IJCRT.ORG ISSN : 2320-2882

INTERNATIONAL JOURNAL OF CREATIVE RESEARCH THOUGHTS (IJCRT)
An International Open Access, Peer-reviewed, Refereed Journal

Thermodynamic Investigations In Binary Mixture Of O-Toluidine With Amides At Atmospheric Pressure

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Abstract— Excess molar volume (V^E), excess isentropic compressibility (K_s^E), excess enthalpy(H^E), excess free length ($L_f{}^E$) and excess Gibbs free energy of activation of viscous flow (ΔG^{*E}) for binary mixtures of O-Toluidine (O-TA) with Formamide (F), N-Methylformamide (NMF) and N,N-Dimethylformamide (N,N-DMF) at selected compositions were determined from the measured values of densities, speeds of sound, and viscosities of pure components and their mixtures at 303.15-313.15 K at ambient atmospheric pressure. The excess molar volumes, excess isentropic compressibility, excess enthalpy, excess free length and excess Gibbs energy of activation of viscous flow have been analyzed in terms of interactions arising due to structural effect, charge-transfer complexes and dipole–dipole interaction between unlike molecules. The excess parameters have been fitted to the Redlich–Kister polynomial equation using multi-parametric nonlinear regression analysis to derive the binary coefficients and to estimate the standard deviation.

Index Terms— Speed of sound, viscosity, density, excess parameters and polynomial equation.

1 INTRODUCTION

The dynamic viscosity (η), density (ρ) and speed of sound (U) of binary or ternary liquids are indispensable in most fluid mechanics, solution theory, molecular thermodynamics, and various methodical uses [1], [2], [3] These values carry a significant role to elucidate the behavior of liquids and their mixtures. The analysis of excess functions and deviations from ideality is essential to interpret the interactions in the mixing process. Therefore, the assessment and forecast of these mixtures as functions of temperature and composition are of noticeable importance. Researchers are highly devoted to discovering the real causes of possible interaction of organic molecules in binary mixtures using physical property data [4], [5], [6], [7], [8] We display ρ and η values in the pure state and for their binary systems of O- Toluidine (O-TA) with amides, formamide (F), N-methylformamide (NMF), and N,N-dimethylformamide (N,N-DMF) at T = (303.15 -313.15) K and atmospheric pressure over the entire range of composition. The experimental ρ, η and U data are used to calculate the excess molar volume (V^E), excess isentropic compressibility (K_s^E), excess enthalpy(H^E), excess free length($L_f{}^E$) and excess Gibb's free energy of activation of viscous flow (ΔG^{*E}). The Redlich−Kister equation[12] is used to fit the excess values in order to obtain the binary coefficients and standard deviation. All liquids are used for laboratory and industrial purposes. Here, the O- Toluidine liquid (O-CA) has been considered as component 1, while the other amides liquids F, NMF, and N,N-DMF have been considered as component 2. [9], [10], [11].

This work is part of our program to give information/data for the characterization of molecular interactions between solvents in binary systems. The liquids were chosen for the present study on the basis of their medical and Industrial importance. O- Toluidine is used an Intermediate in the synthesis of the large-volume herbicides, metolachlor and acetochlor, pigments, pesticides, pharmaceuticals and also used in the clinical laboratory as an ingredient in a reagent for glucose analysis. On the other hand, Formamide is an amide derived from formic acid. it is used as a feedstock in the manufacture of formate esters, as an ionizing solvent, as an RNA stabilizer in gel electrophoresis, and in tissue preservation. More intriguingly, it may be a key compound in the origin of life on Earth. *N*-Methylformamide (NMF) is closely related to other formamides, not ably formamide and N, N-dimethylformamide (N, N-DMF).However, industrial use and production of NMF are far less than for either of these other formamides. N, N-DMF is favored over NMF as a solvent due to its greater stability.NMF is mainly used as a reagent in various organic syntheses with limited applications as a highly polar solvent. *N*, *N*-Dimethylformamide (N, N-

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Chemical Name	Source	Initial mole fraction	Purification method	Final mole fraction	Analysis method
O-Toluidine	Hi Media Labs Pvt. Ltd. Mumbai, India.	0.99	Distillation	0.997	Gas liquid chromatography
Formamide		0.99	Distillation	0.998	
NMF		0.99	Distillation	0.998	
DMF		0.99	Distillation	0.998	

