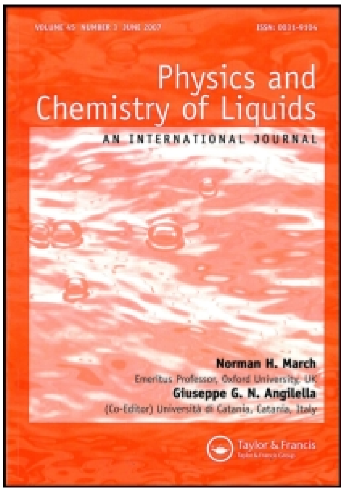

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Acoustic, viscometric and volumetric properties of binary mixtures of *N*-methylacetamide with some aliphatic mono and di alkyl amines at $T = 308.15\,\mathrm{K}$

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Excess molar volume, deviation in viscosity, and deviation in isentropic compressibility for the binary mixtures of amines with *N*-methylacetamide have been calculated from density, viscosity and speed of sound measurements at $T = 308.15\,\mathrm{K}$. The amines chosen for the study are diethyl amine, *n*-propyl amine, diisopropyl amine, *n*-butyl amine and di-*n*-butyl amine. All the properties have been fitted to the Redlich–Kister polynomial using multiparametric non-linear regression analysis to estimate the binary coefficients and standard errors. The results of excess properties have been discussed in terms of molecular interactions.

Keywords: *N*-methylacetamide; aliphatic amine; binary mixture; excess molar volume; deviation in viscosity; deviation in isentropic compressibility

1. Introduction

We have recently reported the thermodynamic properties of binary mixtures of *N*-methylacetamide (NMA) with different organic solvents [1–5]. As part of our ongoing research programme on thermodynamic properties, in this article we report new experimental data for the excess molar volumes (V^{E}), viscosity deviations ($\Delta\eta$) and deviations in isentropic compressibilities ($\Delta\kappa_s$) of binary systems of NMA with aliphatic mono and dialkyl amines: diethyl amine (DEA), *n*-propyl amine (*n*-PA), diisopropyl amine (DIPA), *n*-butyl amine (*n*-BA) and di-*n*-butyl amine (DBA). A survey of the literature reveals that these parameters are not reported for the selected systems. Primary and secondary amines are weakly self-associated through H-bonding. DBA is used as a corrosion inhibitor in the production of rubber, resins, dyes and pharmaceuticals. *n*-PA is used as a raw material for the manufacture of chlorpromazine, an anti-diabetic drug, and solvent dyes. DIPA is used in the synthesis of insecticides with herbicidal properties, in the manufacture of vasodilators, hypotensive drugs, as an intermediate in preparation of vulcanisation accelerators and in the manufacture of vapour phase corrosion inhibitors like diisopropyl ammonium nitrate (DIPAN). *n*-BA is used as a raw material in the manufacture of tolbutamide, an anti-diabetic, for blue shades of solvent dyes and disperse dyes, a hardener for epoxy resins and as a fuel additive for increasing the ignition delay period. Dibutyl amine is used as a catalyst in the manufacture of polyphenylene ethers and

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hydrogen peroxide, raw material for 2-di-*n*-butylamino-ethanol, an emulsifier, as an antifungal agent in water treatment and as a corrosion inhibitor in hydraulic fluids with castrol oil bases. NMA is used as an intermediate for the synthesis of active ingredients in pharmaceuticals.

Recently, substantial research work has been reported in the literature on the excess properties of amines with various organic solvents [6–12], while the work on NMA + amines has been scarce. These considerations influenced us to measure the properties of NMA + amines. In the present study, molecular interactions of five aliphatic mono and dialkyl amines with NMA have been investigated. When amines are mixed with NMA their mixing properties vary depending upon the alkyl chain length from mono to di-alkyl amines. In an effort to investigate this effect, we have measured density (ρ), viscosity (η) and speed of sound (u) of mixtures of NMA with the above mentioned amines over the entire range of mole fractions at a temperature of 308.15 K. From these data, excess molar volume (V^E), deviation in viscosity ($\Delta\eta$) and deviation in isentropic compressibility ($\Delta\kappa_s$) have been computed. These results have been fitted to the Redlich–Kister polynomial equation [13] to derive the binary coefficients and to estimate the standard deviation between the experimental and the calculated data. These data have been used to explain the nature of the molecular interactions between mixing components.

