Chapter - VI

Excess Molar Volume, Excess Energy and Viscosity Deviation of Binary Mixtures of Tetrahydrofuran with Some Hydrocarbons at Various Temperatures

6.1. *Introduction*

Grouping of solvents into classes often is based on the nature of the intermolecular forces because the nature of association between the solute and solvent molecules brings about a marked effect on the resulting properties. After the introduction of the concept of ionization power of solvent,[1] much work has been devoted to the solvent effects on the rate and equilibrium processes.[2] Because of the close relation between liquid structure and macroscopic properties, determination of volumetric and viscometric properties is a valuable tool to learn the liquid state.[3] On the other hand, the obtaining of reliable measurements of solvent properties over a wide range of composition, pressure and temperature often is not feasible; hence, prediction and correlation methods constitute a valuable option to overcome such difficulties.[4,5]

The present work contributes to the study of the structure and interactions of ether (THF) containing binary mixtures. Tetrahydrofuran (THF) is a good industrial solvent. It figures prominently in the high energy battery industry and has found its applications in organic syntheses as manifested from the physicochemical studies.[6,7] Since no reports are available of density and viscosity studies of tetrahydrofuran (THF) with normal aliphatic hydrocarbons, the effect of chain-length of hydrocarbons (C_5, C_6, C_7) on these properties of binary mixtures of tetrahydrofuran with normal hydrocarbons at 288.15, 293.15 and 298.15 K is reported in the present investigation.

6.2. *Experimental Section*

6.2.1 Materials

Tetrahydrofuran (THF, Merck) was kept for several days over KOH, refluxed for 24 h and distilled over $LiAlH_4$ as described earlier.[8] Normal pentane, hexane and heptane were purified according to the standard procedures.[9,10] The purities were

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checked by density determination dilafometrically[11] at (288.15, 293.15 and 298.15 K) ± 0.1 K which almost agreed within the accuracy of ± 1×10^{-4} g cm^{-3} with the available literature[12,13] values. All the studies were prepared volumetrically[14] in stoppered bottles.

6.2.2. *Apparatus and Procedure*

The densities were measured with an Ostwald-Sprengel type pycnometer having a bulb volume of 25 cm^3 and an internal diameter of the capillary of about 0.1 cm. The pycnometer was calibrated at (288.15, 293.5 and 298.15) K with doubly distilled water and benzene. The pycnometer with the test solution was equilibrated in a water bath maintained at ± 0.01 K of the desired temperature by means of a mercury in glass thermoregulator, and the temperature was determined with a calibrated thermometer and an Muller bridge. The pycnometer was then removed from the thermostatic bath, properly dried, and weighed. The evaporation losses remained insignificant during the time of actual measurements. Averages of triplicate measurements were taken into account. The density values were reproducible to $\pm3\times10^{-4}$ g cm^{-3}. Details have been described earlier.[15]

The viscosities were measured by means of suspended-level Ubbelohde[16] viscometer at the desired temperature (accuracy ± 0.01 K). The precision of the viscosity measurements was 0.05%. Details have been described earlier.[17]

The physical properties of tetrahydrofuran (THF), normal pentane, hexane and heptane at various temperatures are given in Table 1. The experimental values of mole-fractions, densities, viscosities and various derived parameters of various binary mixtures of solvents studied here at different temperatures are recorded in Table 2.

6.3. *Results and Discussion*

The experimental density (ρ) values (Table 1) have been used to calculate the excess molar volume (V_m^E), viscosity deviation ($\Delta\eta$) excess Gibbs free energy (G^{*E}) and interaction parameter (d) using the following equations[16] (1-4):

$$V_m{}^E = \frac{M}{\rho} - \sum_{i=1}^{c} x_i \frac{M_i}{\rho_i} \qquad (1)$$

$$\Delta\eta = \eta - \sum_{i=1}^{c} x_i\eta_i \qquad (2)$$

$$G^{*E} = RT\ln\frac{\eta M}{\rho} - RT\sum_{i=1}^{c} x_i \ln\frac{\eta_i M_i}{\rho_i} \qquad (3)$$

$$\ln\eta = \sum_{i=1}^{c} x_i \ln\eta_i + d\prod_{i=1}^{c} x_i \tag{4}$$

Where c stands for the number of components of the mixture, M and M_i are the molar masses of the mixture and of the pure components, ρ and ρ_i represent the density of the mixture and pure components, η and η_i are the corresponding viscosities, R is the gas constant T is the temperature in Kelvin and x_1 is the mole fraction. The values of these functions and d are recorded in Table 2 along with the values of ρ, η and x_1.

The excess molar volume were correlated by Redlich kister equation.

$$V_m^E = X_1 X_2 \Sigma a_j (x_1 - x_2)^i \tag{5}$$

The coefficient in eqn. (5) was estimated by the least-squares fit method, where the squares of the difference between the calculated and experimental excess molar volumes results is minimized. The optimal values of the coefficients in eqn. (5) are listed in table 3. The standard deviation was calculated by

$$\sigma(V_m^E) = \frac{\left[\Sigma(V_{m\,expt}^E - V_{mcalc}^E)^2\right]}{(D-N)^{0.5}} \tag{6}$$

where D and N are the number of data points and parameters, respectively. The experimental values of $\Delta\eta$ are also reported in Table 1. The viscosity deviations were correlated by the following equation:

$$Q = X_1 X_2 \Sigma H_j (x_1 - x_2)^j \tag{7}$$

Where Q = $\Delta\eta$ /(m pa. S). The coefficient in equation (7) are also regressed by employing the least square fit method. Standard deviations for the viscosity calculations were determined by equation (8).

$$\sigma(\Delta\eta) = \frac{\left[\Sigma(\Delta\eta_{expt} - \Delta\eta_{calc})^2\right]}{(D-N)^{0.5}} \tag{8}$$

The optimal parameters in correlating deviations of viscosity are listed in Table 4.

