{E}} = Y_{\mathrm{exp}} - \sum x_{\mathrm{i}} Y_{\mathrm{i}} \qquad \ldots (15)$$

The positive and negative deviations in these functions of the mixture indicate the extent of dissociation or association between diver molecules of the mixture. These deviations may be attributed to different type of interactions between the different molecules of the mixtures.

The excess ultrasonic velocity and acoustic impedance are negative and decrease with increasing temperature. The negative value was indicated by decreasing interactions strength between different molecules of the mixture[38]. The excess properties depend upon several physical and chemical contributions. The physical contribution consists of dispersion forces or weak dipole-dipole interaction that leads to positive values of β^E and V_m^E. Chemical contributions include breaking up of the associates present in pure liquids, resulting in positive of β^E and V_m^E. The positive values of β^E and V_m^E are mainly due to the weak interaction[39]. In fact, Fort and Moore[40] noticed that the positive excess values of these function indicate the increase in strength of interaction between the unlike molecules. Hence, the excess functions studies for these systems have a huge importance for understanding the presence of molecular interactions.

5 Conclusions

From the experimental observation, the physical and acoustical parameters have been determined in the single-phase region of this binary fluid from $T = 321.15 - 325.15$ K. An analysis of the results suggests the presence of strong intermolecular interaction between the components of molecules, resulting from dipole-dipole and dipole-induced dipole interaction through hydrogen bonding. The conclusion highlights the importance of ultrasonic method in detecting weak molecular interaction between the component molecules of the mixtures. The intermolecular interactions increase with temperature. The change of the sign in the excess parameters reveals that there is a strong molecular interaction between different molecules in the system.

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