

## Effect of *N,N'*-Dimethylformamide on Volumetric, Viscometric and Thermodynamic Behavior of Isomeric Butanols at (298.15 - 323.15)K

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### Abstract

Densities and viscosities of the isomers of Butanols ( n-Butanol, iso-Butanol and tert-Butanol ) and their binary mixtures with *N,N'* - Dimethylformamide (DMF) were measured at temperatures 298.15, 303.15, 308.15, 313.15, 318.15 and 323.15 K respectively covering the whole composition range. Densities and viscosities for binary mixtures have been plotted against mole fraction of Butanols at different temperatures. It shows continuous decrease in density on addition of n-Butanol, + iso-Butanol and + tert-Butanol in DMF. Excess molar volumes,  $V^E$  for the systems were found to be negative. The viscosities increase linearly up to 0.5 mole fraction and beyond this concentration the viscosity increases rapidly for all mixtures. Excess viscosities,  $\eta^E$  for the systems were found to be negative, which demonstrate that the Butanol solutions of DMF are highly non-ideal. The change of free energies ( $\Delta G^E$ ) increase gradually with increase in the mole fraction of Butanols for all mixture. Excess free energy change ( $\Delta G^{FE}$ ) for the systems were found to be negative. For all Butanols, excess viscosities and excess free energies show minima in alcohol rich region. The excess viscosities and excess free energies of activation for viscous flow are in the order: DMF + tert-Butanol > DMF + iso-Butanol > DMF + n-Butanol. Excess viscosity and excess free energy data have been fitted by the least squares method to the four parameters Redlich-Kister equation.

### I. Introduction

*N,N'*-Dimethylformamide (DMF) has a large dipole moment and a high dielectric constant [1]; therefore, it may work as an aprotic protophilic solvent used in the synthesis of pharmaceuticals, in agricultural chemistry, and as a solvent for polymers. Alcohols are polar liquids, strongly self-associated by hydrogen bonding. The thermodynamic properties of alcohols and their mixture are of great importance for food processing and chemical industries. Therefore the mode of interactions of alcohols and DMF is of vital importance in the field of solution chemistry as it can provide with important information regarding hydrophilic and hydrophobic interactions. A survey of the literature shows that very few measurements have been made on the densities for alcohol +DMF binary mixtures. Bai et al. [2, 3] reported excess properties for binary and ternary mixtures of ( *N* , *N'* - dimethylformamide + methanol + water) at 298.15 K, as well as of ( *N* , *N'* -dimethylformamide + ethanol + water) at the temperature 298.15 K. Zielkiewicz [4] reported the excess molar volumes in ( *N* , *N'* -dimethylformamide + methanol + water) at 313.15 K. Iloukhani and Rostami [5] gave the data of excess molar volumes for *N* , *N'* - dimethylformamide with 1-alkanol at 303.15 K. However, there are no reports on viscosity and thermodynamic parameters for binary mixtures of n-Butanol, + iso-Butanol, +tert-Butanol, + DMF systems at (298.15 to 323.15) K.

Here, we report density, viscosity, excess viscosity and thermodynamic parameters of three binary mixtures, viz, DMF, + n-Butanol, + iso-Butanol and +tert-Butanol at (298.15 to 323.15) K. The knowledge of interactions of simple systems may be useful sometimes to interpret many complex systems. The data are also useful for the design of mixing, storage and process equipment.

### II. Experimental

#### Materials

The chemicals used were purchased from Aldrich chemical co. with the quoted purities : n-Butanol (99.5%), iso-Butanol (99.0%) and tert-Butanol (99.5%) and *N* , *N'* - dimethylformamide (DMF) (99.5%). These chemicals were used without any further purification, except that they were allowed to stand over molecular sieves (4A) about one week before measurements.

#### Density Measurements

Densities were measured by using 5 mL bicapillary pycnometers. The volumes of the pycnometers were calibrated with deionized and doubly distilled water at (298.15, 303.15, 308.15, 313.15, 318.15 and 323.15) K. The densities of solutions in alkanols + DMF solutions were determined from the mass of the solution in the pycnometer after reaching thermal equilibrium with a water bath at the studied temperatures. Temperatures were controlled by a thermostatic water bath fluctuating to  $\pm 0.05$ K. A HR-200 electronic balance with an accuracy of  $\pm 0.0001$ g was used for the mass determination. Reproducibility of the results was checked by taking each measurement three times.