From Table-2 it is observed that the values of V_m^E for all binary mixtures THF + n-pentane, THF + n-hexane and THF + n-heptane at different temperatures over the entire composition are positive indicating the mutual dissociation of the component molecules. Because of the small difference in the molar volumes of the components,

tetrahydrofuran will not fit into the structure of the normal aliphatic hydrocarbons (C_5H_{12}, C_6H_{14}, C_7H_{16}), thereby increasing the volume of the mixture.[19] Positive V_m^E also suggests that the dispersion forces prevail between THF and normal hydrocarbons.

It is also found from Table-2 that the difference in the molar volumes of the mixtures of the components follows the following order:

THF + n-heptane > THF + n-hexane > THF + n-pentane

at any temperature studied here. This indicates the higher value of V_m^E for THF + n-pentane mixture than that of THF + n-hexane mixture which is in turn higher than that of THF + n-heptane (shown in figures. 1, 2 and 3).

There is a systematic increase in V_m^E with a rise in temperature for all mixtures studied here and such changes or variations are included in Figs. 1, 2 and 3. This increase in V_m^E with temperature is as expected from Theoretical considerations.[20]

Deviations in viscosity ($\Delta\eta$) shown in Figs. 4, 5 and 6 for the mixtures of tetrahydrofuran (THF) with normal aliphatic hydrocarbons (C_5 - C_7) are negative which increase with increasing size of hydrocarbons. The trend in $\Delta\eta$ is

THF - C_5H_{12} < THF - C_6H_{14} < THF - C_7H_{16}.

The dominance of exothermic enthalpy of mixing over endothermic mixing resulting THF-hydrocarbon interaction, leads to less negative $\Delta\eta$ values for higher aliphatic hydrocarbons than for lower ones.[21] It seems that in tetrahydrofuran + hydrocarbon systems, the forces between pairs of unlike molecules are far less than that between pairs of like molecules. Therefore, the binary mixtures of tetrahydrofuran with higher hydrocarbons are less fluid due to enhanced non-covalent forces between like molecules. Negative $\Delta\eta$ values in the present investigation can also be attributed due to unequal molecular sizes of the constituent molecules of the mixture where dispersion forces are dominant.[22]

The effect of temperature increase is to disrupt hetero and homo association of the molecules which causes increase in fluidity of the liquid. So, $\Delta\eta$ values are higher at higher temperatures. Similar results was obtained as reported earlier.[23]

The negative values of excess Gibbs free energy of flow (shown in Figs. 7, 8, 9) for tetrahydrofuran + n-hydrocarbons over the entire range of composition and temperature which indicate the formation of molecular complex between unlike molecules through non-covalent bonds. Subba *et al.*[24] made a similar observation from G^{*E} studies for the binary mixtures of some solvents.

The negative values of Grünberg and Nissan parameter (d) indicates the presence of strong molecular interactions due to appreciable dipole-dipole and dipole-induced dipole interactions[23] while the negative values of d may be attributed to dominance of dispersion type of forces[22] between the like molecules. The last conclusion is in excellent agreement with the values of d (shown in Table-2) obtained from the experiment in our investigation.

6.4 *Conclusion*

. In this chapter , we studied the interactions occurring in binary mixtures of ethers with some members of the normal hydrocarbon series. The work reveals the dominance of dispersive forces between the ethers and hydrocarbons. It seems that, in the Ether+ Hydrocarbon binary systems, the forces between the unlike pairs of molecules are far less than that between like pair of molecules. The effect of temperature increase is to disrupt the homo and hetero association of component molecules in the mixtures. There is scope of improvisation of the conducted study covering the detailed nature of the interacting forces between the mixing components

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Table 1

Physical Properties of Tetrahydrofuran, n-Pentane, n-Hexane and n-Heptane at Various Temperatures

T/K	$\rho \times 10^{-3}$ /kg m^{-3}		η /m. Pa. S	
	This Work	Literature	This Work	Literature
		Tetrahydrofuran (THF)		
288.15	0.88720	0.88720 [12]	0.50924	0.50923 [12]
293.15	0.88358	0.88357 [12]	0.49107	0.49106 [12]
298.15	0.87888	0.87888 [12]	0.45999	0.45999 [12]
		n-Pentane		
288.15	0.62528	0.62527 [9,10,11]	0.23145	0.23144 [9,10,11]
293.15	0.62131	0.62132 [9,10,11]	0.21996	0.21995 [9,10,11]
298.15	0.61883	0.61883 [9,10,11]	0.21210	0.21210 [9,10,11]
		n-Hexane		
288.15	0.66125	0.66124 [9,10,11]	0.33381	0.33381 [9,10,11]
293.15	0.65472	0.65471 [9,10,11]	0.31256	0.31255 [9,10,11]
298.15	0.65253	0.65252 [9,10,11]	0.29191	0.29190 [9,10,11]
		n-Heptane		
288.15	0.68192	0.68192 [9,10,11]	0.42121	0.42121 [9,10,11]
293.15	0.67951	0.67950 [9,10,11]	0.40020	0.40010 [9,10,11]
298.15	0.67669	0.67669 [9,10,11]	0.38450	0.38450 [9,10,11]