DMF) Dimethylformamide is odorless whereas technical grade or degraded samples often have a fishy smell due to impurity of dimethylamine. Dimethylamine degradation impurities can be removed by sparging degraded samples with an inert gas such as argon or by sonicating the samples under reduced pressure. As its name indicates, it is a derivative of formamide, the amide of formic acid. N, N-DMF is a polar (hydrophilic) aprotic solvent with a high boiling point. It facilitates reactions that follow polar mechanisms, such as S_N2 reactions [12], [13]. In the present study, our focus is on the study of liquid mixtures of substituted amides with O-Toluidine because there have been a few studies on these mixtures. It is expected that there will be a significant degree of H-bonding in these binary mixtures we report the densities, speeds of sound and viscosities for three binary systems (O-Toluidine with Formamide, N-Methylformamide and N, N-Dimethylformamide) at T = (303.15–313.15) K ambient atmospheric pressure. The experimental data has been used to compute the excess parameters. The results are used to qualitatively discuss specific interactions between unlike molecules.

2 PROCEDURE FOR PAPER SUBMISSION

2.1 MATERIALS AND METHODS

Chemicals used in the present study are O- Toluidine (Sigma–Aldrich), Formamide, N-Methylformamide and N, N-Dimethylformamide (Sigma–Aldrich). These chemicals were purchased from Hi Media Laboratories Pvt. Ltd. Mumbai. These chemicals were further purified by standard methods [14] like distillation and fractional distillation under reduced pressure, and only the middle fractions were collected. Before use, the chemicals were stored over 0.4 nm molecular sieves for about 72 h to remove water and gas. The purity of the liquid samples was checked by gas chromatography. The water contents were determined by the Karl–Fischer method. The details of the chemicals and purification methods are presented in Table1. All liquid mixtures were prepared by weighing an amount of pure liquids in an electronic balance with a precision of ±0.1 kg m^{-3}. The binary mixtures were prepared just before use. The uncertainty in mole fraction was estimated to be less than ±0.0001.The speed of sound was measured with an ultrasonic interferometer (Mittal Enterprises, Delhi, India) working at 2 MHz frequency and 303.15 - 313.15 K temperature. The uncertainty in the speed of sound was found to be ±0.1 ms^{-1}. An electronically digital operated constant temperature water bath has been used to circulate water through the double walled measuring cell made up of steel containing the experimental solution at the desire temperature [9].

Table 01: Purity analysis

The viscosities were measured with Ostwald viscometer. The viscometer was calibrated at each temperature using distilled water. The uncertainty in viscosity measurement is up to 0.001 mPa s. The flow time has been measured after the attainment of bath temperature by each mixture. The flow measurements were made with an electronic stop watch with precision of 0.01 s. For all pure components and mixtures, 3-4 readings were taken, and the average of these values was used in all the calculations. The densities of the pure compounds and their mixtures were determined accurately using 10 mL specific gravity bottles in digital electronic balance with an uncertainty of ±0.1 kg m^{-3}. The average uncertainty in the measured density was ±0.001 kg m^{-3}

3.Theory

The values of experimentally determined ρ, η and U for the liquid mixtures of O- Toluidine with Formamide, N-Methylformamide and N, N-Dimethylformamide at 303.15-313.15 K over the entire composition range are given in Table 2. The experimentally determined values of ρ, η and U, various thermodynamic parameters like excess isentropic compressibility (K_s^E), excess Enthalpy (H^E), excess molar volume (V^E), excess Gibb's free energy (ΔG^{*E}) and excess free length(L_f^E) were calculated. The excess values of isentropic compressibility, K_s^E were calculated as follows;

$$K_s^E = K_s - K_s^{id} \qquad (1)$$

Where K_s^E is its excess value, K_s^{id} is the ideal isentropic compressibility value and K_s represent the calculated value of isentropic compressibility for the mixture. K_s^{id} for an ideal mixture was calculated from the relation recommended by Kiyohara and Benson [15], [16] and Douheret et al [17]

$$K_s^{id} = \sum \phi_i \left\{ K_{s,i}^{\circ} + \frac{T V_i^{\circ} (\alpha_i^{\circ})^2}{C_{p,i}^{\circ}} \right\} - T\left(\sum x_i V_i^{\circ}\right) \left\{ \frac{(\sum \phi_i \alpha_i^{\circ})^2}{\sum x_i C_{p,i}^{\circ}} \right\} \qquad (2)$$

In which $K_{s,i}^{\circ}$, V_0^{i}, α_i^{o}, $C_{p,i}^{o}$ are the isentropic compressibility, molar volume, isobaric thermal expansion coefficient and molar isobaric heat capacity of pure component i, T represents temperature, ϕ_i is the volume fraction and x_i represents the mole fraction of i in the mixture.