2. Experimental

2.1. *Chemicals*

High purity and analytical grade samples of DEA LR 99.0% (GC), *n*-PA 98.0% (GC), DIPA 99.0% (GC), *n*-BA 99.0% (GC) and DBA 99.0% (GC), obtained from S.D. Fine Chemicals, India, were dried over potassium hydroxide pellets for at least 48 h, distilled twice and the middle fractions were collected [14] and the amines were stored over 1.5 mm diameter type 3A molecular sieve pellets (Merck) to reduce water content if any, and to avoid the absorption of atmospheric moisture and carbon dioxide gas. NMA was obtained from Sigma-Aldrich, Germany, with purity better than 99 mol% (GC), and was used without further purification. The densities and viscosities measured at 308.15 K and ambient pressures of the pure liquids used in this investigation are compiled in Table 1, together with the literature data available. These results are in good agreement with the reference data.

2.2. *Apparatus and procedure*

Binary mixtures were prepared by mass in air tight bottles. The mass measurements were performed on a Dhona 100 DS, India, single pan analytical balance with a precision of 0.01×10^{-6} kg. The required properties of the mixture were measured on the same day. The uncertainty in mole fraction was estimated to be less than $\pm 1 \times 10^{-4}$. Densities of pure liquids and their mixtures were determined by using a $1 \times 10^{-5}\,\mathrm{m^3}$ double arm pycnometer, as described in our previous article [3]. The density values from triplicate replication at the temperature of 308.15 K were reproducible within $\pm 2 \times 10^{-2}\,\mathrm{kg\,m^{-3}}$. The uncertainty in density and excess molar volume values were found to be $\pm 4 \times 10^{-2}\,\mathrm{kg\,m^{-3}}$ and $\pm 0.001 \times 10^{-6}\,\mathrm{m^3\,mol^{-1}}$. An Ubbelohde viscometer [3] having a capacity of about 15 mL, a capillary length of about 90 and 0.5 mm internal diameter was used to measure the flow times of pure liquids and liquid mixtures, and was calibrated with benzene and doubly

Table 1. Physical properties of the pure components at $T = 308.15\,\text{K}$.

Component	ρ This work	ρ Literature	η This work	η Literature
NMA	0.94591	0.9460 [19]	3.3124	3.313 [4]
DEA	0.68827	0.68829 [9]	0.2465	–
n-PA	0.70029	0.7012 [8]	0.3002	0.328 [8]
DIPA	0.70148	–	0.3089	–
n-BA	0.72225	0.7239 [18]	0.3817	0.4249 [18]
DBA	0.74631	–	0.6757	–

distilled water. The experimental procedure for the use of the viscometer was discussed in our previous article [5]. Viscosity values (η) of pure liquids and mixtures were calculated using the relation:

$$\eta = (at - b/t)\rho, \tag{1}$$

where a and b are the characteristic constants of the viscometer, ρ is the density, and t represents the flow time. The flow time measurements of pure liquids and liquid mixtures were repeated five times. The uncertainty of viscosity and deviation in viscosity values are $\pm 0.005 \times 10^{-3}$ and $0.0003 \times 10^{-3}\,\text{kg m}^{-1}\,\text{s}^{-1}$. Speed of sound was determined using an ultrasonic interferometer (model M-82, Mittal Enterprises, India) working at 2 MHz frequency. The working principle used in the measurement of speed of sound through medium was based on the accurate determination of the wavelength of ultrasonic waves of known frequencies produced by a quartz crystal in the measuring cell. The temperature of the solution was controlled by circulating water at a desired temperature through the jacket of the double-walled cell. The uncertainty in speed of sound and deviation in isentropic compressibility was found to be ±0.3% and $0.4 \times 10^{-11}\,\text{m}^2\,\text{N}^{-1}$. In the various property measurements, the temperature was controlled within ±0.01 K using a constant temperature bath (Insref model IRI – 016 C, India).

3. Results and discussion

The values of excess molar volumes (V^E), deviation in viscosity $\Delta\eta$ and deviation in isentropic compressibility $\Delta\kappa_S$ for the binary mixtures of NMA with DEA, *n*-PA, DIPA, *n*-BA and DBA at $T = 308.15\,\text{K}$, along with mole fractions, are given in Table 2.