Table 2

Density (ρ), viscosity (η), excess molar volume (V_m^E), viscosity deviation ($\Delta\eta$), excess free energy of activation of viscous flow (G^{*E}), interaction parameter (d) and mole fractions of tetrahydrofuran (x_1) with different normal hydrocarbons at various temperatures

x_1	ρ × 10^{-3} /kg. m^{-3}	η /m. Pa. S	V_m^E /Cm^3mol^{-1}	$\Delta\eta$ /m. Pa. S	G^{*E} / J mol^{-1}	d
			288.15 K			
			THF + n-Pentane			
0	0.62528	0.23145	0	0	0	0
0.10005	0.64384	0.24404	0.08012	-0.01520	-48.30241	-0.28795
0.20009	0.66314	0.25863	0.22501	-0.02841	-85.07026	-0.29207
0.30012	0.68356	0.27829	0.38213	-0.03653	-87.33218	-0.24928
0.40013	0.70536	0.29859	0.52531	-0.04401	-98.96688	-0.25388
0.50014	0.72910	0.32188	0.60112	-0.04850	-103.4397	-0.25829
0.60013	0.75389	0.34765	0.75452	-0.05051	-104.1155	-0.27671
0.70012	0.78256	0.38240	0.65501	-0.04354	-70.39536	-0.23806
0.80009	0.81536	0.41850	0.35254	-0.03521	-57.71623	-0.24135
0.90005	0.85002	0.46314	0.15231	-0.01833	-19.71319	-0.17868
1	0.88720	0.50924	0	0	0	0
			THF + n-Hexane			
0	0.66125	0.33381	0	0	0	0
0.11713	0.67683	0.34235	0.31001	-0.01201	-27.52968	-0.23410
0.22998	0.69396	0.35205	0.47510	-0.02211	-52.41408	-0.24807
0.33863	0.71227	0.36440	0.58502	-0.03002	-63.79133	-0.24709
0.44335	0.73168	0.37894	0.67511	-0.03265	-65.71838	-0.24491
0.54436	0.75343	0.39784	0.59002	-0.03147	-50.21391	-0.21945
0.64184	0.77676	0.41661	0.47512	-0.02980	-43.40952	-0.21533
0.73598	0.80164	0.43672	0.36013	-0.02631	-36.22659	-0.21676
0.82695	0.82184	0.45847	0.25003	-0.02041	-27.60456	-0.22318
0.91491	0.85643	0.48214	0.14561	-0.01217	-17.23062	-0.24081
1	0.88720	0.50924	0	0	0	0

Contd.

			THF + n-Heptane			
0	0.68192	0.42121	0	0	0	0
0.13376	0.69798	0.42314	0.01801	-0.00984	-7.43921	-0.17964
0.25784	0.71408	0.42721	0.16810	-0.01670	-7.72454	-0.18181
0.37327	0.73105	0.43232	0.29111	-0.02175	-9.27331	-0.19153
0.48091	0.74900	0.43746	0.38112	-0.02608	-17.43592	-0.21398
0.58153	0.76886	0.44679	0.32102	-0.02561	-12.01893	-0.21126
0.67580	0.78996	0.45649	0.24515	-0.02421	-11.42528	-0.21828
0.76430	0.81226	0.46761	0.17321	-0.02088	-9.62714	-0.22511
0.84753	0.83579	0.47985	0.11213	-0.01597	-8.13692	-0.23610
0.92597	0.86066	0.49304	0.06122	-0.00968	-7.39667	-0.26666
1	0.88720	0.50924	0	0	0	0
			293.15 K			
			THF + n-Pentane			
0	0.62131	0.21996	0	0	0	0
0.10005	0.63975	0.23307	0.10009	-0.01401	-40.13625	-0.24946
0.20009	0.65882	0.24979	0.28215	-0.02442	-52.82306	-0.20946
0.30012	0.67932	0.26882	0.42501	-0.03250	-58.50596	-0.19254
0.40013	0.70091	0.28943	0.60002	-0.03901	-64.64248	-0.19537
0.50014	0.72427	0.31156	0.72551	-0.04399	-74.89723	-0.21415
0.60013	0.74962	0.33706	0.80432	-0.04560	-76.91300	-0.22744
0.70012	0.77846	0.36856	0.68511	-0.04121	-61.08626	-0.21975
0.80009	0.81076	0.40335	0.44004	-0.03352	-50.21842	-0.22649
0.90005	0.84594	0.44846	0.18603	-0.01551	-5.25559	-0.11664
1	0.88358	0.49107	0	0	0	0
			THF +n-Hexane			
0	0.65472	0.31256	0	0	0	0
0.11713	0.67030	0.32340	0.34011	-0.01001	-13.78878	-0.18204
0.22998	0.68750	0.33559	0.52012	-0.01802	-24.35637	-0.18527
0.33863	0.70596	0.34898	0.63510	-0.02403	-31.96879	-0.19098
0.44335	0.72573	0.36001	0.70001	-0.03169	-61.24642	-0.23893
0.54436	0.74763	0.37971	0.62503	-0.03002	-41.13155	-0.20693
0.64184	0.77130	0.40073	0.50002	-0.02640	-22.50561	-0.18047

Contd.

0.73598	0.79649	0.42074	0.38215	-0.02320	-18.26332	-0.18161
0.82695	0.82346	0.44201	0.26002	-0.01817	-14.90494	-0.18918
0.91491	0.385273	0.46499	0.10003	-0.01089	-11.68659	-0.20717
1	0.88358	0.49107	0	0	0	0

THF + n-Heptane

0	0.67951	0.40020	0	0	0	0
0.13376	0.69541	0.40303	0.03211	-0.00932	-6.19986	-0.17964
0.25784	0.71116	0.40749	0.23101	-0.01614	-6.62451	-0.18181
0.37327	0.72815	0.41302	0.32905	-0.02110	-8.97066	-0.19153
0.48091	0.74110	0.41907	0.39811	-0.02483	-14.95693	-0.21398
0.58153	0.76587	0.42849	0.33521	-0.02455	-10.38316	-0.21126
0.67580	0.78682	0.43850	0.26115	-0.02302	-9.17771	-0.21828
0.76430	0.80901	0.44949	0.18522	-0.02016	-8.91551	-0.22511
0.84753	0.83246	0.46185	0.11609	-0.01537	-7.42920	-0.23610
0.92597	0.85715	0.47507	0.06713	-0.00927	-7.24725	-0.26666
1	0.88358	0.49107	0	0	0	0