Table 02: Comparison of experimental and literature values of density (ρ), viscosity (η) and speed of sound (U) of pure liquids.

Liquid	Temp T (K)	Density(ρ) Kg. m^3		Viscosity(n) mPa.S		Speed of sound (U)m.s^{-1}	
		expt	Lit	expt	Lit	expt	Lit
O-Toluidine	303.15	0.9903	0.9902[a]	3.4520	3.4520[a]	1577.8	1577.8[a]
	308.15	0.9961	0.9960[a]	3.1253	3.1253[a]	1558.4	1558.38[a]
	313.15	0.9816	0.9816[a]	2.8642	2.8642[a]	1539.6	1539.62[a]
Formamide	303.15	1.1238	1.1237[b]	2.8018	2.8018[b]	1585.4	1585.4[b]
	308.15	1.1195	1.1194[b]	2.4980	2.4980[b]	1580.5	1580.6[b]
	313.15	1.1155	1.1154[b]	2.2435	2.2435[b]	1572.0	1572.0[b]
NMF	303.15	0.9947	0.9946[b]	1.5860	1.5859[b]	1408.6	1408.5[b]
	308.15	0.9904	0.9903[b]	1.4628	1.4627[b]	1398.3	1400.6[b]
	313.15	0.9862	0.9861[b]	1.3521	1.3520[b]	1382.6	1382.5[b]
N, N-DMF	303.15	0.9388	0.9386[b]	0.7532	0.7485[b]	1467.2	1469.8[b]
	308.15	0.9346	0.9344[b]	0.7164	0.7063[b]	1433.4	1433.2[b]
	313.15	0.9294	0.9296[b]	0.6833	0.6683[b]	1418.6	1418.0[b]

a Neeti et al. [19], b Shamim Akhtar et al. [20]

The density values have been used to calculate the excess volumes, V^E, using the following equation,

$$V^E=\frac{X_1M_1+X_2M_2}{\rho}-\left(\frac{X_1M_1}{\rho_1}+\frac{X_2M_2}{\rho_2}\right) \quad (3)$$

Where ρ is the density of the mixture and X_1, M_1, and X_2, M_2, and ρ_1, ρ_2 are the mole fraction, molar mass, and density of pure components 1 and 2, respectively.

The excess values of the free length L_f^E were calculated by using the expression,

$$L_f^E=L_f-K_T(K_s^{id})^{1/2} \quad (4)$$

Where L_f represents the calculated value for the mixture and K_T represent a temperature dependent constant whose value is $K_T = (91.368+0.3565T)\ X10\text{-}8$.

Excess Gibbs free energy of activation ΔG^{*E} was calculated as follows,

$$\Delta G^{*E} = RT\left|ln\left(\frac{\eta V}{\eta_2 V_2}\right)-X_1\ln\left(\frac{\eta_1 V_1}{\eta_2 V_2}\right)\right| \quad (5)$$

Where R represents gas constant, T is absolute temperature, η is the viscosity of the mixture and η_1, η_2 are the viscosities of the pure compounds, V_m is the molar volume of the mixture and V_1, V_2 are the volumes of the R.

Excess enthalpy H^E was calculated from usual relation,

$$H^E=H-(X_1H_1+X_2H_2) \quad (6)$$

Where H represents the calculated value of enthalpy for the mixture and H_1, H_2 represent enthalpy of pure components 1 and 2, respectively.

The conventional smooth curve fitting strategy for excess properties of binary mixture involves Redlich-Kister polynomial non-linear regression,

$$Y^E=X_1(1-X_1)\sum_{i=1}^{n}A_i(2x_1-1) \quad (7)$$

Where $Y^E = K_s^E$, V^E, ΔG^{*E}, H^E. The values of coefficient A_i were determined by a regression analysis based on the least-squares method.