Excess molar volumes (V^E) have been evaluated from density using:

$$V^E = (x_1 M_1 + x_2 M_2)/\rho_m - (x_1 M_1/\rho_1 + x_2 M_2/\rho_2), \tag{2}$$

where ρ_m is the density of the mixture; x_1, M_1, ρ_1 and x_2, M_2 and ρ_2 are the mole fraction, molecular weight and density of pure components 1 and 2, respectively. The deviation in viscosity is calculated using the relation:

$$\Delta\eta = \eta_m - (x_1\eta_1 + x_2\eta_2), \tag{3}$$

Table 2. Densities, ρ, viscosities, n, speeds of sound, u, excess molar volume (V^E), deviation in viscosity ($\Delta\eta$), and deviation in isentropic compressibility ($\Delta\kappa_s$) of the binary mixtures of NMA (1) with DEA, n-PA, DIPA, n-BA and DBA at 308.15 K.

x_1	ρ	η	u	V^E	$\Delta\eta$	$\Delta\kappa_s$
NMA + DEA						
0.0000	0.6883	0.2466	1069.2	0.0000	0.0000	0.0000
0.0413	0.6991	0.2729	1099.7	−0.4501	−0.1004	−3.5547
0.1815	0.7328	0.4061	1145.2	−1.2096	−0.3970	−10.6561
0.3505	0.7746	0.6727	1194.0	−1.6978	−0.6485	−14.2085
0.5056	0.8132	1.0195	1229.0	−1.7001	−0.7772	−14.5157
0.5776	0.8319	1.2539	1243.4	−1.6365	−0.7635	−14.8514
0.6715	0.8569	1.5626	1256.5	−1.4840	−0.7427	−13.8096
0.7881	0.8902	2.0644	1286.7	−1.2988	−0.5984	−11.0055
0.8957	0.9175	2.6139	1319.6	−0.6278	−0.3788	−7.2760
0.9724	0.9386	3.0966	1332.7	−0.1929	−0.1312	−2.5462
1.0000	0.9459	3.3124	1362.0	0.0000	0.0000	0.0000
NMA + n-PA						
0.0000	0.7003	0.2998	1134.8	0.0000	0.0000	0.0000
0.0288	0.7090	0.3203	1147.9	−0.2611	−0.0667	−0.4104
0.161	0.7484	0.4788	1158.8	−1.2738	−0.3064	−1.1297
0.322	0.7890	0.6904	1170.8	−1.4886	−0.5798	−1.5961
0.4077	0.8110	0.8508	1202.1	−1.5827	−0.6775	−2.0696
0.5208	0.8394	1.1600	1205.8	−1.5984	−0.7089	−2.5571
0.6414	0.8695	1.5440	1213.2	−1.5330	−0.6882	−2.6260
0.7368	0.8921	1.8824	1255.8	−1.3458	−0.6372	−2.5527
0.8217	0.9125	2.2223	1293.1	−1.1726	−0.5531	−1.9007
0.9755	0.9417	3.1905	1338.7	−0.1923	−0.0481	−1.2651
1.0000	0.9459	3.3124	1362.0	0.0000	0.0000	0.0000
NMA + DIPA						
0.0000	0.7015	0.3089	1042.2	0.0000	0.0000	0.0000
0.0739	0.7150	0.3617	1072.1	−0.6723	−0.1692	−6.5114
0.2151	0.7398	0.5132	1107.2	−1.2309	−0.4417	−11.4728
0.3226	0.7616	0.7392	1136.4	−1.6814	−0.5386	−14.4783
0.4754	0.7962	1.0693	1195.1	−2.0945	−0.6675	−19.0412
0.6497	0.8382	1.6247	1295.3	−1.7929	−0.6356	−23.1325
0.786	0.8759	2.1667	1302.8	−1.2955	−0.5029	−14.7496
0.8761	0.9035	2.6524	1314.5	−0.8232	−0.2878	−8.4478
0.9277	0.9214	2.9734	1328.7	−0.5850	−0.1218	−4.9478
0.9762	0.9379	3.1945	1347.7	−0.2220	−0.0464	−1.5202
1.0000	0.9459	3.3124	1362.0	0.0000	0.0000	0.0000
NMA + n-BA						
0.0000	0.7223	0.3817	1198.2	0.0000	0.0000	0.0000
0.0335	0.7299	0.4140	1210.6	−0.2543	−0.0659	−1.9340
0.1849	0.7629	0.5985	1244.5	−0.9698	−0.3251	−5.9836
0.3822	0.8050	0.9135	1278.7	−1.2609	−0.5883	−7.8132
0.4385	0.8173	1.0486	1291.6	−1.2847	−0.6182	−8.3679
0.5671	0.8458	1.3872	1310.5	−1.2135	−0.6565	−7.8723
0.6888	0.8743	1.8291	1326.2	−1.1239	−0.5713	−6.6312
0.7756	0.8936	2.1503	1331.0	−0.8498	−0.5044	−4.6669
0.8792	0.9175	2.6754	1347.2	−0.4969	−0.2830	−2.9546
0.9781	0.9401	3.1721	1355.6	−0.0435	−0.0761	−0.2271
1.0000	0.9459	3.3124	1362.0	0.0000	0.0000	0.0000