298.15 K

THF +n-Pentane

0	0.61884	0.21210	0	0	0	0
0.10005	0.63627	0.22409	0.25510	-0.01281	-37.51550	-0.24948
0.20009	0.65502	0.24048	0.46001	-0.02122	-39.51000	-0.18319
0.30012	0.67532	0.25800	0.59870	-0.02850	-45.81660	-0.17346
0.40013	0.69680	0.27677	0.75420	-0.03452	-54.29800	-0.18177
0.50014	0.71991	0.29768	0.88000	-0.03840	-59.58580	-0.19288
0.60013	0.74504	0.32036	0.94801	-0.04051	-67.55300	-0.21753
0.70012	0.77317	0.34700	0.87505	-0.03865	-66.34380	-0.23686
0.80009	0.80424	0.38003	0.71900	-0.03040	-43.53820	-0.22629
0.90005	0.84004	0.42120	0.34599	-0.01401	-1.44872	-0.11918
1	0.87888	0.45999	0	0	0	0

Contd.

Excess Molar Volume ……..…at Various Temperatures.

THF + n-Hexane						
0	0.65253	0.29191	0	0	0	0
0.11713	0.66764	0.30251	0.40111	-0.00909	-10.00730	-0.17017
0.22998	0.68449	0.31417	0.61002	-0.01546	-19.08990	-0.17560
0.33863	0.70279	0.32582	0.71502	-0.02155	-34.60930	-0.19689
0.44335	0.72236	0.33805	0.77501	-0.02838	-51.08960	-0.24782
0.54436	0.74425	0.35700	0.66541	-0.02748	-28.97290	-0.18652
0.64184	0.76770	0.37358	0.53021	-0.02621	-17.33250	-0.17095
0.73598	0.79261	0.39434	0.41001	-0.02127	-15.01330	-0.17459
0.82695	0.81936	0.41503	0.27512	-0.01587	-7.93677	-0.16881
0.91491	0.84793	0.43648	0.15003	-0.00921	-4.84634	-0.17684
1	0.87888	0.45999	0	0	0	0
THF + n-Heptane						
0	0.67669	0.38450	0	0	0	0
0.13376	0.69246	0.38606	0.05512	-0.00854	-5.49011	-0.17200
0.25784	0.70808	0.38915	0.26102	-0.01481	-5.70903	-0.17872
0.37327	0.72492	0.39354	0.35621	-0.01914	-6.60921	-0.18669
0.48091	0.74273	0.39842	0.41111	-0.02238	-11.48670	-0.20288
0.58153	0.76231	0.40609	0.35005	-0.02231	-8.35571	-0.20388
0.67580	0.78308	0.41463	0.28106	-0.02089	-6.83200	-0.20859
0.76430	0.80505	0.42423	0.19514	-0.01797	-5.56499	-0.21470
0.84753	0.82828	0.43559	0.13008	-0.01289	-4.67554	-0.21027
0.92597	0.85272	0.44651	0.07201	-0.00789	-3.80617	-0.24030
1	0.87888	0.45999	0	0	0	0

Table 3

Regression results for the excess volumes of THF + Normal Hydrocarbons mixture at various temperatures

System	Temp (K)	a_0	a_1	a_2	a_3	a_4	a_5	σ
THF + n-Pentane	288.15	2.6628	2.0239	-2.0835	2.8456	-	-	0.0405
	293.15	2.9725	1.8899	-2.0609	-2.3361	-	-	0.0187
	298.15	3.5424	1.3472	-	-	-	-	0.0341
THF + n-Hexane	288.15	2.5410	-1.2884	-2.0980	2.6035	3.2279	-2.9888	0.0138
	293.15	2.6047	-1.0650	0.4984	-	-	-	0.0173
	298.15	2.8035	-1.3676	-	-	-	-	0.0200
THF + n-Heptane	288.15	1.4462	-0.0143	-2.6272	1.1404	1.6129	-	0.0115
	293.15	1.5076	-0.4841	-1.6890	1.6837	-	-	0.0113
	298.15	1.5802	-0.5575	-1.5346	1.6635	-	-	0.0096

Table 4

Regression results for the deviation of viscosity of THF +Normal Hydrocarbons mixture at various temperatures

System	Temp (K)	a_0	a_1	a_2	a_3	a_4	a_5
THF + n-Pentane	288.15	0.1945	-0.0376	-	-	-	0.0011
	293.15	0.1761	-0.0428	-	-	-	0.0015
	298.15	-0.1572	-0.0444	-	-	-	0.0016
THF + n-Hexane	288.15	-0.1311	-0.0102	-	-	-	0.0009
	293.15	-0.1168	-0.0165	-	-	-	0.0014
	298.15	-0.1103	-0.0198	0.0274	-	-	0.0010
THF + n-Heptane	288.15	-0.1021	-0.0288	-	-	-	0.0005
	293.15	-0.0986	-0.0236	0.0206	-0.0097	-0.0487	0.0003
	298.15	-0.0884	-0.0213	-	-	-	0.0003

