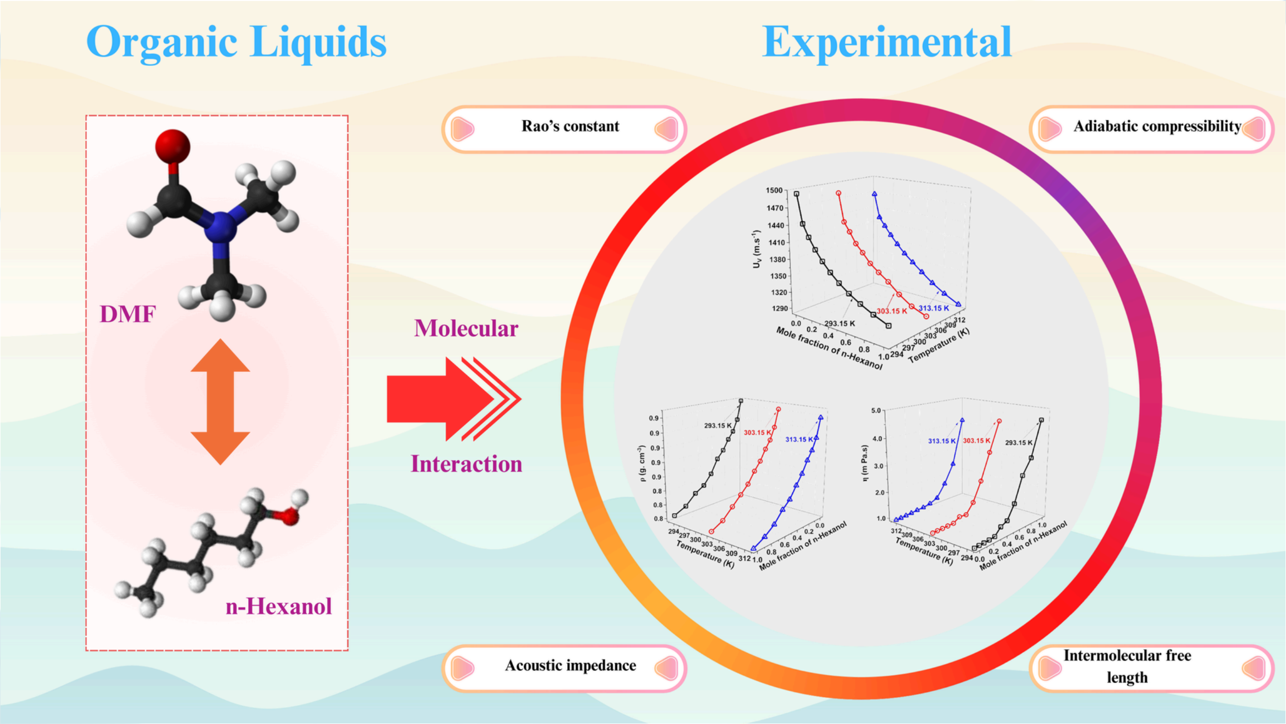
Chapter 6

Acoustic, Volumetric and IR spectra study of Binary Mixtures of n-Hexanol and N, N-Dimethylformamide

Contents



6.1 Introduction

In recent decades, studying the structure and interactions of molecules in liquid mixtures has become important for both science and engineering [1]. Liquid mixtures are used in many industries because of its flexibility in adjusting their properties by changing their composition and concentration. Mixing liquids creates new possibilities, such as the formation of complexes, molecular association, and dissociation, which do not occur in pure liquids. Significant efforts have been made to understand the behavior of liquid mixtures through both theoretical and experimental approaches [1-3]. Experimental investigations of the macroscopic and microscopic properties of liquid mixtures are essential, as they validate theoretical predictions and provide valuable information about the mixtures' physicochemical properties [4]. Various spectroscopic techniques, such as optical and ultraviolet (UV) spectroscopy, X-ray diffraction (XRD), infrared (IR) spectroscopy, nuclear magnetic resonance (NMR) and dielectric broadband spectroscopy (DBS) have been widely used to examine molecular interactions and physicochemical properties of liquid mixtures. Among these, ultrasonic velocity measurements at low frequencies (1–10 MHz) are particularly effective for detecting weak interactions in liquid mixtures [5-11]. Ultrasonic velocity, combined with density data, enables the direct estimation of thermo-acoustic parameters, which are challenging to determine through other methods [11]. Ultrasonic velocity measurements serve as a reliable method for investigating the molecular structure and physicochemical behavior of liquid mixtures. Furthermore, the analysis of phase equilibrium behavior and excess properties is fundamental for the optimization of separation processes and provides significant insights into the nature of molecular interactions [12]. The study of acoustical and thermodynamic properties of binary liquid mixtures is essential for many engineering calculations and practical applications, particularly in fields like mass transport [13-18]. These studies provide important insights into the molecular interactions within liquid mixtures. To better understand these interactions, it is more useful to study excess parameters rather than the actual property values. Excess properties, obtained from acoustical data, give information about molecular arrangement, compactness, and interaction strength in liquid mixtures. These parameters highlight the interactions between molecules, such as hydrogen bonding, dipole-dipole forces, and dipole-induced dipole interactions. Therefore, excess functions of acoustic properties are essential for understanding the behavior of binary mixtures [14,19]. Researchers have extensively studied the nature and strength of molecular interactions in liquid mixtures using these excess properties. Excess quantities from acoustic data are analyzed to understand differences in molecular size and the specific and non-specific interactions between the components. The evaluation of excess thermodynamic properties also provides insights into structural changes in liquids. These properties reveal details about molecular packing, movement, and interaction strength, which depend on the size, shape, and chemical nature of the molecules [20]. Understanding thermodynamic and transport properties of binary liquid systems is crucial for many industrial applications, such as process design, system optimization, and calculations for heat transfer, mass transfer, and fluid flow [21]. FTIR studies, integrated with thermo-acoustical parameters, offer a comprehensive understanding of molecular associations, packing arrangements, characteristic behaviors, and diverse intermolecular interactions in liquid mixtures. This combined approach enhances insights into the structural and dynamic properties of the mixtures, highlighting the complexities of molecular interactions [22]. Intermolecular hydrogen bonding, involving the sharing of hydrogen atoms between molecules, forms strong and stable connections. FTIR is a vital technique for analyzing and characterizing these interactions in binary mixtures, providing critical information on the nature and strength of hydrogen bonds [23]. This study is part of a research problem focused on systematically investigating the molecular interactions between the components of binary mixtures of amide and primary alcohols using physico-chemical, acoustic, and dielectric methods. The primary goal is to identify and characterize the nature of these molecular interactions. While several studies have explored the acoustic properties of amides with various solvents [24-26], there is no existing literature on the interaction between n-Hexanol and DMF.

In this chapter experimentally measured values of ultrasonic velocity (u), and Viscosity (η) over temperature range of 293.15 K to 313.15 K (interval of 10 K) are reported. From the experimental data the physiochemical (Acoustic and Volumetric) parameters such as adiabatic compressibility (β), intermolecular free length (L_f), acoustic impedance (Z), molar sound velocity (R), molar compressibility (B), relaxation time (τ), surface tension (σ) and thermal conductivity (K) were calculated, which are more useful to predict and confirm the molecular interaction in the binary liquid mixtures. Excess of measured acoustic and thermodynamic parameters have been estimated and fitted in Redlich -Kister polynomial [27]. Variations of these parameters are discussed in the light of molecular interaction in the mixture species. Their conformational study is supported by Fourier Transform Infrared (FTIR). The effect of concentration and temperature variation on the acoustical and thermodynamic parameters of the binary mixture of n-Hexanol and N, N-Dimethylformamide are investigated.