#### Viscosity Measurements

The viscosities were measured by calibrated U-type Ostwald viscometer of the British standard institution with sufficiently long efflux time to avoid kinetic energy correction. The provided calibration constants were checked with water, ethanol, and 1-hexane. The flow time of liquids was recorded by a timer to  $\pm 0.1$ sec. Temperatures were controlled by a thermostatic water bath fluctuating to  $\pm 0.05$ K. The viscosity,  $\eta$  of the solutions was calculated by  $\eta = A\rho t$ , where  $t$  is the flow time,  $\rho$  is the density of the solution, and  $A$  is the viscometer constant. The viscosity,  $\eta$  and the density,  $\rho$  were reproducible to within  $\pm 2 \times 10^{-3}$  mPa s and  $\pm 2 \times 10^{-4}$  g.cm<sup>-3</sup> respectively.

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**Table 1. Comparison of experimental and literature values of density,  $\rho$ (g.cm<sup>-3</sup>) and viscosity,  $\eta$  (mPa.s) of pure components at different temperatures**

Component	Temperature (K)	Density (g.cm <sup>-3</sup> )		Viscosity (m.Pa.s)	
		$\rho_{lit}$	$\rho_{exp}$	$\eta_{lit}$	$\eta_{exp}$
N,N-Dimethylformamide	298.15	0.9442 [6]	0.9443	0.790 [25]	0.793
		0.9445 [7, 8]			
	303.15	0.9394 [6]	0.9392	0.750 [25]	0.754
		0.9395 [9]			
	308.15		0.9345	0.710 [25]	0.709
	313.15	0.9298 [6]	0.9298	0.681 [25]	0.667
		0.9298 [10, 11]			
	318.15		0.9250		0.631
	323.15		0.9200		0.597
	n-Butanol	298.15	0.8073 [17]	0.8074	2.600 [26]
0.8060 [18]				2.571 [28]	
303.15		0.8020 [18]	0.8029	2.287 [29]	2.245
		0.8055 [20, 21]			
308.15		0.7995 [17]	0.7971		1.988
		0.7956 [17]			
313.15			0.7917	1.765 [27]	1.746
318.15			0.7878		1.545
323.15			0.7823		1.363
iso-Butanol		298.15	0.8023 [23,22]	0.7988	3.174 [29]
	0.7943 [36]		0.7956	2.842 [31]	2.840
	308.15		0.7919	2.279 [30]	2.398
		0.7902 [38]			
	313.15		0.7878	2.080 [31]	2.053
		0.7861 [38]			
	318.15		0.7834	1.692 [30]	1.770
		0.7822 [39,40]		1.861 [32]	
	323.15	0.7779 [37]	0.7779	1.482 [30]	1.533
				1.602 [33]	
tert-Butanol	298.15	0.7816 [24]	0.7832	4.371 [34,35]	4.235
			0.7784		3.307
	303.15		0.7737	2.690 [30]	2.627
		0.7697 [38]		2.589 [31]	
	308.15		0.7684	2.142 [30]	2.102
				2.047 [31]	
	313.15	0.7650 [37]			
			0.7629	1.736 [30]	1.712
	318.15	0.7596 [37]		1.690 [32]	
			0.7573	1.428 [30]	1.416
323.15	0.7541[37]		1.409 [33, 34]		

### III. Results and Discussion

Densities of *N,N'*-Dimethylformamide (DMF), + n-Butanol, + iso-Butanol and +tert-Butanol were measured at temperature at (298.15, 303.15, 308.15, 313.15, 318.15 and 323.15)K respectively covering the whole composition range. The densities of the pure components are shown in Table 1 together with the literature values for all temperature range, wherever possible for comparison.

The agreement between the measured values and literature values has been found to be almost satisfactory. Figure 1 represents the density,  $\rho$  vs.  $x_2$  curves at 308.15K for all the

systems as sample. It shows continuous decrease in density on addition of n-Butanol, + iso-Butanol and +tert-Butanol in DMF. In pure state the density of alcohol has been found to be in the order of,

n-Butanol > iso-Butanol > tert- Butanol

Viscosities,  $\eta$  of *N,N'*-Dimethylformamide (DMF), + n-Butanol, + iso-Butanol and +tert-Butanol were measured at temperature at (298.15, 303.15, 308.15, 313.15, 318.15 and 323.15)K respectively covering the whole composition range. The viscosities of the pure components are shown in Table 1 together with the literature values at different

temperatures. The agreement between the measured values and literature values of the pure components has been found to be almost satisfactory.