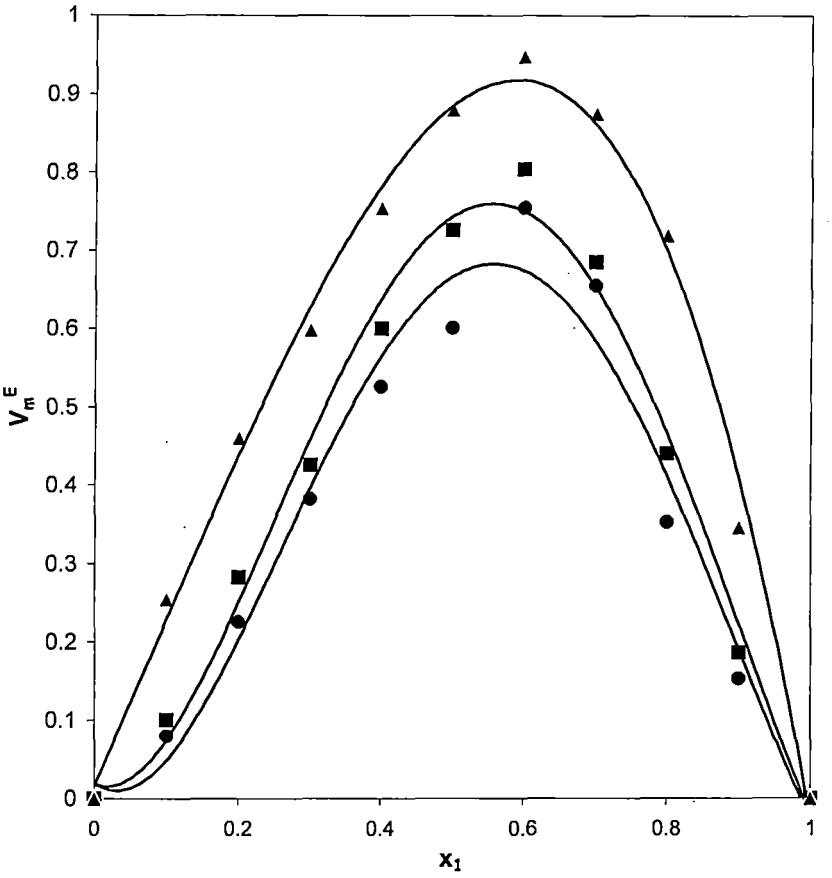

Fig. 1: Plot of excess molar volume (V_m^E) vs. mole fraction (x_1) of THF + n-Pentane mixtures at 288.15 K (•); 293.15 K (■) and 298.15 K (▲).

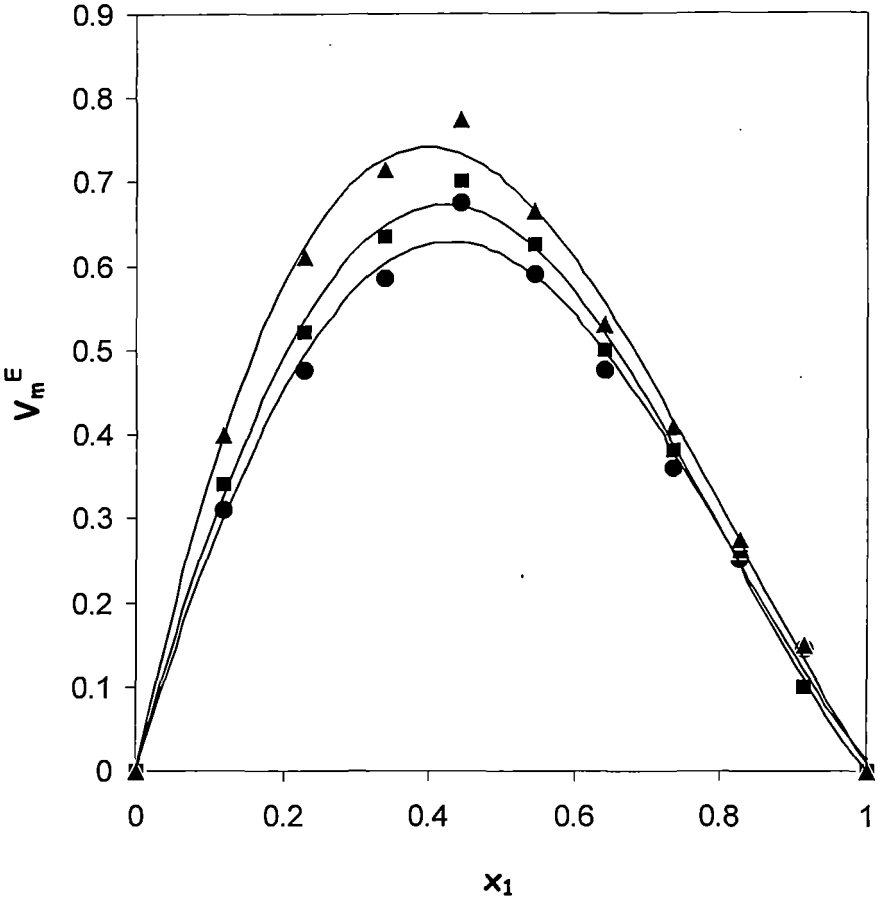

Fig. 2: Plot of excess molar volume (V_m^E) vs. mole fraction (x_1) of THF + n-Hexane mixtures at at 288.15 K (•); 293.15 K (■) and 298.15 K (▲).