6.2 Material and Experimental Details

n-Hexanol (AR Grade) and N, N-Dimethylformamide (DMF) (AR grade) were procured from Loba Chemi Pvt Ltd, India, and used without further purification. Binary mixtures of n-Hexanol and DMF were prepared at eleven different volume-based concentrations. These concentrations were subsequently converted into the mole fraction of DMF using a Equation (3.1) (chapter 3) [28]. The ultrasonic velocity (Uv) of the binary mixtures was measured using a digital ultrasonic interferometer (Model F-05, Mittal Enterprises, New Delhi, India) with an accuracy of ±2 m/s. The viscosity (η) of the pure liquids and their binary mixtures was measured using an Ostwald viscometer. FTIR measurements of binary liquid mixtures of n-hexanol with DMF were carried out with an ALPHA (Bruker, Germany) FTIR spectrometer with precision of 0.05 cm^{-1}. The FTIR spectra were obtained in the region of 4000–400 cm^{-1}. All the measurements were carried out at three different temperatures and were controlled by the constant temperature water bath with an accuracy of ± 0.1 K. The experimentally determined ultrasonic velocity and viscosity values for pure n-Hexanol and N, N-Dimethylformamide (DMF) at various temperatures (293.15 K, 303.15 K and 313.15 K) were compared with corresponding literature data, as presented in Table 6.1. A satisfactory agreement was observed between the experimental and literature values.

Table 6.1 Comparison of experimental and literature values of ultrasonic velocity (Uv) and viscosity (η) for pure liquids (n-Hexanol and N, N-Dimethylformamide (DMF)) at different temperatures.

Parameters	Temperatures	n-Hexanol		DMF	
		Exp.	Lit.	Exp.	Lit.
(Uv)	293.15 K	1311.85	1314.40 [29]	1493.65	1492.00 [32]
	303.15K	1296.06	1288.20 [30]	1481.93	1467.20 [26]
	313.15 K	1287.90	1251.92 [31]	1466.86	1418.00 [33]
(η)	293.15 K	4.5900	4.7310 [34]	0.9200	0.9250 [37]
	303.15K	4.3210	3.8610 [35]	0.9198	0.8710 [38]
	313.15 K	4.1214	2.9340 [36]	0.9160	0.7480 [35]

6.3 Results and Discussion

6.3.1 Experimental and evaluated acoustic and volumetric study

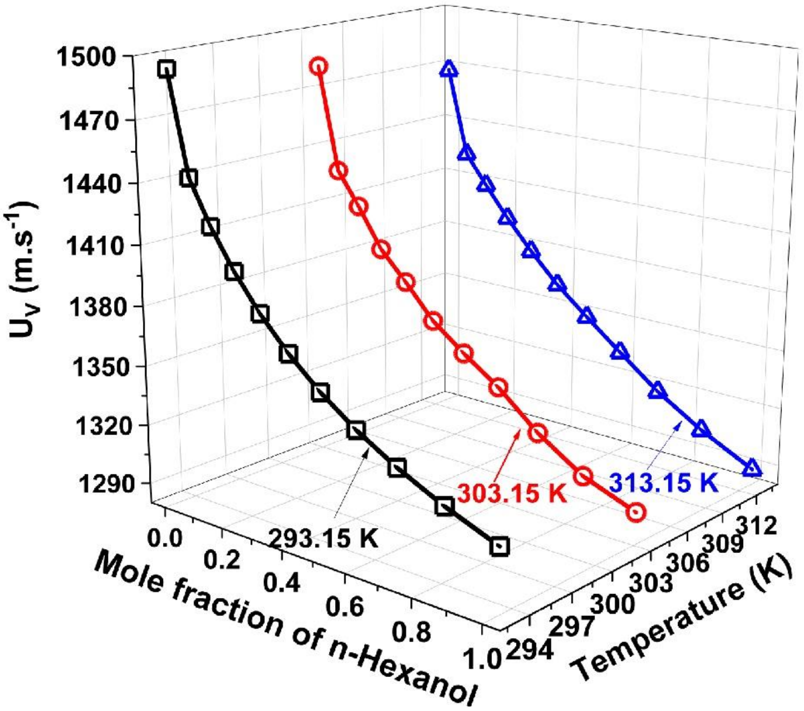


Fig. 6.1 Ultrasonic velocity (Uv) against mole fraction of n-Hexanol in DMF at different temperatures.

Ultrasonic velocity, an important acoustical parameter, provides valuable insights into the molecular interactions within the studied mixture solutions. Figure 6.1 illustrates the variation of ultrasonic velocity as a function of concentration and temperature for the binary mixture of n-Hexanol and N, N-Dimethylformamide (DMF). The nonlinear trend observed in ultrasonic velocity with changes in the mole fraction of n-Hexanol confirms the presence of molecular interactions between the components of the mixture. At 293.15 K, the ultrasonic velocity of n-Hexanol is 1311.85 m·s^{-1} and for DMF it is 1493.65 m·s^{-1}, aligning well with previously reported values [29,32]. An increase in n-Hexanol concentration leads to an increase in ultrasonic velocity. The nonlinear variation of velocity with the mole fraction of n-Hexanol suggests the formation of hydrogen-bonded complexes between the different molecules. Additionally, as the temperature increases, ultrasonic velocity decreases due to the disruption of both homo and hetero-molecular clusters and the impact of thermal agitation [39-41]. Furthermore, observed the decrease in ultrasonic velocity for the mixture compositions is attributed to dispersion forces or weak dipole-dipole interactions. Moses Ezhil Raj et al. [48] have similarly reported nonlinear behavior in ultrasonic velocity measurements for the binary mixture of dimethylformamide and methanol [33].

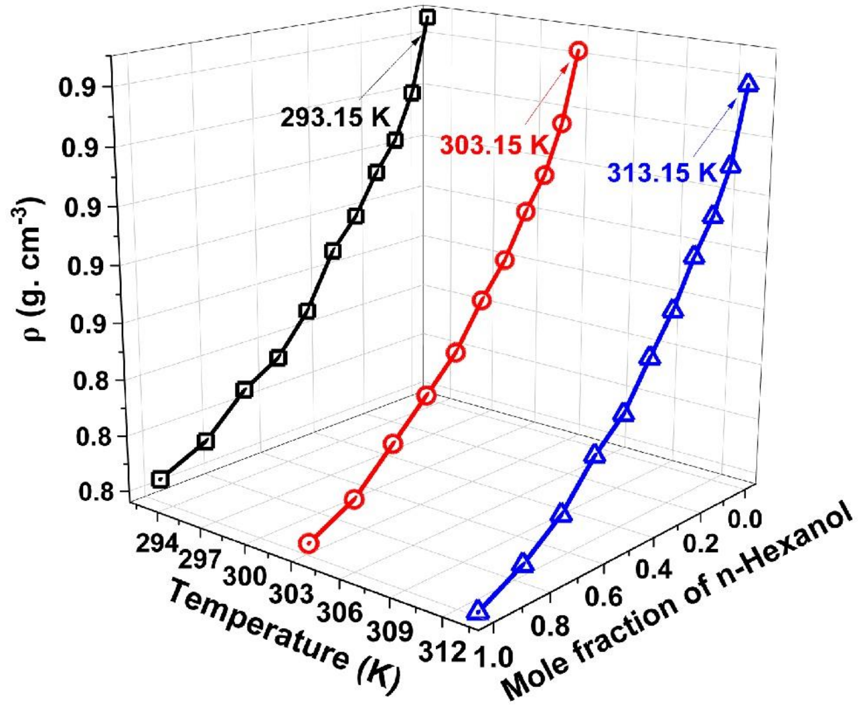


Fig. 6.2 Variation of density (ρ) against mole fraction of n-Hexanol in DMF at different temperatures.