The standard deviation (σ) was calculated using the relation,

$$\sigma(Y^E)=[\sum(Y_{obs}^E\ Y_{cal}^E)^2/n\text{-}m]^{1/2} \quad (8)$$

Where n represents the number of experimental points and m is the number of adjustable parameters.

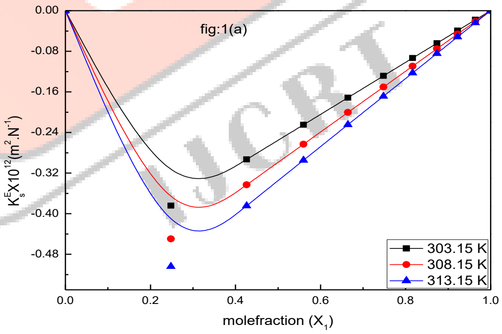
4 Results and Discussions

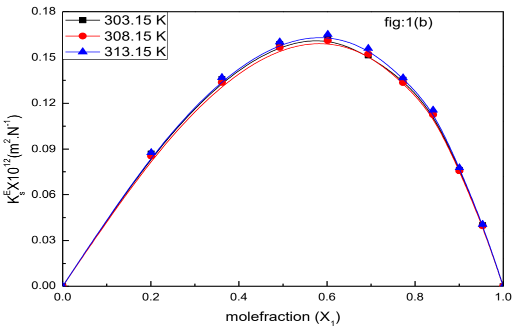
Figures 1–5 show the dependence of K^E_s, V^E, L_f^E, ΔG^{*E} and H^E on composition. The values of K^E_s, V^E and L_f^E are negative for all the systems over the entire mole fraction range and their magnitude decreases with rise in temperature from 303.15-313.15 K. The deviations observed in the excess parameters indicate the strength of interactions present between the component molecules of the binary mixtures under study [18]. Figures 1a, b and c show the excess isentropic compressibility K^E_s for the binary liquid mixtures of O- Toluidine with Formamide, N-Methylformamide and N, N-Dimethylformamide. Respectively, over the entire mole fraction range and at different temperatures T = (303.15-313.15) K. The excess isentropic compressibility (K^E_s) graphically presented in 1a, b and c. The excess isentropic compressibility has positive values for all studied systems over the whole composition range and become more positive with increasing temperature for all three binary mixtures. The observed values of excess isentropic compressibility (K^E_s) can be qualitatively explained by considering the following factors:

(i) Disruption of associated structure/molecular order in the pure liquids,

(ii) The formation of weak bonds dipole-induced–dipole interaction between unlike molecules and

(iii) Free volume changes from mixing of components of different sizes.





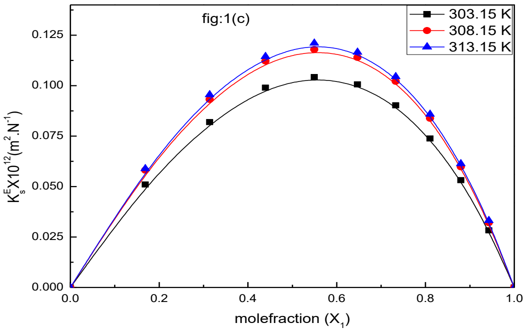
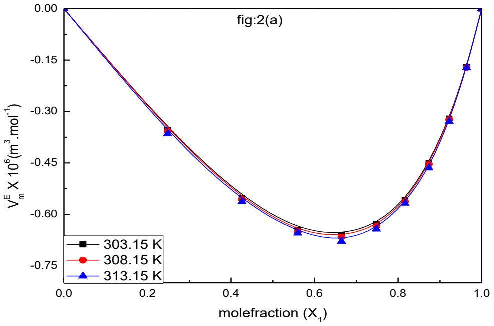
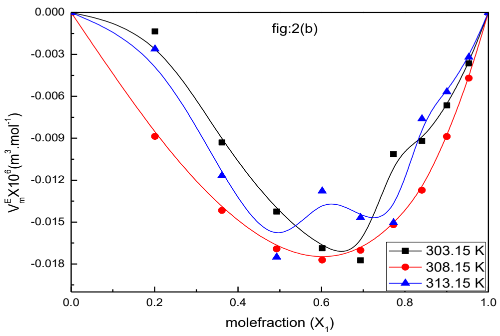


Fig.1 Variation of excess isentropic compressability (K_s^E) with mole fraction (x_1) in the binary liquid mixtures of O-Toluidine with formamide(1.a),N-methylformamide(1.b) and N,N-Dimethylformamide(1.c) at different temperatures.