(*continued*)

Table 2. Continued.

x_1	ρ	η	u	V^E	$\Delta\eta$	$\Delta\kappa_s$
NMA + DBA						
0.0000	0.7463	0.6757	1014.0	0.0000	0.0000	0.0000
0.0611	0.7524	0.7366	1019.8	−0.1019	−0.1003	−0.4520
0.2864	0.7795	1.1161	1046.5	−0.5398	−0.3147	−2.0432
0.4546	0.8042	1.5222	1074.4	−0.6067	−0.3522	−2.7161
0.5799	0.8265	1.8904	1102.5	−0.5800	−0.3143	−2.8244
0.6904	0.8501	2.2501	1131.7	−0.5332	−0.2459	−1.8986
0.7866	0.8745	2.5837	1163.7	−0.4494	−0.1661	−0.2711
0.8542	0.8937	2.8283	1189.5	−0.3101	−0.0996	1.8010
0.9262	0.9171	3.0599	1252.6	−0.1364	−0.0579	1.3913
0.9879	0.9409	3.2669	1341.0	−0.0269	−0.0136	0.1527
1.0000	0.9459	3.3124	1362.0	0.0000	0.0000	0.0000

where η_m, η_1 and η_2 are viscosities of the liquid mixture and of the pure components 1 and 2, respectively; x_1 and x_2 are the mole fractions of the pure components 1 and 2 in the liquid state.

The deviations in isentropic compressibility have been evaluated using the equation:

$$\Delta\kappa_S = \kappa_S - (\Phi_1\kappa_{S1} + \Phi_2\kappa_{S2}), \tag{4}$$

where Φ_i is the volume fraction of pure components and is calculated from the individual pure molar volumes, V_i, with the relation:

$$\Phi_i = x_i V_i / (\Sigma x_i V_i), \tag{5}$$

where κ_{S1}, κ_{S2} and κ_S are the isentropic compressibility of the pure components and observed isentropic compressibility of liquid mixture, respectively.

The excess properties, Y^E, were fit by the method of non-linear least squares to a Redlich–Kister fitting method [13] in the form of Equation (6) at the studied temperature.

$$Y^E = x_1 x_2 \Sigma A_i (x_1 - x_2)^i. \tag{6}$$

In each case, the optimum number of coefficients A_i is determined from an examination of the variation of standard deviation (σ), as calculated by:

$$\sigma(Y^E) = \left[\Sigma\left(Y^E_{obs} - Y^E_{cal}\right)/(n-m)\right]^{1/2}, \tag{7}$$

where n represents the number of experimental points and m is the number of coefficients used in fitting the data. The optimum number of parameters A_i was optimised using the F-test and was found to be five ($m = 5$). Values of A_i parameters, together with the standard deviation of the fits, are shown in Table 3.

3.1. *Excess molar volume*

Figure 1 displays plots of V^E *versus* x_1 (referring to NMA) at 308.15 K. From Figure 1, the observed negative excess volumes may be explained in terms of two opposing

Table 3. The binary coefficients (A_i) and standard errors (σ) of NMA (1) + aliphatic amines (2) at 308.15 K.