The viscosities of the binary systems have been shown in Table 3 as sample for n-Butanol at different temperatures. Figure 2 represent the viscosity,  $\eta$  vs.  $x_2$  curves at 298.15K for all the systems as sample.

The following characteristic features of viscosity are observed:

- The viscosities increase linearly up to 0.5 mole fraction of Butanols. Beyond this concentration, the viscosity increases rapidly until that of pure alcohol is reached specially at lower temperature.
- At the alcohol rich region rapid change of viscosity are observed for all three of alcohols but the change is pronounced for the branched alcohol (tert-Butanol) than the linear alcohol (n-Butanol).

In pure state the viscosity of alcohols has been found to be in the order of,

tert-Butanol > iso-Butanol > n- Butanol

The excess viscosities,  $\eta^E$ , have been calculated from viscosity data according to the equation:

$$\eta^E = \eta_{obs} - \eta_{id} \quad (1)$$

Where,  $\eta_{obs}$  is the experimentally observed viscosity of the mixture and  $\eta_{id}$  is the ideal viscosity of the mixture and

$$\eta_{id} = \exp(X_1 \ln \eta_1 + X_2 \ln \eta_2) \quad (2)$$

Where,  $X_1$  and  $\eta_1$  are the mole fraction and viscosity of component 1 (DMF),  $X_2$  and  $\eta_2$  are the corresponding values of component 2 (Butanols).

The excess viscosities,  $\eta^E$  were fitted to a Redlich- Kister polynomial equation of the form [29],

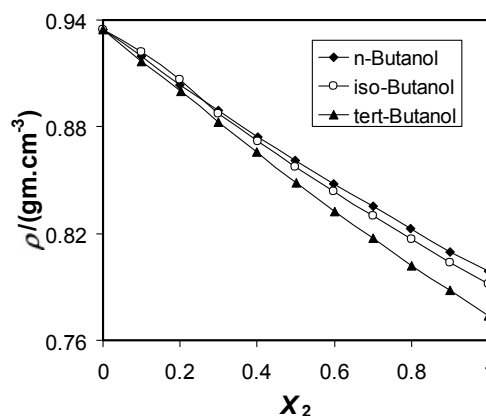
$$\eta^E (mPa.s) = X_1 X_2 \sum_{i=0}^n a_i (1 - 2X_1)^i \quad (3)$$

Where,  $A_i$  is the  $i^{th}$  fitting coefficient. Using  $n=3$ , four  $A_i$  were obtained through the least squares method.

In each case, the optimum number of coefficients  $A_i$  was determined from an examination of the variation of the standard derivation

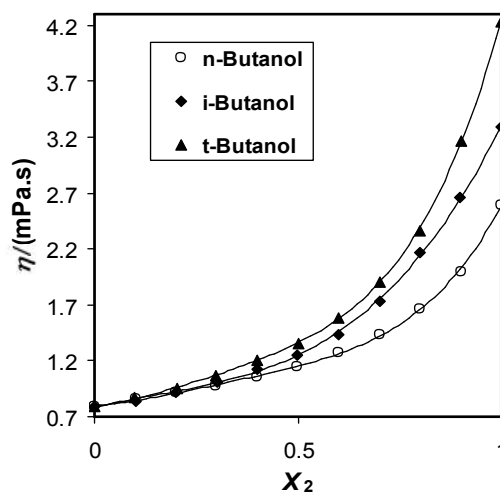
$$\sigma(Y) = \left[ \sum (Y_{cal} - Y_{exp})^2 / (n - m) \right]^{1/2} \quad (4)$$

where,  $n$  is the total number of experimental values and  $m$  is the number of parameters.



**Fig. 1.** Plots of density,  $\rho$  versus  $X_2$  for  $\blacklozenge$  n-Butanol,  $\circ$  iso-Butanol and  $\blacktriangle$  t-Butanol in DMF systems at 308.15 K.

The values of the fitting parameters along with the standard deviation are presented in Table 2. Excess viscosity,  $\eta^E$  for the systems of +n-Butanol, + iso-Butanol and +tert-Butanol in (DMF) systems have been plotted for comparison in Figure 3 at 308.15K.