It is clear from Fig. 1a, that the K^E_s value is negative over the entire mole fraction range for the systems under study and at investigated temperature; this indicates the presence of strong interactions in this mixture (O-TA+F). As the temperature increases, it has been observed that the negative K^E_s values are found to increase in the system, and the changes in K^E_s values with respect to temperature are small in these mixtures. Also with the increase in temperature, the solute solvent interactions get weaker causing the excess values to decrease at higher temperature [21]. The negative values of K^E_s are of the order (O-TA + F, NMF and N, N-DMF).The sign of excess isentropic compressibility plays a relevant role in assessing the compactness due to molecular interaction in liquid mixtures through charge transfer, dipole–dipole interactions, and dipole-induced dipole interactions interstitial accommodation, and orientational ordering leading to more compact structure making, which enhances excess isentropic compressibility to have negative values. Fort and Moore suggested that the liquids having different molecular sizes and shapes mix well there by reducing the volume which causes the values of K^E_s to be negative. It also suggests that the liquids are less compressible when compared to their ideal mixtures signifying the chemical effects including charge transfer forces, formation of hydrogen bond, and other complex forming interactions.





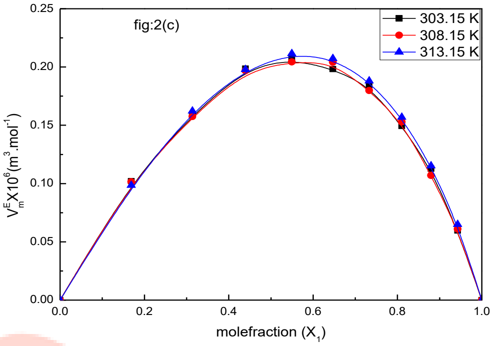
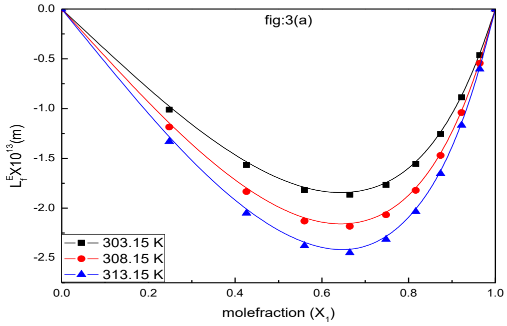
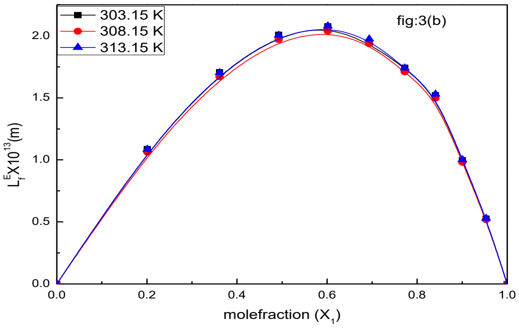


Fig.2 Variation of excess molar volume (V^E) with mole fraction (x_1) in the binary liquid mixtures of O-Toluidine with formamide(1.a),N-methylformamide(1.b)andN,N-Dimethylformamide(1.c) at different temperatures.

It can also be said that the molecular interactions are strong in these binary liquid mixtures and that the medium is highly packed. Similar results were obtained by earlier workers Fort rj, Moore [22].The K^E_s for the liquid mixtures under study in Fig. 1b and c. It can be observed that the K^E_s values are positive in case of O-TA+NMF and O-TA+N, N-DMF over the entire mole fraction range indicating the presence of weak interactions in these mixtures and also that the molecules are loosely packed in the mixtures due to their shape and size.
The V^E data for all the binary systems of O- Toluidine with Formamide, N-Methylformamide and N,N-Dimethylformamide graphically represented in Figs. 2 a, b and c.