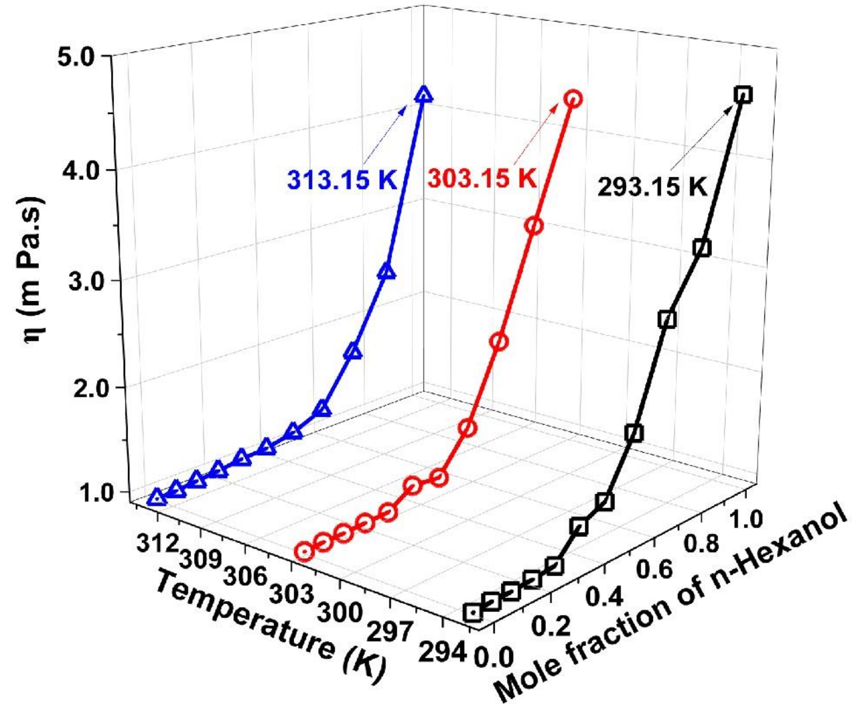


Fig. 6.3 Variation of viscosity (η) against mole fraction of n-Hexanol in DMF at different temperatures.

Figure 6.2 shows the density variation with the mole fraction of n-Hexanol for the mixtures at different temperatures (293.15 K, 303.15 K and 313.15K). The density shows a nonlinear decrease as the concentration of n-Hexanol increases across all measured temperatures. A decrease in density with increasing temperature is observed for all n-Hexanol and DMF mixtures. As the temperature increases, the molecular structure becomes less ordered, and the spacing between molecules increases, resulting in lower density [41]. Molecular interactions, including hydrogen bonding, dipole-dipole interactions, and complex formation, contribute to changes in the structural configuration of molecules. These interactions enable geometric fitting of molecules into each other's structures, despite differences in molecular size [42]. The strength of these molecular fittings significantly influences the viscosity of the solution. In the present study, the increase in viscosity with rising mole fraction of n-Hexanol in DMF at various temperatures is shown in Fig. 6.3. This plot shows that viscosity increases nonlinearly with an increase in the mole fraction of n-Hexanol, a trend observed consistently across all temperatures. Similar nonlinear variations or elevated viscosity values compared to ideal mixture behavior in polar compounds have been reported by Yue et al. [43] and Fort and Moore [44]. This nonlinear behavior suggests the presence of specific interactions among the components of the mixture. Additionally, the measured viscosity of n-Hexanol decreases from 4.5900 mPa.s to 4.1214 mPa.s as the temperature increases from 293.15 K to 313.15 K. This decrease in viscosity is consistently observed across all mixture compositions in the system.

The experimentally measured values of ultrasonic velocity (Uv) and density (ρ)were used to determine various acoustic parameters, including adiabatic compressibility (β), intermolecular free length (L_f), acoustic impedance (Z), molar sound velocity (R), and molecular compressibility (B). These parameters are described in Chapter 3 and were calculated using Equations (3.43) to (3.47) [45-48]. The calculated results are tabulated in Table 6.2. A close perusal of Table 6.2 indicates that as the concentration of n-Hexanol increase in the mixture, adiabatic compressibility (β), inter-molecular free length (L_f), molar sound velocity (R) and molar compressibility (B) increase while ultrasonic velocity (Uv) and acoustic impedance (Z) decreases with increase in temperatures. Tables 6.2 indicate that adiabatic compressibility increases (β) with the mole fraction of n-Hexanol and increasing temperature. The increase in adiabatic compressibility as the temperature increases suggests that there are strong molecular interactions within the mixture [42]. This trend is due to structural changes in the mixture that influence ultrasonic velocity. The observed changes in adiabatic compressibility suggest a slight contraction upon mixing, possibly due to complex formation [39]. The addition of interacting molecules disrupts existing molecular clusters, releasing dipoles that contribute to intermolecular interactions. This structural rearrangement enhances adiabatic compressibility, indicating the presence of specific molecular interactions. Similar trend have been reported in other liquid mixtures by Kannappan and Shanthi [49]. Table 6.2 shows that intermolecular free length (L_f) changes non-linearly with the mole fraction of n-Hexanol. The results show that L_f increases as the mole fraction of n-Hexanol increases, indicating that there are intermolecular interactions in the liquid mixture. Similarly, L_f also increases with temperature [33]. The trend of L_f is similar to that of compressibility (β). When compressibility decreases, the molecules pack more closely together, which causes a decrease in L_f. Additionally, L_f is important in explaining changes in ultrasonic velocity (U) in solutions. The relationship between L_f and Uv is explained by the sound propagation model of Eyring and Kincaid [50]. Acoustic impedance is closely related to the elastic properties of a medium, making it important to study how specific acoustic impedance changes with concentration and temperature [39]. Acoustic impedance shows a non-linear variation with mole fraction of n-Hexanol. Furthermore, it was observed that both molar sound velocity and molar compressibility increase non-linearly with the increasing mole fraction of n-Hexanol, as well as with rising temperature. These trends suggest a strong association between the solute and solvent molecules.

Table 6.2 The values of ultrasonic velocity (Uv), adiabatic compressibility (β), intermolecular free length (Lf), acoustic impedance (Z), Rao constant (R) and molar compressibility (B) for binary mixtures of DMF + n-Hexanol at different temperatures.

X_A	U_v (m/s)	$\beta \times 10^{-10}$ ($m^2.N^{-1}$)	$L_f \times 10^{-11}$ (m)	$Z \times 10^6$ ($Kg.m^{-2}.s^{-1}$)	$R \times 10^{-3}$ ($S^{1/3} \cdot mol^{-1}$)	$B \times 10^{-3}$ $m^3 \cdot Pa^{1/7} \cdot mol^{-1}$
T=293.15 K						
0.0000	1493.65	4.73	4.43	1.41	0.88	1.66
0.0633	1444.02	5.22	4.65	1.33	1.51	2.83
0.1319	1422.96	5.47	4.77	1.29	2.21	4.13