		Binary coefficients					
Binary system	Function	A_0	A_1	A_2	A_3	A_4	σ
NMA (1) + DEA (2)	V^E	−6.85160	−0.18653	−1.81133	2.94147	−1.15466	0.49727
	$\Delta\eta$	−3.09355	−0.52829	0.27870	−0.76198	−1.07947	0.07438
	$\Delta\kappa_s$	−56.84528	24.96995	−39.94765	−19.44226	81.78622	2.87253
NMA (1) + *n*-PA (2)	V^E	−6.13599	0.78535	−7.79647	−0.06630	5.46473	0.38261
	$\Delta\eta$	−2.80569	−1.37098	−1.28642	1.62586	2.17537	0.29980
	$\Delta\kappa_s$	−9.93402	−3.34912	5.27497	−5.67775	−13.65620	1.03763
NMA (1) + DIPA (2)	V^E	−8.34306	−0.80792	5.62935	2.20472	−9.20567	0.24294
	$\Delta\eta$	−2.69680	−0.59011	−0.22373	1.03354	0.94787	0.25365
	$\Delta\kappa_s$	−89.05957	8.88345	110.25930	68.83975	−148.24390	6.49424
NMA (1) + *n*-BA (2)	V^E	−5.01708	−0.00285	−3.33489	3.26788	3.64676	0.27358
	$\Delta\eta$	−2.64272	−0.33823	1.09172	−0.42836	−1.35268	0.16948
	$\Delta\kappa_s$	−31.64352	5.88170	2.75369	32.67511	−16.58307	2.89740
NMA (1) + DBA (2)	V^E	−2.47854	0.25350	−0.54300	−0.69954	1.44752	0.21596
	$\Delta\eta$	−1.42821	0.55010	1.03943	−0.10638	−1.20567	0.07139
	$\Delta\kappa_s$	−7.28103	27.52250	22.63131	−17.42132	−23.04047	1.45036

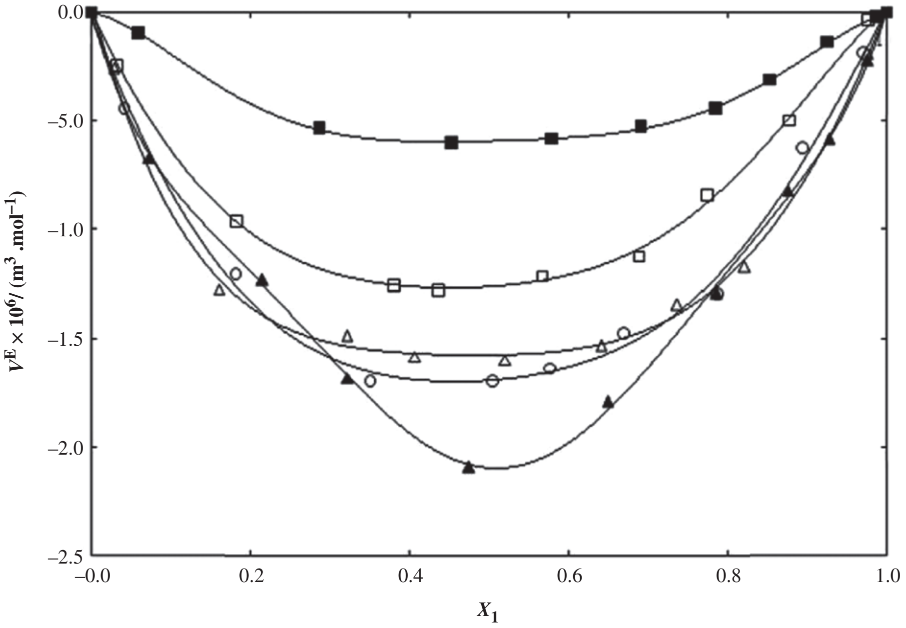

Figure 1. Excess molar volumes, V^E, at 308.15 K of binary mixtures of NMA with DEA (○), *n*-PA (△), DIPA (▲), *n*-BA (□), DBA (■).

effects: mixing of NMA with amines will induce the mutual dissociation of component molecules but also the formation of hydrogen bonds between unlike molecules. The former effect leads to positive excess volumes and the latter effect leads to negative excess volumes. The actual volume change depends upon the relative strengths of these two effects. The observed negative values of V^E show that the main contribution to V^E is due to hydrogen bond formation N–H$\cdots$O=C between unlike molecules. Moreover, the negative values of V^E may also be partly due to the specific acid–base interactions between NMA and amine molecules by considering NMA as a lewis acid and amine as a lewis base. Very recently Nam-Tran [15] emphasised the importance of acid–base interactions between *t*-butyl alcohol and *N*,*N*-dimethyl formamide/*N*,*N*-dimethylacetamide in order to evaluate the interaction energy of alcohol-amine systems. The large negative values of V^E for mixtures of NMA with DIPA is probably due to the more positive inductive effect of the isopropyl group, which increases the electron density on the nitrogen atom, resulting in an energetically favoured cross-bonding [8] N–H$\cdots$O=C compared to O=C–N–H$\cdots$NH.