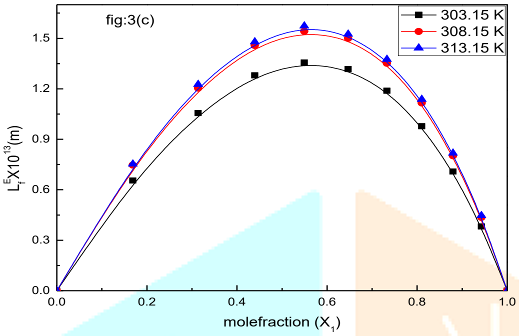
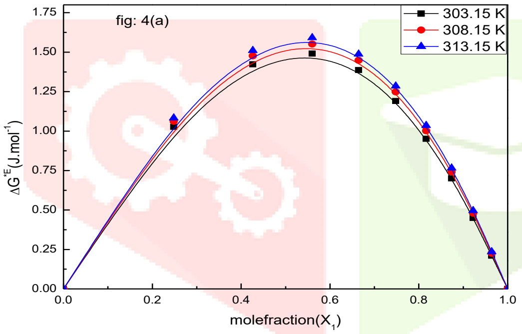
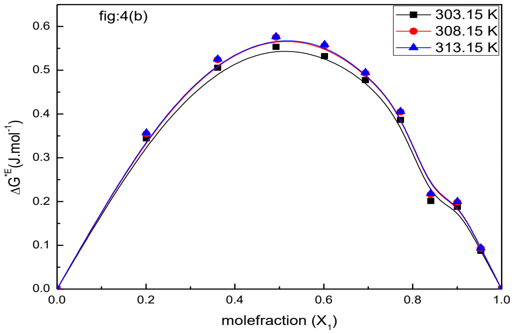


Fig.3 Variation of excess Free length (L_f^E) with mole fraction (x_1) in the binary liquid mixtures of O-Toluidine with formamide(1.a),N-methylformamide(1.b) and N,N-Dimethylformamide(1.c) at different temperatures.





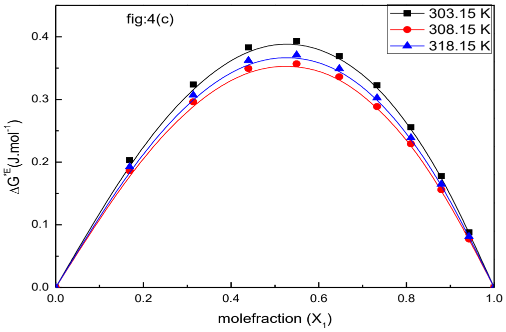
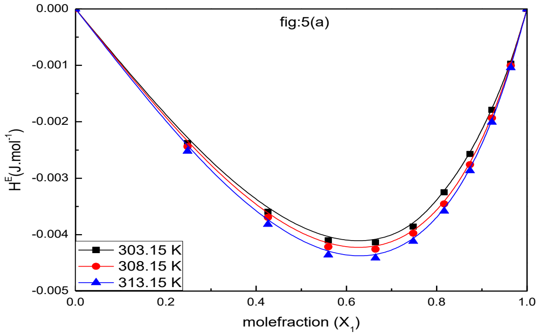


Fig.4 Variation of excess Gibb's function (ΔG^{*E}) with mole fraction (x_1) in the binary liquid mixtures of O-Toluidine with formamide(1.a),N-methylformamide(1.b) and N,N-Dimethylformamide(1.c) at different temperatures.

In these figures suggests that excess volume (V^E) data for the mixtures O- Toluidine with Formamide, N-Methylformamide are negative over the entire composition range at all temperatures. The experimental V^E values can be ascribed qualitatively by considering the factors which influence the excess functions, and these functions depend upon several contributions arising from physical, chemical and structural effects [23]. The physical contributions comprising of dispersive forces or weak dipole–dipole interactions lead to positive V^E values. Where as in O-TA+N, N-DMF, the positive values indicate a loose packing of molecules in the binary mixture compared to those in the pure component. Similar results were observed by earlier workers. Chemical contributions include breaking up of associates present in pure liquids resulting in positive V^E and specific interactions, like formation of strong hydrogen bonds, charge-transfer complexes and other complex forming interactions including strong dipole–dipole interactions between component molecules, resulting in negative V^E values. The structural contributions include the geometrical fitting of molecules of different molecular sizes into each other's, yielding negative V^E values. It can be observed from Fig. 3a, that the L_f^E values have a negative trend similar to what we have observed in case of the K^E_s at all the temperatures under study.



