

Density and Speed of Sound of Binary Mixtures of *N*-Methylacetamide with Ethyl Acetate, Ethyl Chloroacetate, and Ethyl Cyanoacetate in the Temperature Interval (303.15 to 318.15) K

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The excess isentropic compressibilities are computed by measuring the density and speed of sound of binary mixtures of *N*-methylacetamide with ethyl acetate, ethyl chloroacetate, and ethyl cyanoacetate over the entire range of volume fraction in the temperature range of (303.15 to 318.15) K. The experimental results are fitted to a Redlich–Kister type polynomial equation. Estimated coefficients and standard deviation values are also presented.

Introduction

The variation of density, speed of sound, and isentropic compressibility of binary liquid mixtures of protic, aprotic, and associating liquids with changing volume fraction of one of the components has been investigated by some researchers. The trends of changes, either positive or negative, have been interpreted by these workers in terms of differences in size of the molecules and the strength of specific and nonspecific interactions taking place between the components of the mixtures.^{1–4} In our laboratory, we had investigated the ultrasonic, transport, and volumetric properties for the binary mixtures of *N*-methylacetamide (NMA) with aromatic ketones and observed that the chemical forces were acting between the unlike molecules.^{5,6}

As a part of our research program of measuring the physicochemical properties of binary mixtures of NMA with non-aqueous solvents, a study has been made on NMA + ethyl acetate, + ethyl chloroacetate, and + ethyl cyanoacetate over the entire range of volume fraction in the temperature interval of (303.15 to 318.15) K. Ethyl acetate was chosen because of its importance in polymer processing industries as a plasticizer.⁷ Due to its insecticidal activity, ethyl chloroacetate was chosen as a solvent in organic synthesis.⁸ Ethyl cyanoacetate was used as an intermediary for the synthesis of active ingredients in pharmaceuticals, agrochemicals, and pesticides. Detailed investigations on transport and acoustic properties of such mixtures are scarce in the literature.^{9–11} This prompted us to undertake a study on the measurements of density ρ and speed of sound u at $T = (303.15 \text{ to } 318.15) \text{ K}$. Using these data, excess isentropic compressibilities $\Delta\kappa_S$ have been calculated. These results have been fitted to the Redlich–Kister type equation to derive the binary coefficients.¹²

Experimental Section

Materials. High-purity and analytical grade sample of ethyl acetate with purity of 99.5 %, procured from S. D. Fine Chemicals Ltd., India, was used after a single distillation. Ethyl chloroacetate 99 % (GC), ethyl cyanoacetate 98 % (GC), and

Table 1. Comparison of Experimental Density $\rho/\text{kg}\cdot\text{m}^{-3}$ and Speed of Sound $u/\text{m}\cdot\text{s}^{-1}$ of Pure Liquids at $T = 298.15 \text{ K}$ with Literature Data

component	$\rho \times 10^{-3}/\text{kg}\cdot\text{m}^{-3}$		$u/\text{m}\cdot\text{s}^{-1}$	
	expt	lit	expt	lit
<i>N</i> -methylacetamide	0.94591 ^a	0.94600 ¹⁸	1362 ^a	1360 ⁵
ethyl acetate	0.89456	0.89455 ¹⁸	1145	1144 ⁴
ethyl chloroacetate	1.15603	1.15600 ²¹	1249	1249 ⁸
ethyl cyanoacetate	1.05642	1.05640 ¹⁸	1385 ^a	

^a At $T = 308.15 \text{ K}$.

NMA 99 % (GC) were purchased from Sigma-Aldrich Chemicals Pvt. Ltd., St. Louis, MO. These samples were used without further purification. The purities of the solvents were further ascertained by GLC and comparing their densities and speeds of sound at $T = 298.15 \text{ K}$, which agreed reasonably with the corresponding literature values (Table 1).

Apparatus and Procedure. Binary mixtures were prepared by mass in air-tight glass bottles.¹³ The mass measurements were performed on a Dhona 100 DS, India, single pan analytical balance with a precision of $0.01 \times 10^{-6} \text{ kg}$. The required properties were measured within 1 day of the mixture. 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 using a $1 \times 10^{-5} \text{ m}^3$ double-arm pycnometer as described by Nikam et al.¹⁴ The density values from triplicate replication at each temperature were reproducible within $\pm 2 \times 10^{-2} \text{ kg}\cdot\text{m}^{-3}$. The uncertainty in density values was found to be $\pm 4 \times 10^{-2} \text{ kg}\cdot\text{m}^{-3}$.