From an examination of the results in Table 2 and Figure 1, it is observed that the negative values of V^E at equimolar mixture compositions ($x_1 = x_2$) increases with the increasing molar volumes, chain length and branching of the amines, as per the sequence:

$$\text{DIPA} > \text{DEA} > n\text{-PA} > n\text{-BA} > \text{DBA}.$$

The proton-accepting ability of these amines decrease and electron density will be less on the nitrogen atom of the $-NH_2$ group due to steric hindrance. Hence, as the chain length and branching of the amine increases, the interaction ability (hydrogen bonding ability) of amine with NMA decreases. The V^E values are all negative and pass through a minimum with increasing chain length from mono to di alkyl amines, except for DIPA. The magnitude of the V^E is least for straight chain DBA and highest for the secondary DIPA. It appears that chain branching leads to less negative values for V^E.

3.2. *Viscosity behaviour*

The viscosity measurements are carried out in order to elucidate the order of molecular interactions. A correlation between the sign of $\Delta\eta$ and V^E has been observed for binary solvent systems, $\Delta\eta$ being positive where V^E is negative or vice-versa [16]. Figures 1 and 2 clearly indicate that the isotherms of V^E and $\Delta\eta$ do not obey the above general statement except in the case of DBA, for which a sigmoid trend is observed. Therefore, the strength of specific or dispersion forces is not the only factor influencing the viscosity deviation of the liquid mixtures. The molecular size and shapes of the components are also equally important. Rastogi *et al.* [17] suggested that the observed excess property is a combination of an interaction and a non-interaction part.

Thus, we may write

$$X^E_{(\text{observed})} = X^E_{(\text{interaction})} + X^E_{(\text{size effect})},$$

where $X^E_{(\text{observed})}$ refers to the excess or deviation in the property. The non-interaction part in the form of a size effect can be comparable to the interaction part and may be sufficient to reverse the trend set by the latter. Based on this theory, the observed negative values may be due to the smaller size of the NMA molecule as compared to the amines. Figure 2 shows that the deviation in viscosity $\Delta\eta$ at 308.15 K for the systems of NMA+ five amines are parabolic in nature, indicating negative deviations over the entire range of composition. $\Delta\eta$ values for all the systems become less negative with an increase in