parameter	Temperature (K)	A0	A1	A2	A3	σ
O-TA + Formamide						
$10^{12}K_S^E$/ ($m^2.N^{-1}$)	303.15	-1.0128	1.122	-1.415	0.914	0.002
	308.15	-1.1857	1.314	-1.6572	1.069	0.002
	313.15	-1.3283	1.472	-1.8566	1.197	0.003
10^6V^E/ ($m^3.mol^{-1}$)	303.15	-2.4408	-1.275	-0.7303	-0.709	0.001
	308.15	-2.4637	-1.287	-0.7378	-0.717	0.001
	313.15	-2.4870	-1.314	-0.8311	-0.643	0.002
$10^{13}L_f^E$/ (m)	303.15	-6.9054	-3.581	-2.0451	-1.603	0.002
	308.15	-8.0869	-4.178	-2.4040	-1.920	0.003
	313.15	-9.0364	-4.697	-2.8041	-2.010	0.005
ΔG^{*E}/ ($J.mol^{-1}$)	303.15	5.9504	0.951	-0.1503	-0.590	0.003
	308.15	6.1819	1.062	-0.0882	-0.538	0.003
	313.15	6.3334	1.135	-0.0639	-0.509	0.002
H^E/ ($J.mol^{-1}$)	303.15	-0.0157	-0.003	-0.0030	-0.017	0.000
	308.15	-0.0161	-0.007	-0.0039	0.002	0.000
	313.15	-0.0167	-0.007	-0.0042	-0.003	0.000
O-TA + NMF						
$10^{12}K_S^E$/ ($m^2.N^{-1}$)	303.15	0.6358	0.182	0.0833	0.072	0.001
	308.15	0.6280	0.196	0.0844	0.043	0.001
	313.15	-1.3204	-0.681	-0.4224	-0.328	0.458
10^6V^E/ ($m^3.mol^{-1}$)	303.15	-0.0602	-0.055	0.0568	0.003	0.001
	308.15	-0.0679	-0.026	-0.0121	-0.006	0.000
	313.15	-0.0633	-0.026	0.0578	-0.043	0.001
$10^{13}L_f^E$/ (m)	303.15	-0.5878	-0.291	-0.1698	-0.125	1.945
	308.15	7.8896	2.656	1.3956	0.848	0.034
	313.15	8.0389	2.711	1.4684	0.849	0.036
ΔG^{*E}/ ($J.mol^{-1}$)	303.15	2.2347	0.103	-0.4124	-0.521	0.027
	308.15	2.3232	0.107	-0.4315	-0.445	0.026
	313.15	2.3294	0.108	-0.4108	-0.449	0.026
H^E/ ($J.mol^{-1}$)	303.15	-0.0142	-0.004	-0.0010	-0.000	0.000
	308.15	-0.0138	-0.004	-0.0011	-0.000	0.000
	313.15	-0.0135	-0.004	-0.0010	-0.000	0.000
O-TA + N,N-DMF						
$10^{12}K_S^E$/ ($m^2.N^{-1}$)	303.15	0.4114	0.090	0.0306	0.009	0.000
	308.15	0.4663	0.100	0.0359	0.008	0.000
	313.15	0.4773	0.101	0.0334	0.019	0.000
10^6V^E/ ($m^3.mol^{-1}$)	303.15	0.8137	0.187	0.1043	0.069	0.002
	308.15	0.8107	0.215	0.1023	-0.004	0.002
	313.15	0.8295	0.219	0.0968	0.096	0.000
$10^{13}L_f^E$/ (m)	303.15	5.3393	1.293	0.5002	0.189	0.002
	308.15	6.0764	1.473	0.5852	0.185	0.002
	313.15	6.1876	1.484	0.5600	0.316	0.003
ΔG^{*E}/ ($J.mol^{-1}$)	303.15	1.5732	0.179	-0.0426	-0.037	0.001
	308.15	1.4332	0.145	-0.0683	-0.072	0.001
	313.15	1.4877	0.160	-0.0571	-0.058	0.001
H^E/ ($J.mol^{-1}$)	303.15	-0.0079	-0.002	-0.0021	-0.000	0.000
	308.15	-0.0075	-0.002	-0.0002	-0.000	0.000
	313.15	-0.0075	-0.002	-0.0002	0.000	0.000