0.2066	1404.05	5.68	4.86	1.25	2.96	5.54
0.2883	1387.01	5.92	4.96	1.22	3.82	7.12
0.3780	1371.55	6.13	5.05	1.19	4.75	8.86
0.4769	1357.48	6.40	5.16	1.15	5.87	10.90
0.5864	1344.60	6.64	5.25	1.12	7.09	13.15
0.7085	1332.79	6.82	5.32	1.10	8.42	15.60
0.8454	1321.90	7.05	5.41	1.07	10.00	18.49
1.0000	1311.85	7.22	5.48	1.06	11.73	21.67
T=303.15 K						
0.0000	1481.93	4.83	4.56	1.40	0.88	1.66
0.0633	1430.41	5.33	4.79	1.31	1.51	2.83
0.1319	1414.11	5.56	4.89	1.27	2.21	4.13
0.2066	1394.52	5.78	4.99	1.24	2.97	5.54
0.2883	1380.53	6.00	0.00	1.21	3.83	7.14
0.3780	1363.97	6.24	5.18	1.18	4.78	8.89
0.4769	1351.36	6.47	5.28	1.14	5.87	10.90
0.5864	1338.76	6.68	5.36	1.12	7.07	13.12
0.7085	1321.22	6.96	5.48	1.09	8.43	15.62
0.8454	1306.11	7.25	5.59	1.06	10.00	18.49
1.0000	1296.06	7.44	5.66	1.04	11.74	21.69
T=313.15 K						
0.0000	1466.86	4.95	4.70	1.38	0.89	1.66
0.0633	1422.63	5.42	4.92	1.30	1.52	2.84
0.1319	1407.34	5.63	5.01	1.26	2.21	4.14
0.2066	1391.58	5.84	5.10	1.23	2.98	5.56
0.2883	1376.12	6.08	5.21	1.20	3.85	7.17
0.3780	1360.84	6.31	5.31	1.16	4.81	8.94
0.4769	1346.87	6.56	5.41	1.13	5.90	10.96
0.5864	1332.07	6.78	5.50	1.11	7.09	13.16
0.7085	1315.89	7.08	5.62	1.07	8.49	15.71
0.8454	1301.38	7.33	5.72	1.05	10.03	18.54
1.0000	1287.9	7.56	5.81	1.03	11.76	21.71

From Table 6.3, it is clear that the experimental values of ultrasonic velocity (Uv) and viscosity (η) were used to determine various thermodynamic parameters, including free volume (V_f), surface tension (σ) and thermal conductivity (K). These parameters are described in Chapter 3 and were calculated using Equations (3.49) to (3.52) [51-54]. Viscosity is a transport property related to the transfer of momentum between moving particles in the flow of liquids [55]. The variation in viscosity of binary mixtures with the mole fraction of n-Hexanol is presented in Table 6.3 at different temperatures. The nonlinear increase in viscosity with rising mole fraction suggests specific interactions between unlike molecules, which may lead to hydrogen bond formation and charge transfer complexes, resulting in a higher viscosity in the mixtures compared to the pure components [56]. The observed relaxation time (τ) increases with the concentration of n-Hexanol in DMF across all studied temperatures. This trend indicates that viscous forces play a significant role in the relaxation process. The strong interactions between the molecules in the binary liquid mixtures lead to an increase in viscosity, which consequently enhances the relaxation time. This behavior suggests molecular rearrangement through cooperative processes and the strengthening of hydrogen bonds [42]. Free volume is a critical parameter for understanding variations in the physico-chemical properties of liquids and their mixtures. It provides a quantitative measure of the cohesive or binding forces between solute and solvent molecules, offering insights into the molecular interactions and packing efficiency within the system. The observed non-linear decrease in V_f with increasing mole fraction of n-Hexanol highlights the influence of composition on the mixture's behavior. Additionally, the physicochemical properties of the liquid mixture at a fixed temperature can be effectively analyzed through Vf data, as it reflects variations in constituent concentrations. The free volume value is primarily determined by molecular characteristics, such as size and shape, and the nature of intermolecular interactions [48]. The surface tension values of the studied system decrease with an increasing mole fraction of n-Hexanol and further diminish with rising temperature, reflecting the influence of molecular composition and thermal energy on intermolecular interactions. This means that interactions in the mixture are strong and hence rise in the σ values have been observed when mole fraction increases. It also establishes the decrease in surface tension with respect to increase in temperature supports the basic nature of surface tension about formation of interface with the migration of species having high surface tension liquid state and low surface tension vapor state [57]. The determination of thermal conductivity (K) is vital for evaluating the suitability of fluid or nanofluid in various heat transfer applications, as it reflects the fluid's heat conduction efficiency and plays a key role in optimizing heat transfer systems. The observed decrease in thermal conductivity with increasing n-Hexanol concentration and rising temperature suggests a significant influence of intermolecular interactions between the constituent molecules [58].

Table 6.3 The values of viscosity (η), relaxation time (τ) free volume (V_f) surface tension (σ) and thermal conductivity (K) for binary mixtures of DMF + n-Hexanol at different temperatures.

X_A	η (mPa.s)	τ (ps)	$V_f \times 10^{-7}$ $m^3.mol^{-1}$	$\sigma \times 10^{-2}$ N/m	K ($W.m^{-1}.K^{-1}$)
T=293.15 K					
0.0000	0.9200	0.5806	1.46	3.50	0.2271
0.0633	0.9329	0.6487	1.41	3.23	0.1522
0.1319	0.9316	0.6793	1.44	3.10	0.1162
0.2066	0.9420	0.7138	1.45	3.01	0.0939
0.2883	0.9620	0.7593	1.44	2.90	0.0781
0.3780	1.2104	0.9891	1.05	2.82	0.0666
0.4769	1.3219	1.1284	0.95	2.71	0.0571
0.5864	1.8340	1.6225	0.61	2.63	0.0498
0.7085	2.7601	2.5085	0.34	2.57	0.0439
0.8454	3.3014	3.1023	0.28	2.50	0.0388
1.0000	4.5900	4.4215	0.18	2.45	0.0345
T=303.15 K					
0.0000	0.9199	0.5929	1.44	3.44	0.2245
0.0633	0.9314	0.6617	1.39	3.18	0.1505
0.1319	0.9315	0.6899	1.43	3.06	0.1152
0.2066	0.9400	0.7250	1.44	2.96	0.0930
0.2883	0.9460	0.7574	1.46	2.87	0.0775
0.3780	1.1006	0.9151	1.20	2.78	0.0659
0.4769	1.0681	0.9213	1.30	2.69	0.0568
0.5864	1.4402	1.2830	0.87	2.62	0.0496
0.7085	2.1721	2.0169	0.49	2.53	0.0434

0.8454	3.1940	3.0869	0.29	2.44	0.0382
1.0000	4.3210	4.2862	0.19	2.39	0.0340
T=313.15 K					
0.0000	0.9160	0.6050	1.43	3.37	0.2216
0.0633	0.9231	0.6668	1.40	3.13	0.1491
0.1319	0.9420	0.7073	1.39	3.03	0.1143
0.2066	0.9614	0.7481	1.38	2.94	0.0925
0.2883	0.9909	0.8028	1.36	2.84	0.0770
0.3780	1.0051	0.8454	1.37	2.75	0.0655
0.4769	1.0647	0.9311	1.30	2.66	0.0564
0.5864	1.1894	1.0759	1.15	2.58	0.0492
0.7085	1.6771	1.5826	0.71	2.49	0.0430
0.8454	2.3988	2.3443	0.44	2.42	0.0380
1.0000	4.1214	4.1542	0.20	2.36	0.0337

6.3.2 Excess parameters study

To gain probe into the molecular interactions within liquid mixtures, it is more informative to analyze these interactions using excess parameters rather than actual values. Non-ideal liquid mixtures often exhibit significant deviations from linearity with respect to concentration, which are attributed to the presence of strong or weak intermolecular interactions. The degree of deviation is influenced by the nature of the individual components and the composition of the mixtures [59]. Excess ultrasonic velocity $(Uv)^E$ for binary mixtures of n-Hexanol+DMF at three different temperatures (293.15 K, 303.15K and 313.15K) along with the Redlich–Kister correlation is plotted in Fig. 6.4. The analysis of the figure indicates that the excess ultrasonic velocity demonstrates negative deviations, which systematically diminish with increasing mole fraction of n-Hexanol in the mixture (n-Hexanol+DMF), consistent across all investigated temperatures. Negative excess ultrasonic velocity for all concentration ranges $(0.0\rightarrow1.0)$ values suggest weak interactions between the different types of molecules in the mixtures, indicating the dominance of long-range dispersive forces [60]. A similar temperature dependence of negative ultrasonic velocity has also been reported in the case of ethylene glycol monophenyl ether and 1-hexanol by Rana et al. [61].