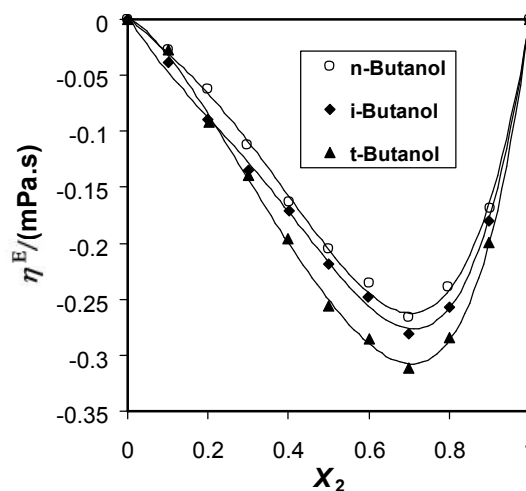


**Fig. 2.** Plots of viscosity,  $\eta$  versus  $X_2$  of  $\circ$  n-Butanol,  $\blacklozenge$  iso-Butanol and  $\blacktriangle$  t-Butanol in DMF systems at 298.15 K. The excess viscosities decrease in absolute values as the temperature is increased. The  $\eta^E$  values are negative and large in magnitude, which demonstrate that the DMF solutions of alcohols are highly non-ideal. All the curves pass through minima in the alcohol rich region. For the binary mixtures containing lower alkanols, sharp minima are not observed, but with an increase in the size of the alkanols, the  $\eta^E$  curves exhibit sharper minima, indicating varying molecular interactions between the mixing molecules depending upon their sizes and branches [17, 19]. In the present investigation at 308.15K, the minimum values  $\eta^E$  have been found to be -0.26 (at  $x_2=0.7$ ), -0.28 (at  $x_2=0.7$ ), -0.31 (at  $x_2=0.8$ ) for the DMF+ n-Butanol, + iso-Butanol

and + tert-Butanol mixtures respectively. The magnitude of the minima is in the order:

$$\text{tert-Butanol} > \text{iso-Butanol} > \text{n- Butanol}$$

The magnitude of excess viscosity,  $\eta^E$  increases with branch of alkanols, while it decreases with rise of temperature. This reveals that the strength of the intermolecular hydrogen bonding through C-O and O-H in DMF and Butanols, is not the only factor influencing the excess viscosity of liquid mixtures, but the orientation of groups, molecular sizes and shapes of the components are also equally important. Larger the branch of alkanols, greater is decrease in the average degree of association, as a result more negative excess viscosity vs. mole fraction curve is observed.



**Fig. 3.** Plots of excess viscosity,  $\eta^E$  versus  $X_2$  of  $\circ$  n-Butanol,  $\blacklozenge$  iso-Butanol and  $\blacktriangle$  t-Butanol in DMF systems at 308.15 K.

**Table 2 . Coefficient,  $A_i$ , of Redlich- Kister Eq (3) expressing excess viscosity,  $\eta^E$  and standard deviation,  $\sigma$  for the DMF +n-Butanol, + iso-Butanol and +tert-Butanol systems**

Systems	T/K	$A_0$	$A_1$	$A_2$	$A_3$	$\Sigma$
DMF+n-Butanol systems	298.15	-1.1275	-1.1829	-1.0972	-1.0662	0.0081
	303.15	-0.9614	-1.0176	-0.9201	-0.6515	0.0072
	308.15	-0.8138	-0.8251	-0.5479	-0.4664	0.0033
	313.15	-0.6765	-0.6978	-0.4518	-0.3465	0.0048
	318.15	-0.5848	-0.5960	-0.3458	-0.2001	0.0025
	323.15	-0.4854	-0.4805	-0.2236	-0.1038	0.0044
DMF+iso-Butanol systems	298.15	-1.4625	-1.4299	0.0376	1.0554	0.0119
	303.15	-1.1921	-1.1888	0.2447	1.4688	0.0105
	308.15	-0.8927	-0.7898	0.1579	0.9675	0.0090
	313.15	-0.7414	-0.6376	-0.0553	0.5365	0.0059
	318.15	-0.5674	-0.4423	-0.4183	-0.1406	0.0028
	323.15	-0.4578	-0.3704	-0.3611	-0.0125	0.0033
DMF+tert-Butanol systems	298.15	-1.8908	-2.5583	-1.5729	0.0317	0.0183
	303.15	-1.3734	-1.5060	-0.9708	-0.3360	0.0047
	308.15	-1.0017	-0.9210	-0.4410	-0.3749	0.0048
	313.15	-0.6992	-0.6679	-0.1468	0.2314	0.0040
	318.15	-0.4829	-0.3465	-0.0506	0.1244	0.0030
	323.15	-0.3397	-0.1859	0.0756	0.1218	0.0046