Speed of sound values were measured by using a single-crystal variable-path interferometer (Mittal Enterprises, India) operating at 2 MHz, which was calibrated with water and benzene.¹² The detailed procedure was described in our previous papers.^{15,16} The uncertainty in speeds of sound values was found to be 0.2 %. The isentropic compressibilities were calculated from

$$\kappa_S = 1/u^2\rho \quad (1)$$

where u is speed of sound and ρ is density. The uncertainty in excess isentropic compressibility was found to be $0.4 \times 10^{-11} \text{ m}^2\cdot\text{N}^{-1}$.

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Table 2. Values of Density ρ , Speed of Sound u , Isentropic Compressibility κ_S , and Deviations in Isentropic Compressibility $\Delta\kappa_S$ as a Function of Volume Fraction Φ_1 for Binary Mixtures of *N*-Methylacetamide (1) with Esters (2) from $T = (303.15 \text{ to } 318.15) \text{ K}$

ϕ_1	$\rho \times 10^{-3}$ kg·m ⁻³	u m·s ⁻¹	$\kappa_S \times 10^{11}$ m ² ·N ⁻¹	$\Delta\kappa_S \times 10^{11}$ m ² ·N ⁻¹	ϕ_1	$\rho \times 10^{-3}$ kg·m ⁻³	u m·s ⁻¹	$\kappa_S \times 10^{11}$ m ² ·N ⁻¹	$\Delta\kappa_S \times 10^{11}$ m ² ·N ⁻¹	ϕ_1	$\rho \times 10^{-3}$ kg·m ⁻³	u m·s ⁻¹	$\kappa_S \times 10^{11}$ m ² ·N ⁻¹	$\Delta\kappa_S \times 10^{11}$ m ² ·N ⁻¹
$T = 303.15 \text{ K}$														
Ethyl Acetate (2)														
0.0000	0.8889	1119	89.92	0.00	0.3991	0.91749	1223	72.83	-3.64	0.7437	0.93768	1306	62.53	-2.33
0.0538	0.89387	1132	87.32	-0.79	0.4982	0.92367	1248	69.56	-3.58	0.9463	0.94819	1354	57.56	-0.47
0.1843	0.90306	1167	81.25	-2.46	0.594	0.9293	1271	66.63	-3.27	1.0000	0.9513	1367	56.22	0.00
0.2972	0.91076	1197	76.59	-3.31	0.6955	0.93503	1295	63.82	-2.66					
Ethyl Chloroacetate (2)														
0.0000	1.13929	1229	58.07	0.00	0.2903	1.08592	1272	56.92	-0.61	0.7118	1.00688	1329	56.26	-0.50
0.0547	1.12833	1238	57.84	-0.14	0.4356	1.05931	1293	56.50	-0.77	0.8656	0.97731	1350	56.17	-0.30
0.1540	1.11027	1252	57.47	-0.32	0.5321	1.04112	1305	56.37	-0.71	1.0000	0.9513	1367	56.22	0.00
0.2473	1.09379	1265	57.11	-0.51	0.6203	1.02432	1317	56.30	-0.62					
Ethyl Cyanoacetate (2)														
0.0000	1.0514	1397	48.74	0.00	0.3672	1.01895	1386	51.08	-0.41	0.7307	0.98097	1375	53.95	-0.26
0.0529	1.04811	1396	48.98	-0.16	0.4502	1.01093	1384	51.66	-0.44	0.8795	0.96497	1370	55.20	-0.12
0.1860	1.03606	1391	49.86	-0.27	0.5435	1.00123	1380	52.42	-0.38	1.0000	0.9513	1367	56.22	0.00
0.2745	1.02782	1389	50.44	-0.36	0.5664	0.99875	1379	52.63	-0.35					
$T = 308.15 \text{ K}$														
Ethyl Acetate (2)														
0.0000	0.8826	1098	94.01	0.00	0.3988	0.91143	1208	75.25	-3.99	0.7434	0.93286	1296	63.80	-2.69
0.0537	0.88758	1114	90.87	-1.16	0.4979	0.91794	1234	71.49	-4.09	0.9488	0.94396	1348	58.32	-0.57
0.1841	0.89686	1149	84.52	-2.68	0.5937	0.92393	1259	68.30	-3.73	1.0000	0.9459	1362	56.99	0.00
0.2969	0.90458	1180	79.46	-3.56	0.6952	0.93002	1284	65.19	-3.09					
Ethyl Chloroacetate (2)														
0.0000	1.1325	1212	60.07	0.00	0.2902	1.08037	1256	58.68	-0.49	0.7118	1.00205	1318	57.49	-0.39