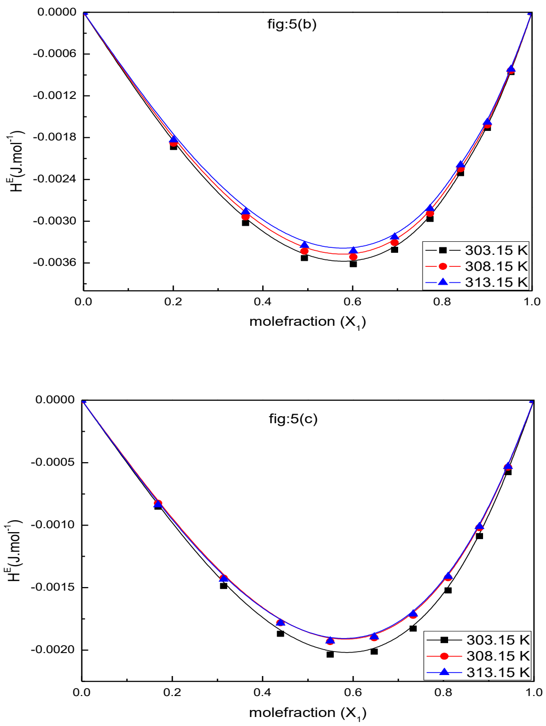


Fig.5 Variation of excess Enthalpy (H^E) with mole fraction (x_1) in the binary liquid mixtures of O-Toluidine with formamide(1.a),N-methylformamide(1.b) and N,N-Dimethylformamide(1.c) at different temperatures.

The negative values of L_f^E suggest that specific interactions are present between unlike molecules in this binary system. The positive values in Fig. 3b, c indicate the specific interactions are present between unlike molecules between the component molecules of the O-TA+NMF and O-TA+N, N-DMF liquid mixtures under study [24].

Table 03: Parameters (A_i) and Standard Deviation (σ) for O-Toluidine with Amides at different temperatures.

Figure 4a, b and c represent the excess Gibb's free energy of activation (ΔG^{*E}) with respect to mole fraction x_1, over the entire composition range and at T = (303.15-313.15) K. It can be seen from Fig. 4a, b and c that the ΔG^{*E} values are positive. These positive values indicate the existence of strong intermolecular interaction through hydrogen bonding between the component molecules of the liquid mixtures under study and negative values indicate weak inter molecular interaction. The maximum deviation is observed in the system indicating the strength of bond formation. Similar results were observed by earlier workers [25].

From Fig. 5a, b and c the excess values of enthalpy (H^E) are negative with respect to the mole fraction x_1, over the entire composition range and at T = (303.15-313.15) K. The negative values of H^E tend to decrease with increase in temperature, this insist the fact that there are strong specific interactions between unlike molecules and positive values indicate weak intermolecular interaction in these liquid mixtures. The variations in these above excess parameters with mole fraction x_1 and temperature predict the presence of hydrogen bonding between the compounds in both the binary mixtures. The excess parameters calculated in the present study correlate with one another and at the same time each parameter supports the formation of hydrogen bonding in these binary liquid mixtures.

5 CONCLUSION

Ultrasonic speeds of sound (U) , viscosity (η) and density (ρ) of mixtures of (O-TA with Formamide, NMF and N, N-DMF) over the entire composition range have been measured at T = 303.15-313.15 K. The values of K_s^E, V^E, L_f^E, H^E and ΔG^{*E} are calculated from this data. The excess and deviation properties have been fitted to Redlich-Kister type polynomial and corresponding standard deviations have been evaluated. The observed negative and positive values of K_s^E,V^E, L_f^E, H^E and ΔG^{*E} for all the liquid mixtures studied and clearly indicate. In these mixtures O-TA+Formamide have strong interactions than O-TA + NMF/N, N-DMF indicate strong and weak interactions present in the systems. i.e. O-TA+F> O-TA + NMF> O-TA+N, N-DMF.

6 **ACKNOWLEDGMENT**

"Dr.Shaik.Babu wishes to acknowledge the Department of Science and Technology for providing funding through DST-FIST Level-1 Scheme to Department of Physics, KLEF; File No:SR/FST/PS-1/2018/35".

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