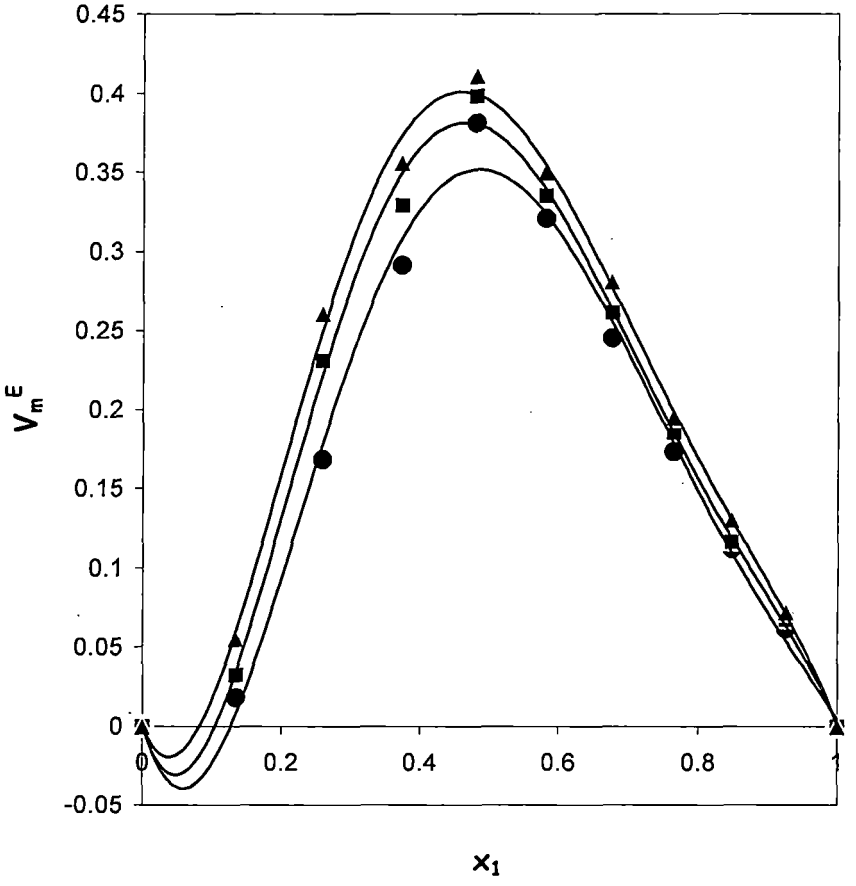

Fig. 3: Plot of excess molar volume (V_m^E) vs. mole fraction (x_1) of THF + n-Heptane mixtures at at 288.15 K (•); 293.15 K (■) and 298.15 K (▲).

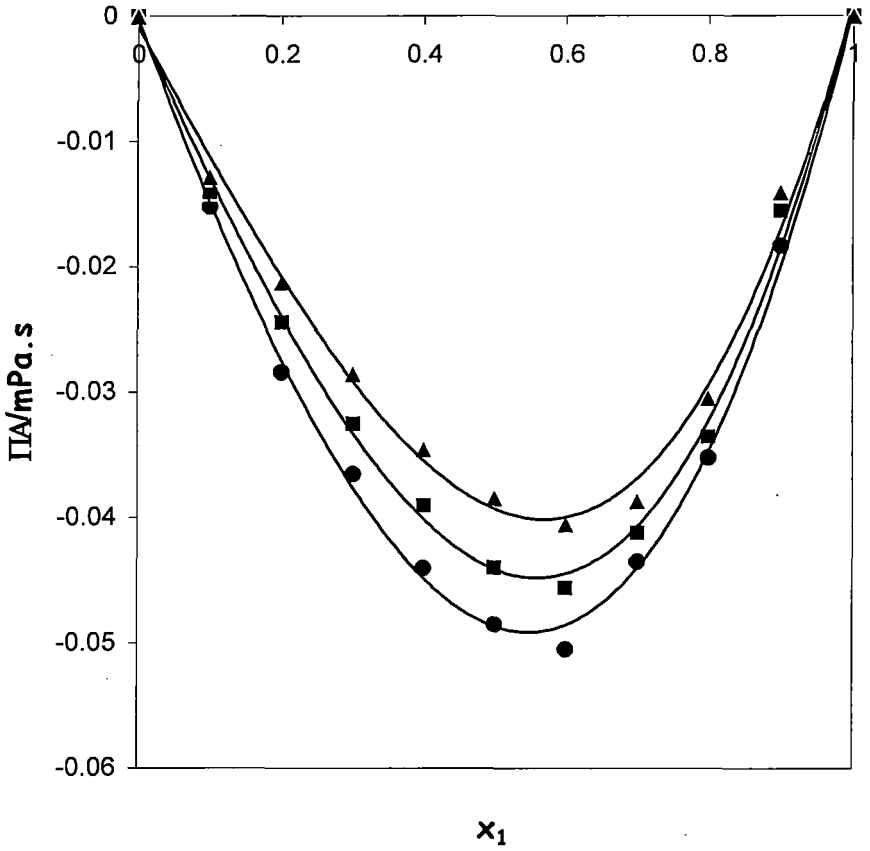

Fig. 4: Plot of viscosity deviation ($\Delta\eta$/m. Pa. S) vs. mole fraction (x_1) of THF + n-Pentane mixtures at at 288.15 K (•); 293.15 K (■) and 298.15 K (▲).