0.0547	1.12326	1221	59.74	-0.17	0.4356	1.05383	1277	58.18	-0.55	0.8656	0.97228	1342	57.15	-0.25
0.1540	1.10523	1235	59.28	-0.31	0.5320	1.03602	1291	57.92	-0.51	1.0000	0.9459	1362	56.99	0.00
0.2473	1.0882	1249	58.88	-0.43	0.6202	1.01938	1304	57.70	-0.46					
Ethyl Cyanoacetate (2)														
0.0000	1.0465	1385	49.83	0.00	0.3675	1.01263	1375	52.23	-0.23	0.7309	0.97466	1367	54.90	-0.17
0.0529	1.04191	1384	50.12	-0.09	0.4504	1.00444	1373	52.79	-0.27	0.8796	0.95876	1365	55.98	-0.15
0.1862	1.02963	1380	50.97	-0.19	0.5438	0.99468	1371	53.48	-0.25	1.0000	0.9459	1362	56.99	0.00
0.2747	1.02142	1378	51.60	-0.20	0.5666	0.99226	1370	53.68	-0.28					
$T = 313.15 \text{ K}$														
Ethyl Acetate (2)														
0.0000	0.8759	1081	97.66	0.00	0.3976	0.90715	1188	78.17	-4.35	0.7431	0.92967	1274	66.23	-3.14
0.0534	0.88097	1097	94.32	-1.31	0.4966	0.91379	1214	74.28	-4.48	0.9460	0.94122	1322	60.81	-0.83
0.1833	0.8913	1132	87.49	-3.19	0.5924	0.92009	1238	70.91	-4.19	1.0000	0.9436	1334	59.59	0.00
0.2958	0.89984	1161	82.45	-3.96	0.6941	0.92671	1263	67.63	-3.61					
Ethyl Chloroacetate (2)														
0.0000	1.1259	1198	61.93	0.00	0.2895	1.07453	1239	60.66	-0.59	0.7111	0.99715	1296	59.74	-0.52
0.0545	1.11523	1207	61.58	-0.22	0.4347	1.04813	1259	60.23	-0.68	0.8652	0.9685	1317	59.57	-0.34
0.1535	1.09823	1220	61.15	-0.42	0.5312	1.03046	1271	60.03	-0.65	1.0000	0.9436	1334	59.59	0.00
0.2466	1.08214	1233	60.78	-0.57	0.6194	1.01419	1283	59.86	-0.62					
Ethyl Cyanoacetate (2)														
0.0000	1.0408	1368	51.36	0.00	0.3667	1.00736	1356	54.03	-0.35	0.7303	0.97138	1343	57.10	-0.27
0.0528	1.0363	1366	51.69	-0.11	0.4497	0.99941	1353	54.64	-0.42	0.8793	0.95639	1337	58.49	-0.10
0.1857	1.02428	1362	52.60	-0.29	0.5430	0.99026	1349	55.48	-0.35	1.0000	0.9436	1334	59.59	0.00
0.2741	1.01597	1359	53.29	-0.32	0.5659	0.98792	1348	55.71	-0.30					
$T = 318.15 \text{ K}$														
Ethyl Acetate (2)														
0.0000	0.8696	1058	102.83	0.00	0.3968	0.90246	1172	80.62	-5.68	0.7418	0.92521	1261	67.98	-3.95
0.0533	0.87513	1074	99.03	-1.58	0.4958	0.90937	1198	76.57	-5.61	0.9458	0.93726	1309	62.31	-1.13
0.1828	0.8861	1113	91.18	-4.03	0.5916	0.91567	1223	72.98	-5.21	1.0000	0.9399	1319	61.17	0.00
0.2951	0.89479	1144	85.33	-5.20	0.6934	0.92212	1249	69.48	-4.46					
Ethyl Chloroacetate (2)														
0.0000	1.1199	1177	64.42	0.00	0.2892	1.06623	1212	63.88	0.39	0.7108	0.99068	1272	62.44	0.32
0.0544	1.10919	1187	63.95	-0.29	0.4344	1.04002	1231	63.43	0.42	0.8650	0.96331	1296	61.83	0.22
0.1533	1.09085	1202	63.42	-0.51	0.5308	1.02275	1244	63.18	0.48	1.0000	0.9399	1319	61.17	0.00
0.2464	1.07398	1207	63.94	0.32	0.6191	1.00706	1257	62.81	0.39					
Ethyl Cyanoacetate (2)														
0.0000	1.0347	1359	52.31	0.00	0.3663	1.00185	1348	54.91	-0.65	0.7299	0.96683	1332	58.34	-0.44
0.0527	1.03052	1358	52.61	-0.16	0.4492	0.99413	1345	55.59	-0.70	0.8791	0.95207	1324	59.91	-0.19
0.1854	1.01859	1356	53.42	-0.53	0.5425	0.98518	1341	56.46	-0.66	1.0000	0.9399	1319	61.17	0.00
0.2737	1.01048	1352	54.16	-0.58	0.5654	0.98297	1340	56.67	-0.65					