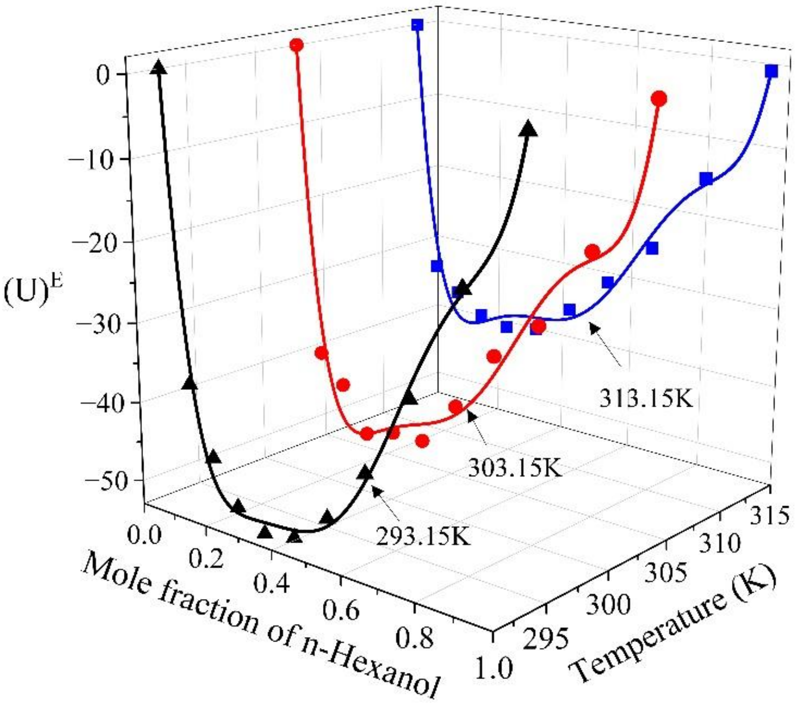


Fig.6.4 Excess ultrasonic velocity (U)^E against mole fraction of n-Hexanol at different temperatures.

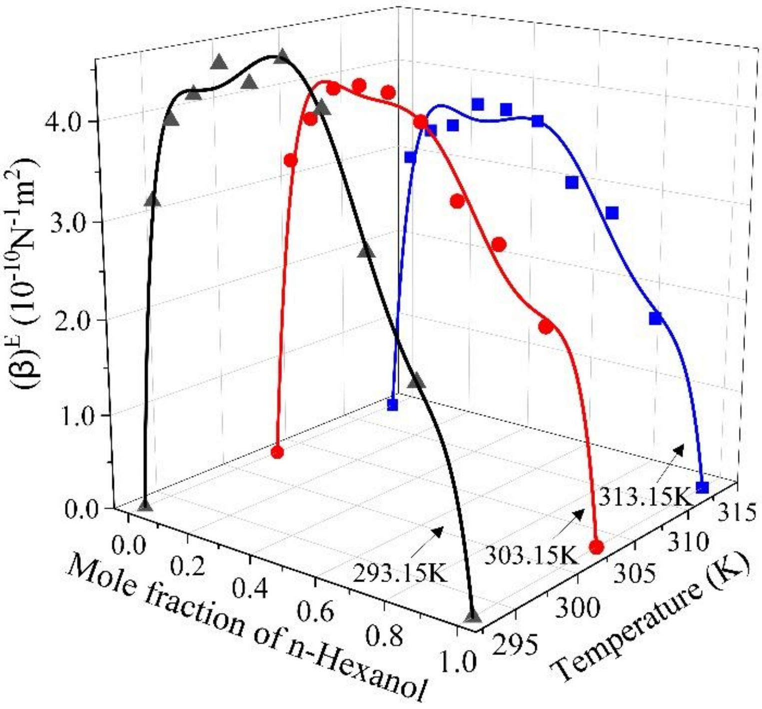


Fig.6.5 Excess adiabatic compressibility (β)^E against mole fraction of n-Hexanol at different temperatures.

Furthermore, the negative deviation and non-linear dependence indicate the presence of weak interactions between the components of the mixture. The negative excess velocity suggests structural deformation, where weak interactions among the mixture's components cause the molecular aggregates to deform, leading to a looser-packed structure. As a result, sound travels faster through the mixture via longitudinal waves, causing the speed of sound to exhibit negative behavior compared to the expected linear trend [62]. Figure 6.5 shows the positive deviation of excess adiabatic compressibility $(\beta)^E$ entire composition range (0.0→1.0~ mole fraction of n-Hexanol) at different temperatures respectively. In the present investigation, the positive contribution of $(\beta)^E$ indicates the presence of dispersive forces. Additionally, the decreasing trend of $(\beta)^E$ values with increasing mole fraction of n-Hexanol and rising temperature suggests the existence of weak interactions within the mixtures. Excess properties arise from a combination of physical and chemical contributions. Physical contributions, such as dispersion forces and weak dipole-dipole interactions, predominantly result in positive values of $(\beta)^E$ [14]. Chemical contributions involve the disruption of molecular associations present in the pure components, which also contribute to positive $(\beta)^E$ values. The positive excess values are indicative of weak intermolecular interactions [63]. Fort and Moore have established that such positive excess values signify an increase in the interaction strength between unlike molecules [64]. Consequently, the analysis of excess functions provides critical insights into the molecular interactions within binary systems, making them essential for understanding the underlying physicochemical behavior. Figure 6.6 shows the excess intermolecular free length $(L_f)^E$ against mole fraction of n-Hexanol at different temperatures (293.15 K, 303.15 K and 313.15K). Fort et al. attributed positive values of excess free length $(L_f)^E$ to dispersive forces, whereas negative values were associated with interactions such as charge transfer, dipole-induced dipole, and dipole-dipole interactions [64]. In the present study, the observed positive $(L_f)^E$ values suggest the predominance of dispersive forces between the components in the mixture. This indicates a looser molecular packing, resulting in reduced intermolecular cohesion and weaker molecular associations [65]. Figure 6.7 shows the changes in excess acoustic impedance $(Z)^E$ at different temperatures. The negative $(Z)^E$ values across all mole fractions of n-Hexanol indicate weaker interactions between the molecules in the mixture. A similar trend of negative $(Z)^E$ values was reported by Mahajan et al. for binary liquid mixtures of n-octane, n-decane, n-dodecane, and n-tetradecane with octan-2-ol. This behavior was attributed to weak interactions between the constituent molecules [66].