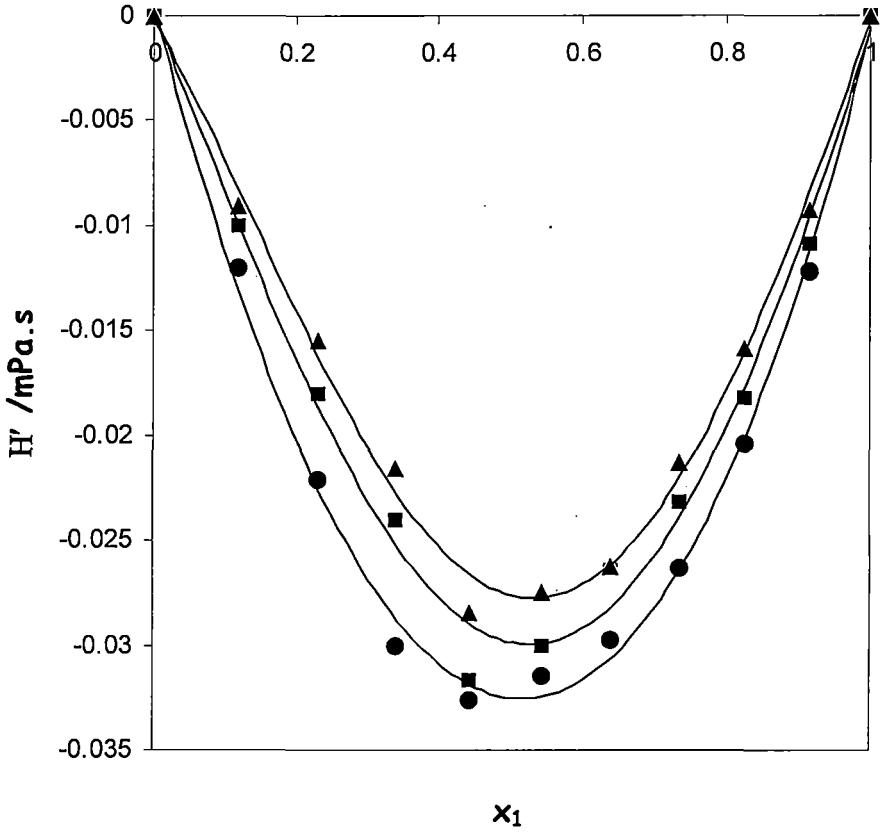

Fig. 5: Plot of viscosity deviation ($\Delta\eta$ /m. Pa. S) vs. mole fraction (x_1) of THF + n-Hexane mixtures at at 288.15 K (•); 293.15 K (■) and 298.15 K (▲).

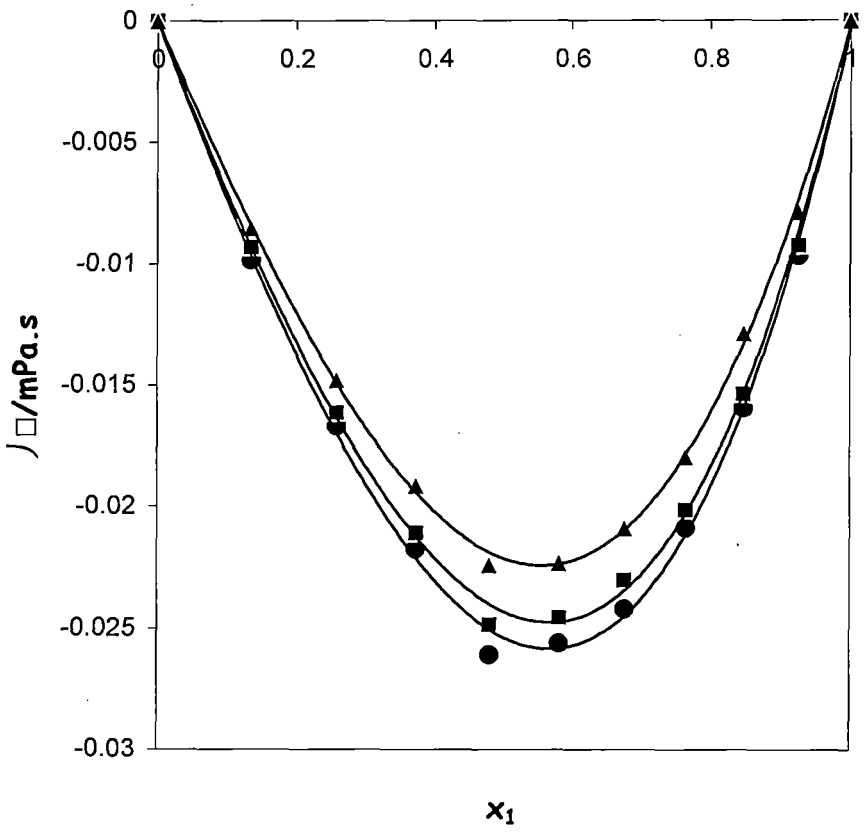

Fig. 6: Plot of viscosity deviation ($\Delta\eta$/m. Pa. S) vs. mole fraction (x_1) of THF + n-Heptane mixtures at at 288.15 K (•); 293.15 K (■) and 298.15 K (▲).