In all measurements, the temperature stability was controlled within $\pm 0.01 \text{ K}$ using a constant temperature bath

(INSREF model IRI-016 C, India) by circulating water from the thermostat. For all the properties, three readings were

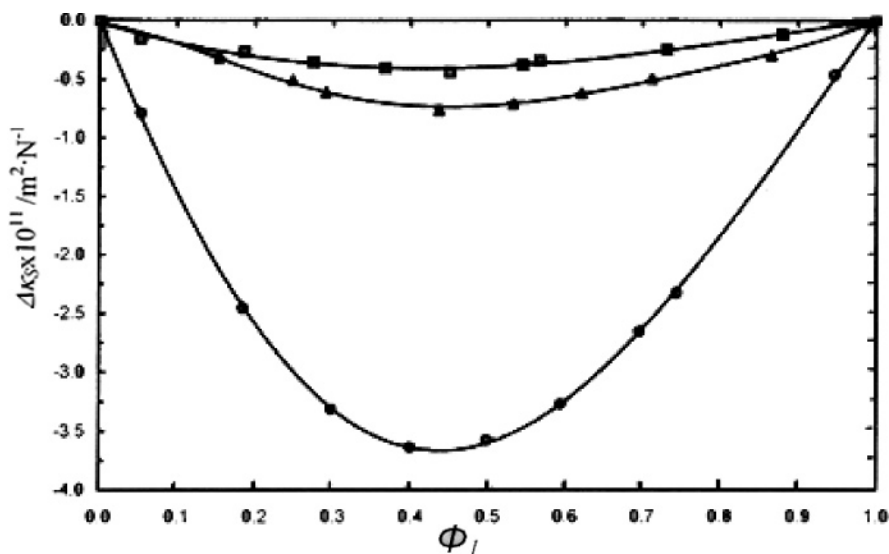


Figure 1. Variation of deviation in isentropic compressibility $\Delta\kappa_S$ with volume fraction Φ_1 of the binary mixtures of *N*-methylacetamide (1) with \circ , ethyl acetate; Δ , ethyl chloroacetate; \square , ethyl cyanoacetate at $T = 303.15$ K.

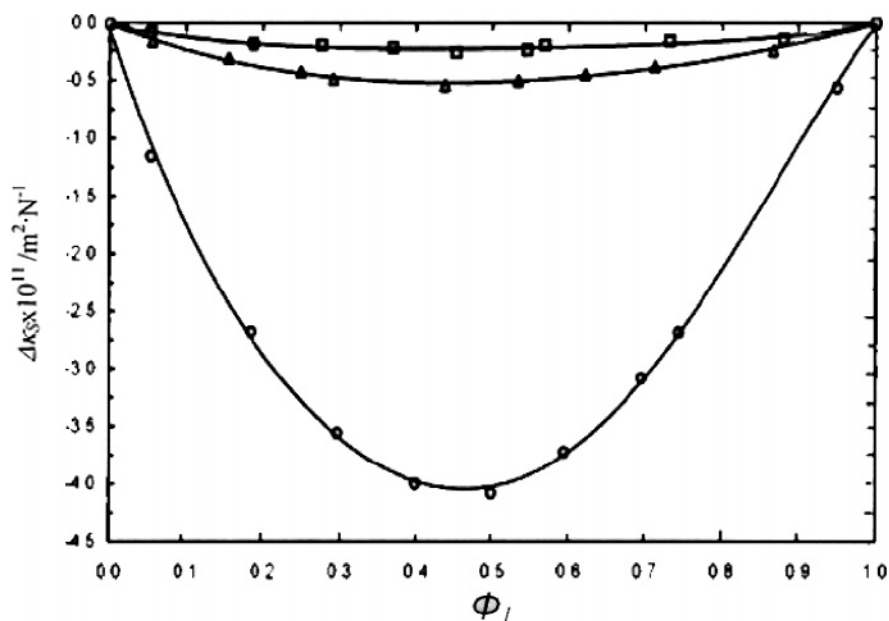


Figure 2. Variation of deviation in isentropic compressibility $\Delta\kappa_S$ with volume fraction Φ_1 of the binary mixtures of *N*-methylacetamide (1) with \circ , ethyl acetate; Δ , ethyl chloroacetate; \square , ethyl cyanoacetate at $T = 308.15$ K.

taken for each composition; the average of these values is given in Table 2.