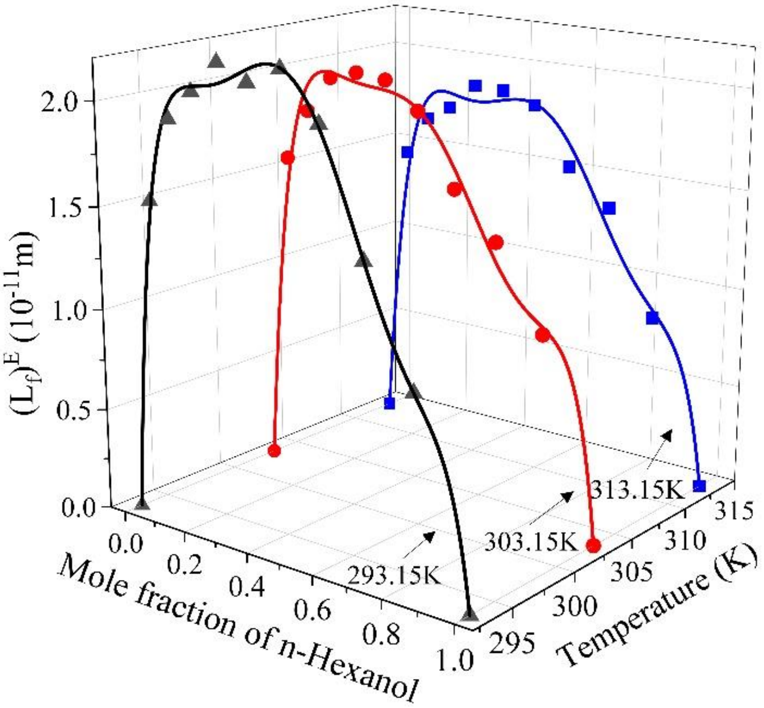


Fig.6.6 Excess intermolecular free length $(L_f)^E$ against mole fraction of n-Hexanol at different temperatures.

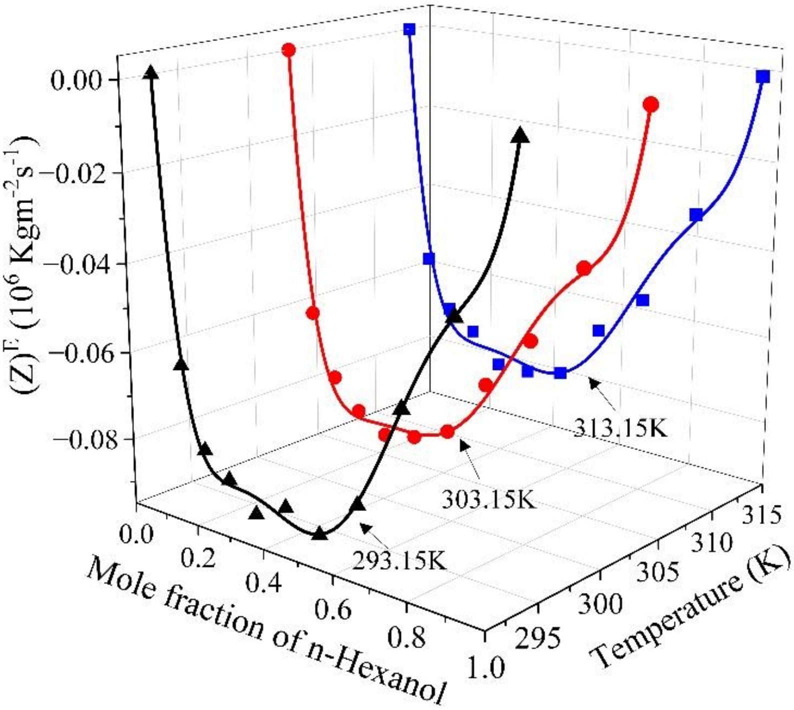


Fig.6.7 Excess acoustic impedance $(Z)^E$ against mole fraction of n-Hexanol at different temperatures.

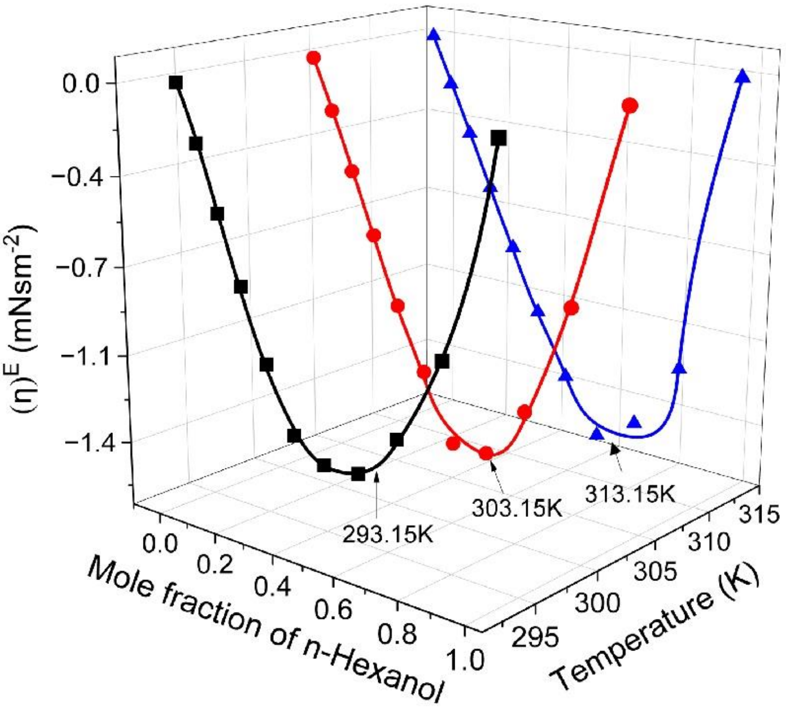


Fig. 6.8 Plot of excess viscosity $(\eta)^E$ against mole fraction of n-Hexanol in binary mixtures at different temperatures.

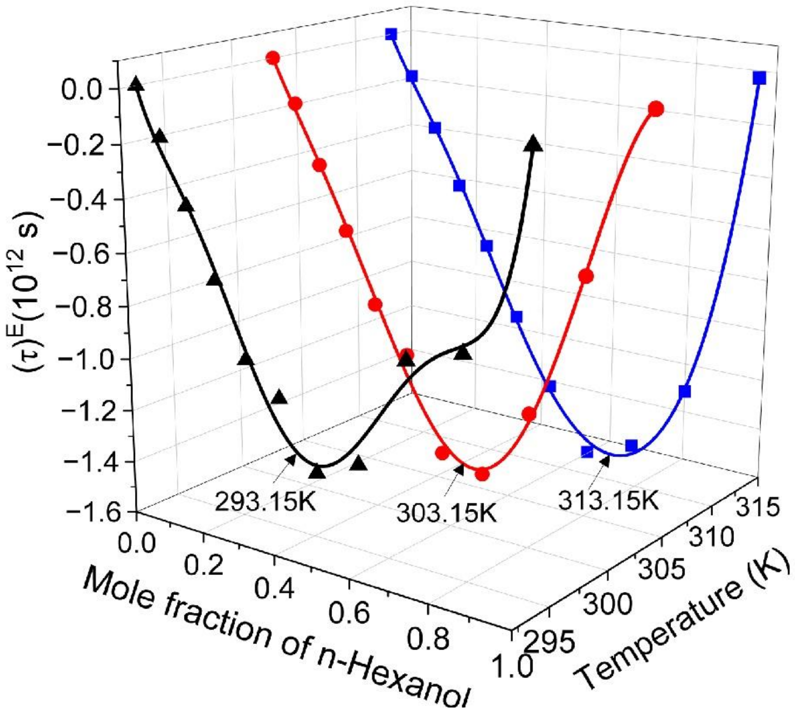


Fig. 6.9 Plot of excess relaxation time $(\tau)^E$ against mole fraction of n-Hexanol in binary mixtures at different temperatures.