As seen in Figure 3, the values of  $\eta^E$  for all studied systems are negative over the entire range of mole fractions at all temperatures and the curves are asymmetrical in nature and skewed to the alcohol-rich range. Similar to the excess molar volumes ( $V^E$ ), viscosity ( $\eta$ ) is related to the molecular

interaction between the components of mixtures as well as of the size and shape of molecules. The negative excess viscosity supports the main factor of breaking of the self-associated alcohols and weak interactions between unlike molecules.

**Table 3.** Viscosities ( $\eta$ ), change of free energy ( $\Delta G^*$ ), excess free energy change ( $\Delta G^{*E}$ ) for the Binary Mixtures of n-Butanol at (298.15, 303.15, 308.15, 313.15, 318.15 and 323.15) K

$x_2$	$\eta$ mPa.s	$\Delta G^*$ kJ.mol <sup>-1</sup>	$\Delta G^{*E}$ kJ.mol <sup>-1</sup>	$x_2$	$\eta$ mPa.s	$\Delta G^*$ kJ.mol <sup>-1</sup>	$\Delta G^{*E}$ kJ.mol <sup>-1</sup>
(1) DMF + (2) n-Butanol							
<i>T</i> = 298.15K							
0.0000	0.793	12.5182	0.0000	0.5997	1.271	13.9271	-0.5868
0.1009	0.857	12.7489	-0.1050	0.7003	1.432	14.2612	-0.5874
0.1990	0.915	12.9535	-0.2269	0.7989	1.663	14.6699	-0.5069
0.2982	0.975	13.1509	-0.3597	0.9006	1.998	15.1600	-0.3551
0.4008	1.051	13.3789	-0.4731	1.0000	2.596	15.8460	0.0000
0.4999	1.151	13.6426	-0.5391				
<i>T</i> = 303.15K							
0.0000	0.754	12.6162	0.0000	0.5997	1.163	13.9489	-0.5527
0.1009	0.798	12.7958	-0.1376	0.7003	1.293	14.2555	-0.5623
0.1990	0.858	13.0230	-0.2189	0.7989	1.484	14.6395	-0.4883
0.2982	0.909	13.2061	-0.3476	0.9006	1.764	15.1141	-0.3335
0.4008	0.980	13.4402	-0.4361	1.0000	2.245	15.7601	0.0000
0.4999	1.053	13.6598	-0.5281				
<i>T</i> = 308.15K							
0.0000	0.709	12.6774	0.0000	0.5997	1.080	14.0053	-0.5011
0.1009	0.759	12.8945	-0.0907	0.7003	1.193	14.2993	-0.5140
0.1990	0.808	13.1014	-0.1830	0.7989	1.356	14.6653	-0.4487
0.2982	0.851	13.2740	-0.3128	0.9006	1.611	15.1490	-0.2752
0.4008	0.909	13.4848	-0.4150	1.0000	1.988	15.7273	0.0000
0.4999	0.982	13.7225	-0.4795				
<i>T</i> = 313.15K							
0.0000	0.667	12.7391	0.0000	0.5997	0.984	14.0086	-0.4831
0.1009	0.715	12.9640	-0.0700	0.7003	1.095	14.3252	-0.4605
0.1990	0.751	13.1391	-0.1816	0.7989	1.218	14.6434	-0.4305
0.2982	0.796	13.3289	-0.2816	0.9006	1.438	15.1157	-0.2553
0.4008	0.850	13.5437	-0.3667	1.0000	1.746	15.6616	0.0000
0.4999	0.909	13.7611	-0.4389				
<i>T</i> = 318.15K							
0.0000	0.631	12.8096	0.0000	0.5997	0.906	14.0280	-0.4558
0.1009	0.669	13.0067	-0.0846	0.7003	0.998	14.3269	-0.4378
0.1990	0.706	13.1971	-0.1681	0.7989	1.111	14.6496	-0.3903
0.2982	0.743	13.3778	-0.2643	0.9006	1.288	15.0815	-0.2423
0.4008	0.791	13.5854	-0.3431	1.0000	1.545	15.6013	0.0000
0.4999	0.838	13.7811	-0.4241				
<i>T</i> = 323.15K							
0.0000	0.597	12.877	0.0000	0.5997	0.835	14.0468	-0.4209
0.1009	0.633	13.0764	-0.0679	0.7003	0.920	14.3522	-0.3823
0.1990	0.662	13.2508	-0.1538	0.7989	1.008	14.6347	-0.3614
0.2982	0.697	13.4318	-0.2359	0.9006	1.164	15.0606	-0.2053
0.4008	0.736	13.6267	-0.3133	1.0000	1.363	15.5297	0.0000
0.4999	0.781	13.8261	-0.3767				