Results and Discussion

The excess isentropic compressibilities have been evaluated using

$$\Delta\kappa_S = \kappa_S - (\Phi_1\kappa_{S1} + \Phi_2\kappa_{S2}) \quad (2)$$

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

$$\Phi_i = x_i V_i / (\sum x_i V_i) \quad (3)$$

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

Graphical representation of $\Delta\kappa_S$ values as a function of mole fraction of NMA over the range of temperatures studied is given in Figures 1 to 4. The excess isentropic compressibility is fitted to a Redlich–Kister polynomial equation:¹²

$$\Delta\kappa_S = \Phi_1(1 - \Phi_1)\sum A_j(1 - 2\Phi_1)^j \quad (4)$$

where $j = 0, 1, 2$, etc. and A_0, A_1, A_2 , etc. are adjustable parameters. These parameters are evaluated by fitting $\Delta\kappa_S/\Phi_1(1 - \Phi_1)$ into eq 4 by the method of least-squares. A best fit was obtained with five adjustable parameters. These values of parameters along with the standard deviation $\sigma(\Delta\kappa_S)$ as defined by

$$\sigma(\Delta\kappa_S) = [\sum(\Delta\kappa_{S\text{ obs}} - \Delta\kappa_{S\text{ calc}})^2/(n - m)]^{1/2} \quad (5)$$

are recorded in Table 3 for all liquid mixtures. Here n is total number of experimental points, and m is the number of parameters.

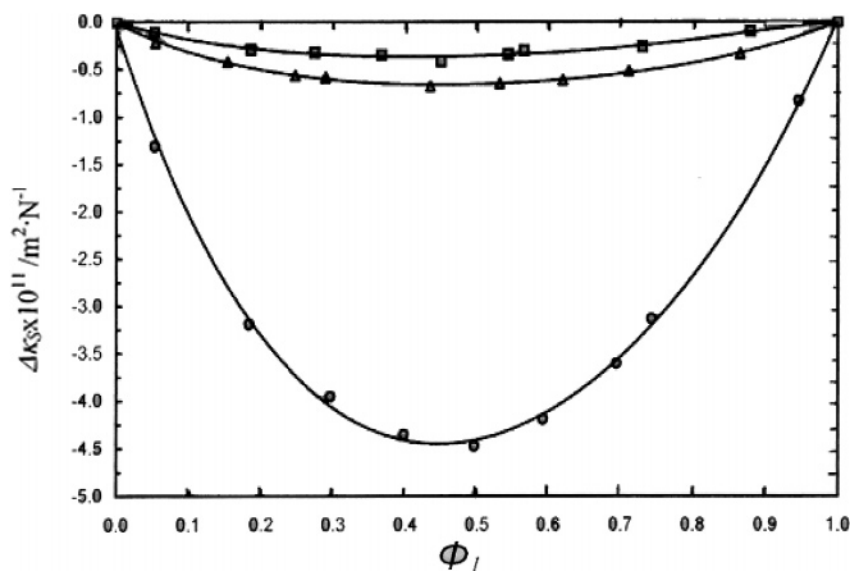


Figure 3. Variation of deviation in isentropic compressibility $\Delta\kappa_S$ with volume fraction Φ_1 of the binary mixtures of *N*-methylacetamide (1) with O, ethyl acetate; Δ , ethyl chloroacetate; \square , ethyl cyanoacetate at $T = 313.15$ K.