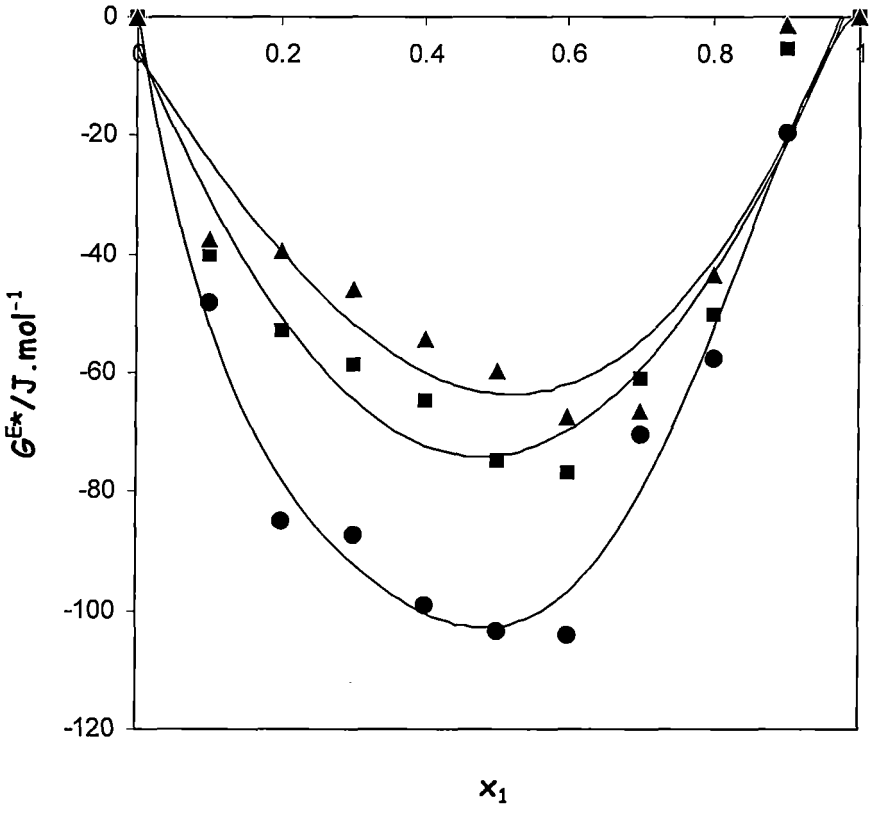

Fig. 7: Plot of excess free energy of activation of viscous flow (G^{*E}/J. mol^{-1}) vs. mole fraction (x_1) of THF + n-Pentane mixtures at at 288.15 K (•); 293.15 K (■) and 298.15 K (▲).

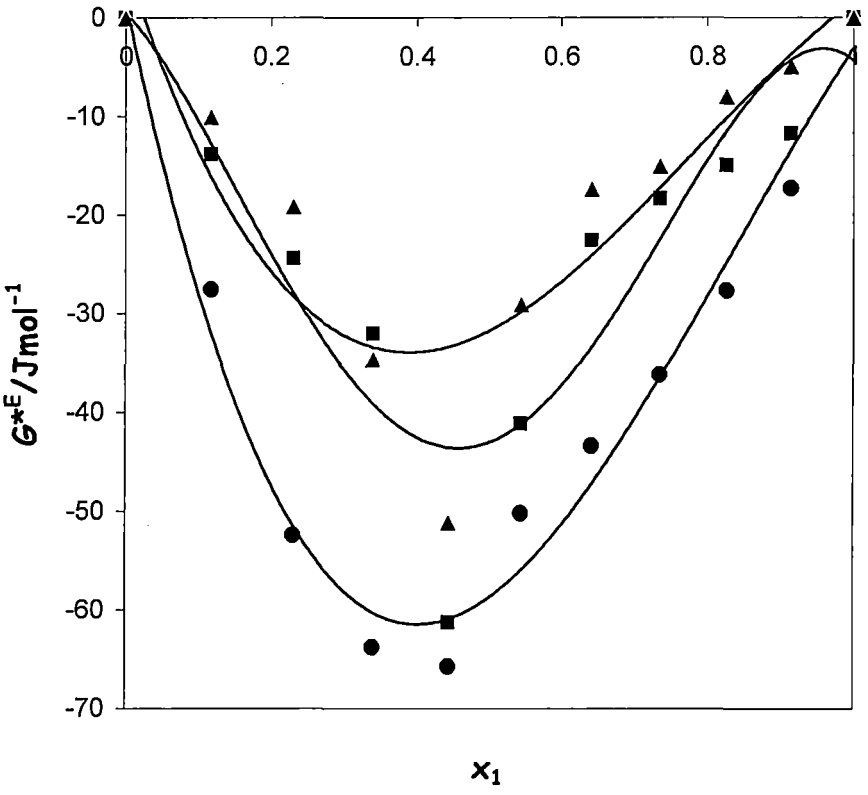

Fig. 8: Plot of excess free energy of activation of viscous flow (G^{*E}/J. mol^{-1}) vs. mole fraction (x_1) of THF + n-Hexane mixtures at at 288.15 K (•); 293.15 K (■) and 298.15 K (▲).

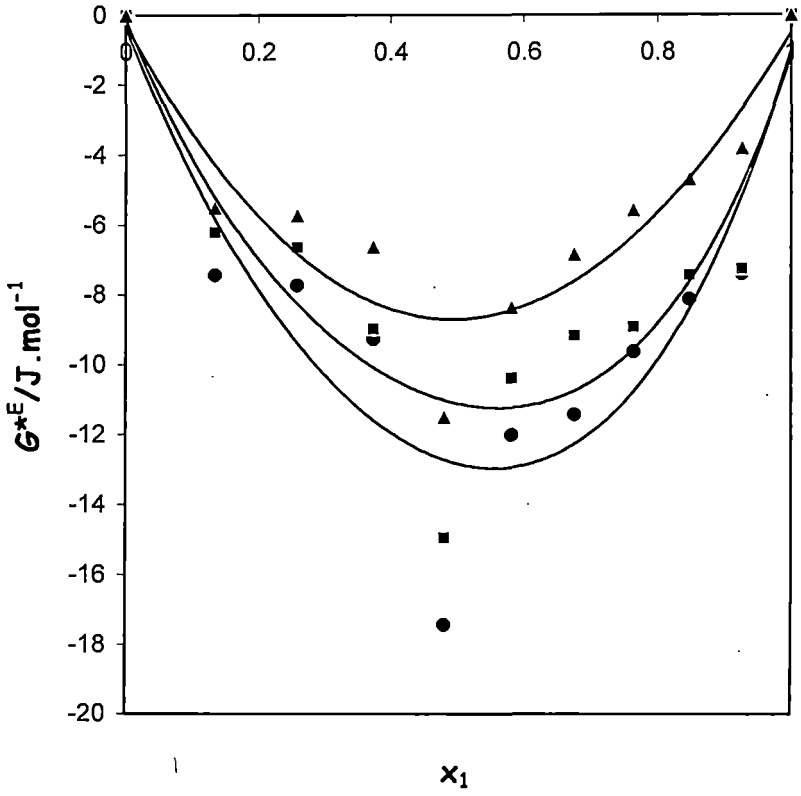

Fig 9 excess free energy of activation of viscous flow (G^{*E}/J. mol^{-1}) vs. mole fraction (x_1) of THF + n-Heptane mixtures at at 288.15 K (•); 293.15 K (■) and 298.15 K (▲).