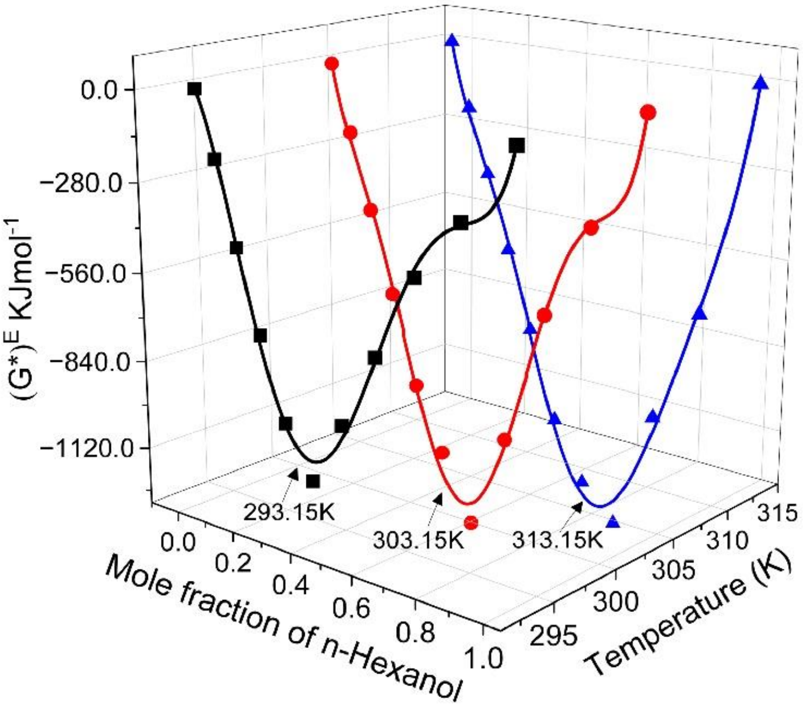


Fig. 6.10 Plot of excess Gibbs free energy $(G^*)^E$ against mole fraction of n-Hexanol in binary mixtures at different temperatures.

Figure 6.9 show the excess relaxation time $(\tau)^E$ negative deviation for all composition range (0.0→1.0) at studied temperatures. In general, relaxation time is influenced by the viscosity of the liquid. It follows a similar trend as viscosity, indicating a close relationship between the two properties. Relaxation times on the order of 10^{-12} seconds are associated with structural relaxation processes. In such cases, it is suggested that molecular rearrangements occur through a cooperative mechanism, where groups of molecules adjust their positions collectively [67]. The negative $(\tau)^E$ values observed for the DMF and n-Hexanol mixtures suggest increased molecular dynamics of DMF and n-Hexanol within their hydrogen-bonded, heterogeneous networks. This indicates that the interaction between the molecules promotes more significant molecular mobility within the system [48]. Figure 6.10 shows the variation of excess Gibbs free energy of activation for viscous flow $(G^*)^E$ as a function of the mole fraction of n-Hexanol at different temperatures. This parameter provides insight into molecular orientation hindrance, which reflects the extent of short-range intermolecular interactions within the liquid mixture. Across the entire composition range (0.0→1.0), the observed negative values of $(G^*)^E$ suggest weak interactions between the dissimilar molecules in the solvent mixture. This study found that the excess Gibbs free energy $(G^*)^E$ values were negative and became more negative as the temperature increased. This means that when the temperature rises, the energy added to the system is quickly turned into movement of the liquid molecules. This movement is linked to their energy and activity within the mixture. These results suggest that the molecules in the system interact with each other weakly, meaning the forces between them are not very strong [62]. The evaluated excess parameters values such as (U^E), (β^E), (L_f^E), (Z^E), (η^E), (τ^E) and (G^{*E}) [17,33] were fitted to the Redlich-Kister polynomial equation [27] using the least squares method, with equal weighting for all data points. The coefficients (a_0), (a_1), (a_2) and (a_3), along with the correlation coefficients (δ), are tabulated in Table 6.4.

Table 6.4 Value of coefficients of R. K. Polynomial, correlation coefficients (R) for excess ultrasonic velocity$(u)^E$, excess adiabatic compressibility$(\beta)^E$, excess intermolecular free length $(L_f)^E$, excess acoustic impedance $(Z)^E$, excess viscosity $(\eta)^E$, excess relaxation time $(\tau)^E$ and excess Gibb's free energy of activation $(G^*)^E$ at different temperatures.

Parameters	Temperatures	a_0	a_1	a_2	a_3	R
$(u)^E$	293.15 K	-197.730	98.100	42.940	172.290	0.9945
	303.15 K	-167.890	95.130	21.370	132.920	0.9821
	313.15 K	-141.130	60.130	46.430	161.580	0.9841
$(\beta)^E$	293.15 K	19.1100	-4.4700	-10.9900	-16.5700	0.9967
	303.15 K	15.4200	-7.2200	-2.7800	-7.5300	0.9881
	313.15 K	14.3800	-3.5100	-5.2000	-13.5900	0.9814
$(L_f)^E$	293.15 K	8.9300	-2.5300	-4.5700	-7.7600	0.9970
	303.15 K	7.5700	-3.5800	-1.3900	-4.3000	0.9898
	313.15 K	7.1800	-2.0600	-2.5300	-6.5500	0.9849
$(Z)^E$	293.15 K	-0.3652	0.1146	0.1814	0.3299	0.9973
	303.15 K	-0.3180	0.1485	0.0639	0.2045	0.9947
	313.15 K	-0.3066	0.1048	0.1031	0.2411	0.9901
$(\eta)^E$	293.15 K	-5.4697	-0.4973	0.4182	-2.026	0.9995
	303.15 K	-5.8371	-2.0672	1.857	1.0449	0.9992
	313.15 K	-5.7112	-4.0134	-1.429	-0.2738	0.9990
$(\tau)^E$	293.15 K	-4.9900	1.1200	5.6300	-5.1100	0.9861
	303.15 K	-5.6800	-2.5600	3.3500	3.4400	0.9960
	313.15 K	-5.7300	-4.0600	-0.1800	-1.1000	0.9987

$(G^*)^E$	293.15 K	-3612.77	4163.68	1332.62	-5169.10	0.9971
	303.15 K	-5026.31	1899.64	3893.87	-1531.90	0.9922
	313.15 K	-5841.20	-1842.20	2501.50	1353.50	0.9964