The more negative  $\eta^E$  of tert-Butanol arises due to breaking of H-bonds in the self-associated alcohol and steric hindrance due to the bulky groups. Unfavorable packing may, however, result due to disruption of the closely associated alkanols on addition of DMF and formation of new association between the unlike DMF and alkanol molecules and lead to negative excess viscosity.

Free energy of activation  $\Delta G^\ddagger$  for viscous flow of liquid mixtures of various composition and of the respective pure components was obtained with the help of Eyring equation [41-43, 44- 45]. The values of  $\Delta G^\ddagger$  have been listed in the Table 3 as sample for n-Butanol at the working temperatures of (298.15, 303.15, 308.15, 313.15, 318.15 and 323.15)K respectively covering the whole composition range at an interval of 5K. The excess free energy,  $\Delta G^{#E}$  were fitted to a Redlich- Kister polynomial equation (3). The excess free energy of activation for viscous flow,  $\Delta G^{#E}$  of the systems are shown in Table 3 as sample for n-Butanol. The values of the fitting parameters of  $\Delta G^{#E}$  along with the standard deviation are in the range, they are not shown in Table.

The negative excess free energies for all the systems are accounted for due to the dissociation of the associated structures of butanols in DMF. As pointed earlier excess viscosity and excess free energy follow the order: tert-Butanol > iso-Butanol > n- Butanol , which in turn reflect the extent of dissociation mainly due to dispersion forces. D. Aprano et al [46] calculated the Kirkwood correlation coefficient,  $I_k$ , from the dielectric constant values of pentanol isomers in the temperature range of 303.15 and 323.15K and found that the values of  $I_k$  vary in the order: 1-pentanol > 2-pentanol > 3-pentanol. Since  $I_k$  is a measure of the short range order in polar liquids , it follows that the pentanol whose  $I_k$  is larger i.e., which is more strongly bounded by H-bond, is less likely to be dissociated than the pentanol with smaller  $I_k$  i.e. which is less strongly bound by H-bonds.

This suggest that the relative ease of dissociation of the three Butanols either by thermal effect or by the force of dispersion in DMF should thus follow the order : tert-Butanol > iso-Butanol > n- Butanol. This is of course as expected in terms of the branching of the hydrocarbon moieties in the isomeric Butanols. This provides a satisfactory explanation for the relative thermal effect on the viscosity of Butanols (Fig 2) and relative magnitude of excess viscosities (Fig 3) as well as excess free energies of these systems. The negative values of  $\Delta G^{#E}$  of all the mixtures at all the compositions indicate that the flow process is facilitated by the mixing process.

#### IV. Conclusion

Density and viscosity of the binary mixtures of  $N,N'$  - Dimethylformamide (DMF), + n-Butanol, + iso-Butanol and + tert-Butanol were measured at temperatures (298.15 - 323.15) K covering the whole composition range.

Excess molar volumes, excess viscosities and excess free energy change of DMF + Butanol systems were found to be negative and large in magnitude. All the excess properties show minima in alcohol rich region. The disruption of H-bonds in Butanols either by thermal effect or by the force of disruption is in the order: tert-Butanol > iso-Butanol > n-Butanol. This effect is considered to be quite significant in explaining the temperature dependence of viscosity of pure Butanols, negative excess values of viscosity, and free energy for viscous flow as well as their orders for all the systems. Larger the branch of Butanol isomers, greater is decrease in the average degree of association, as a result more negative excess properties vs. mole fraction curve is observed.

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