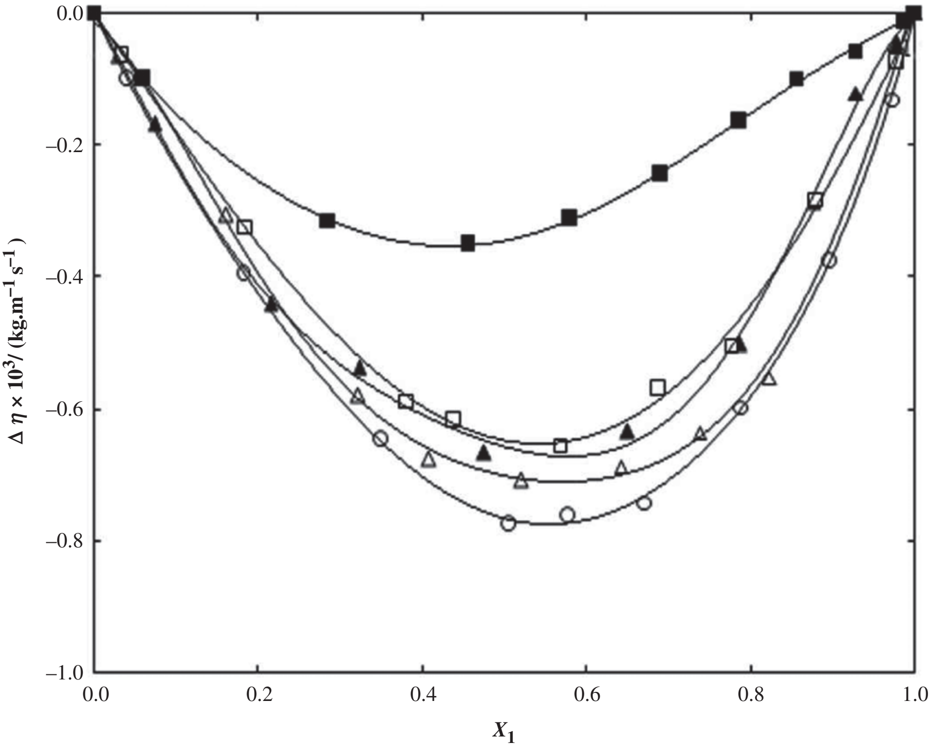

Figure 2. Deviation in viscosity, $\Delta\eta$, at 308.15 K of binary mixtures of NMA with DEA (○), *n*-PA (△), DIPA (▲), *n*-BA (□), DBA (■).

chain length and branching of amines. The negative $\Delta\eta$ values at equimolar concentration of NMA and amines vary, as per the sequence:

$$\mathrm{DEA} > n\text{-PA} > \mathrm{DIPA} > n\text{-BA} > \mathrm{DBA}.$$

3.3. *Speed of sound and compressibility*

The variation of $\Delta\kappa_S$ with composition of the mixture (Φ_1 of NMA) is displayed in Figure 3. From Figure 3, it is seen that $\Delta\kappa_S$ values in general are negative, except for DBA and become less negative with increasing chain length and branching. But in the case of *n*-PA the position is reversed. The case of DBA shows a minimum in this $\Delta\kappa_S$ curve yielding a sigmoid at Φ_1 (0.6) from negative to positive with increasing NMA composition. Further, it is observed that the order of magnitude of $\Delta\kappa_S$ for the present systems is approximately the same as that of V^E. The algebraic values of $\Delta\kappa_S$ for the five systems are in the order:

$$\mathrm{DIPA} > \mathrm{DEA} > n\text{-PA} > n\text{-BA} > \mathrm{DBA}.$$

Thus, the sign of $\Delta\kappa_S$ supports the postulates used to interpret the sign of excess molar volume.

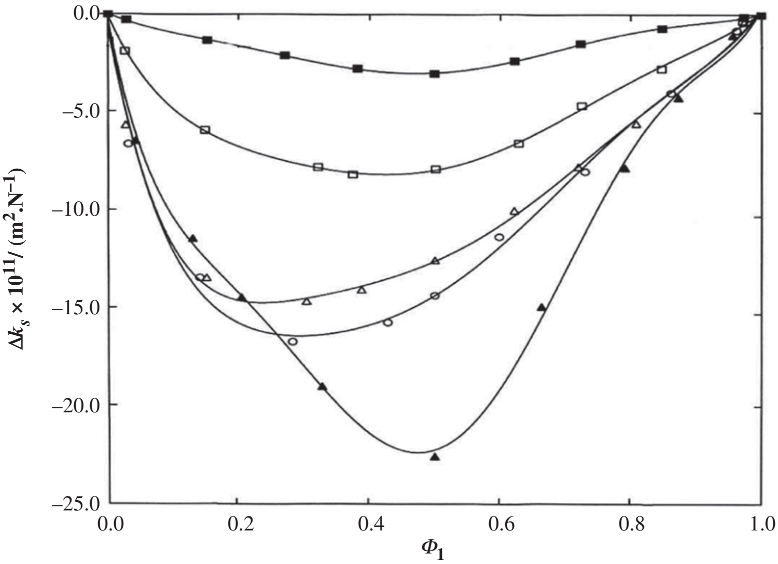

Figure 3. Deviation in isentropic compressibility, $\Delta\kappa_s$, at 308.15 K of binary mixtures of NMA with DEA (○), *n*-PA (Δ), DIPA (▲), *n*-BA (□), DBA (■).

4. Conclusions

This article reports experimental data for density, viscosity and speed of sound at $T = 308.15$ K for five binary mixtures of NMA + aliphatic mono and di alkyl amines. From these data, several thermodynamic excess functions have been calculated and studied to explain the intermolecular interactions and interaction ability between mixing components in terms of H-bonding (N–H ⋯ O=C), + inductive effect, lewis acid–base interactions and proton donor acceptor ability. From the study, it may be concluded that the chain length and branching of amine molecules with NMA show a significant effect on V^E, $\Delta\eta$ and $\Delta\kappa_S$ properties.

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