6.3.3 FTIR study

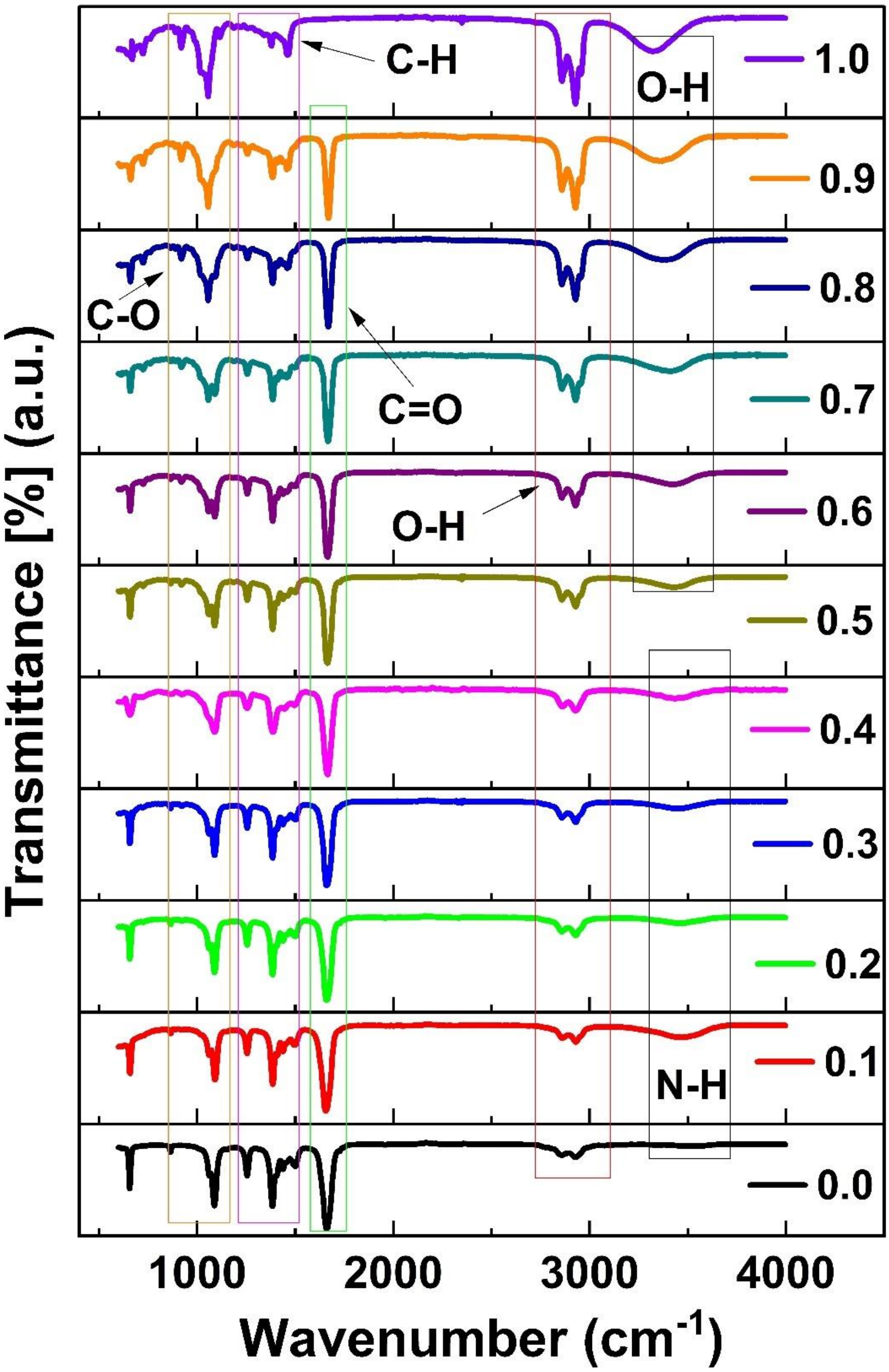


Fig. 6.11 Variation in IR spectrum for n-Hexanol+DMF binary liquid mixtures.

FTIR spectroscopy is performed to provide an enhanced understanding of the interactions in specific binaries as predicted by acoustic study. The spectra are taken for pure liquid and liquid mixtures of n-Hexanol+DMF for the entire composition range (0.0→1.0) at room temperature. The IR spectra of n-hexanol with DMF binary liquid mixtures for all the concentrations (0.0→1.0) are shown in Fig. 6.11. Here, an increase in the intensity of the O–H peak at 3500 cm^{-1} is observed as a function of n-hexanol concentration. [68,69] The second peak of the carbonyl group (C=O) is characteristic of amides, for which a strong absorption band is observed in the region of 1500-1900 cm^{-1} due to the C=O stretching vibration. It is observed in Fig. 6.10 and table 6.5 that in the concentration range $0.0 \leq XA \leq 0.4$, a small-magnitude N–H peak is present, whereas a broad O–H peak is clearly seen. The wavenumber of the stretching vibration increases if the surrounding groups increase the double-bond nature of the carbonyl group. The absorption spectra of these substances in solutions, therefore, contain characteristic bands of N–H and O–H vibrations of the monomer and the associated molecules. [70] The intensity of the C–O stretching peak at around 1000-1200 cm^{-1} and C–H stretching at around 1200-1400 cm^{-1} is decreased at lower DMF concentrations.

Table 6.5 FT-IR spectra functional group analysis of pure n-Hexanol and DMF and their binary mixtures (0.0→1.0) at room temperature.

X_A	C-H Bond		C-O Bond		C-H Bond		C=O Bond	
	ν (cm^{-1})	T. (%) (a.u)	ν (cm^{-1})	T. (%) (a.u)	ν (cm^{-1})	T. (%) (a.u)	ν (cm^{-1})	T. (%) (a.u)
0.0	657.6	0.5147	1088.4	0.3289	1383.2	0.3211	1659.4	0.0815
0.1	657.6	1.4947	1094.6	1.4423	1387.3	1.3844	1655.3	1.1070
0.2	657.6	2.5028	1090.5	2.3617	1383.2	2.3450	1657.3	2.0749
0.3	657.6	3.4668	1090.5	3.3498	1385.2	3.3220	1657.3	3.0510
0.4	659.6	4.0800	1090.5	4.0558	1389.4	4.0563	1667.6	3.9971
0.5	659.6	4.6179	1090.6	4.5422	1387.3	4.5246	1665.6	4.2112
0.6	659.6	5.4644	1090.5	5.4162	1385.2	5.3791	1659.4	5.1003
0.7	659.6	6.0962	1059.5	6.0627	1389.4	6.0780	1667.6	5.8946
0.8	661.7	6.5917	1059.5	6.4970	1381.1	6.6039	1665.6	6.3481
0.9	663.8	7.1153	1057.5	6.9802	1385.2	7.1227	1673.8	6.9470

1.0	663.8	7.6344	1057.5	7.4561	1385.2	7.7113	-	-

X_A	**O-H Bond**		**O-H Bond**		**N-H Bond**	
	ν (cm^{-1})	T. (%) (a.u)	ν (cm^{-1})	T. (%) (a.u)	ν (cm^{-1})	T. (%) (a.u)
0.0	2929.2	0.8532	-	-	3586.8	0.9815
0.1	2931.7	1.8015	-	-	3465.1	1.8491
0.2	2927.1	2.7478	-	-	3478.6	2.8952
0.3	2931.3	3.6835	-	-	3446.6	3.8188
0.4	2931.3	4.0864	-	-	3423.9	4.1033
0.5	2931.3	4.7143	-	-	3430.1	4.8821
0.6	2929.2	5.5103	3423.9	5.6900	-	-
0.7	2933.3	6.0698	3415.7	6.1770	-	-
0.8	2927.1	6.4909	3368.3	6.7146	-	-
0.9	2929.2	6.9754	3351.8	7.2026	-	-
1.0	2925.1	7.4286	3320.9	7.6552	-	-

X_A* Volume Fraction of n-Hexanol, T* Transmittance [%] and ν* Wavenumber cm^{-1}

Conclusion

- The ultrasonic velocity, density, and viscosity of n-Hexanol and, N,N-Dimethylformamide (DMF) mixtures were experimentally measured across the entire composition range (0.0→1.0) at different temperatures of 293.15, 303.15 and 313.15. Good agreement was found between the experimental and literature values for the ultrasonic velocity and viscosity of the n-hexanol and DMF mixture at studied temperatures.
- The nonlinear variation in experimental measured value of U, ρ, η for n-Hexanol and DMF mixtures at all studied temperatures indicates the presence of specific interactions between the components of the mixture.
- The observed trends and variations in acoustic and thermodynamic parameters with the molar concentration of n-Hexanol offer valuable insights into the nature of intermolecular forces present in the mixture.

- From the experimental data, various physico-chemical parameters, including adiabatic compressibility (β), intermolecular free length (Lf), acoustic impedance (Z), molar sound velocity (R), molar compressibility (B), relaxation time (τ), surface tension (σ), and thermal conductivity (K), were calculated. The analysis of these parameters indicates strong intermolecular interactions between the components, primarily due to dipole-dipole and dipole-induced dipole interactions facilitated by hydrogen bonding.
- The positive and negative variations in the excess values of acoustic parameters with changes in concentration and temperature confirm the presence of interactions between unlike molecules. Infrared spectral analysis further revealed the dominant role of N, N-dimethylformamide over n-Hexanol, highlighting its ability to bind molecules together effectively.
- The variations in position and intensity of the IR bands related to the functional groups indicated the molecular association in the liquid mixtures.

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