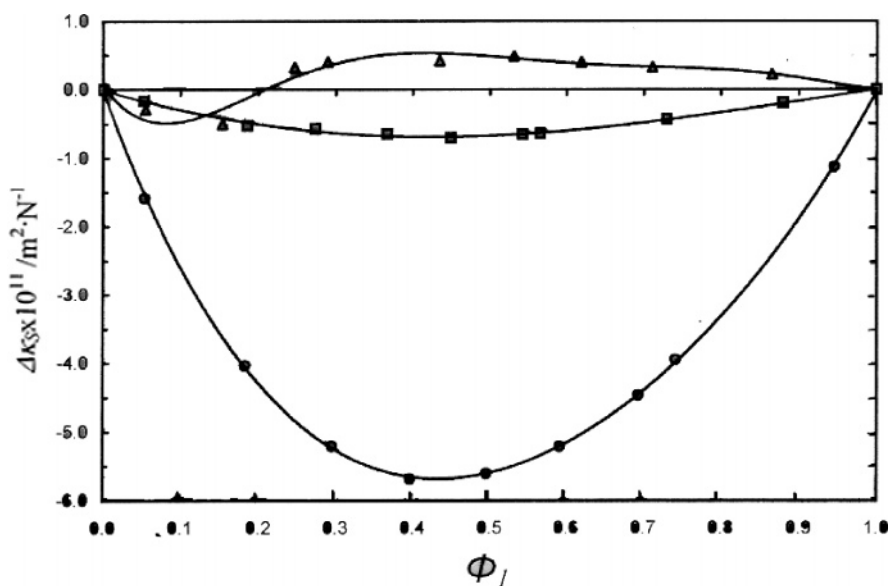


Figure 4. Variation of deviation in isentropic compressibility $\Delta\kappa_S$ with volume fraction Φ_1 of the binary mixtures of *N*-methylacetamide (1) with O, ethyl acetate; Δ , ethyl chloroacetate; \square , ethyl cyanoacetate at $T = 318.15$ K.

Table 3. Values of the Parameters of Equation 4 and Standard Deviation σ for the Binary Mixtures of *N*-Methylacetamide (1) with Esters (2) at Various Temperatures

binary system	function	T/K	A_0	A_1	A_2	A_3	A_4	σ
ethyl acetate (2)	$\Delta\kappa_S \times 10^{-11}/m^2 \cdot N^{-1}$	303.15	-14.30	4.26	-0.18	-0.90	3.23	0.06
		308.15	-16.32	2.09	5.30	5.09	-8.07	0.23
		313.15	-17.76	2.21	-0.58	3.95	-4.52	0.19
		318.15	-22.51	5.02	-1.38	0.26	-4.77	0.13
ethyl chloroacetate (2)	$\Delta\kappa_S \times 10^{-11}/m^2 \cdot N^{-1}$	303.15	-3.02	0.93	2.89	-1.85	-3.78	0.08
		308.15	-2.17	0.61	1.17	-0.71	-2.98	0.09
		313.15	-2.70	0.45	0.51	-0.15	-2.61	0.07
		318.15	2.17	-1.30	-4.74	8.29		1.13
ethyl cyanoacetate (2)	$\Delta\kappa_S \times 10^{-11}/m^2 \cdot N^{-1}$	303.15	-1.66	0.47	1.65	0.51	-3.13	0.14
		308.15	-0.96	0.40	0.35	-0.71	-1.91	0.09
		313.15	-1.45	0.61	-0.11			0.14
		318.15	-2.70	0.95	0.27			0.16

The system NMA + ethyl acetate, NMA + ethyl cyanoacetate, and NMA + ethyl chloroacetate show negative $\Delta\kappa_S$ values over the entire range of mole fraction and over the range of temperatures studied (Figures 1 to 4). In the case of the NMA + ethyl chloroacetate system, the positive $\Delta\kappa_S$ values exhibit

sigmoidal trend with an inversion of sign at $x = 0.3$ and temperature 318.15 K.

NMA is a dipolar protic solvent, with a very high dielectric constant (191.3) that is attributed to the liquid being highly structured with polymeric chains linked by hydrogen bonding.¹⁷

On the other hand, the molecules of esters have relatively low values of dielectric constants (5–20).¹⁸ In the present investigation, negative $\Delta\kappa_S$ values obtained over the range of temperatures studied for all mixtures (Figures 1 to 4) are attributed to chemical forces operating between unlike molecules of the binary mixtures.^{19,20}

Conclusions

New experimental data of density and speed of sound for the system of NMA with ethyl acetate, ethyl chloroacetate, and ethyl cyanoacetate were measured over the whole range of composition from the temperature range of (303.15 to 318.15) K. The excess isentropic compressibilities of these systems are correlated using the Redlich–Kister type polynomial equation. Estimated coefficients and standard deviation values are also presented. The results show that the volume reduction factors are dominating in all the systems over the entire range of temperatures studied.

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