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Physiochemical Properties of Binary Mixture of Methanol + Dimethylsulfoxide (DMSO) and Methanol + Dimethylformamide (DMF)

W. B. Shirsath

*Karm. A. M. Patil Arts, Commerce & Kai. Annasaheb N. K. Patil Science Sr. College, Pimplaner, Dist-Dhule, Maharashtra, India, shirsatsiramp@gmail.com

Abstract: **Physiochemical Properties of Densities and Viscosities of Pure Liquids Methanol (ME) and its binary mixtures with DMSO and DMF have been measured as a function of composition over the entire range at room temperature and atm. Pressure the excess volume V^E excess viscosity, viscosity deviation and interaction parameter have been calculated from experimental data as a function of composition. All the excess functions are found to the either positive or –ve over the entire range of composition depending upon the molecular interaction and nature of liquid mixture. The properties are discussed in terms of the molecular interaction between the component molecules. Neat FTIR spectra of were recorded on a FTIR spectrometer (Model: SIMADZU 8400S PC) by using KBr pellet in the region 400-4000 cm^{-1} with 4.0 cm^{-1} resolution. An UV Spectra were recorded on a UV 2400 PC in the wavelength range 200-400 nm using air as a reference. The DFT (B3LYP) calculations were performed using Gaussian 03W program package. Geometry of the individual and two molecules together was optimized at 6-31G (d) basis set to know the exact nature of intermolecular interactions.**

Index Terms: **Density, DFT, FT-IR, Molecular-interaction, Viscosity**

I. INTRODUCTION

Due to the recent developments made in the theories of liquid mixtures and experimental techniques, the study of binary liquid mixtures has attracted several researchers in the field (Nikam, P. S et al., 1988). The prediction of the viscosity of liquid mixture is a goal of long standing, with both theoretical and practical importance. A truly fundamental theory would predict the viscosity along with other thermodynamic and transport properties from the knowledge of the intermolecular forces and radial distribution function alone. Such a programme had appreciable success in application to pure simple liquids such as liquefied rare gases (Aminabhvi, T, M. et al.1998).

Binary liquid mixture formation is accompanied by increase, decrease or zero viscosity change. The non-ideal behaviors of binary associated solvents may be due to intermolecular interactions, which could be successfully interpreted with the help of excess thermodynamic functions.

Mixed solvents are often used in chemistry to modify molecular environment in order to modulate process. Physical properties of solvent mixture are often studied to get information about the mutual interaction between the solvent molecules. In the present study we have studied the excess volume V^E and deviation of viscosity ($\Delta\eta$).

Viscosity is an important property of liquid mixtures required for the design of flow system that are widely used in engineering application especially in heat exchanges as well as mass transfer equipment. The influence of H-bonding on solution properties is great interest to researchers.

In this study interaction of Methanol (ME) with DMSO and DMF has been investigated ME exist in associated form whereas DMSO exists in associated form whereas DMSO, DMF have associated / non-associated in the liquid state. When these liquids are mixed with each other mixing properties with varying intermolecular interaction may be generated.

To investigate this effect in the present investigation the density (δ) and η viscosity of binary mixtures of Methanol (ME) with DMSO and DMF have been studied over the entire range of composition at room temp.

DMSO was chosen because of wide range of applicability as a solvent in chemical and biological process involving in both plants and animals. It is highly polar aprotic solvent because of its S=O group and has a large dipole moment and relative permittivity (μ=3.9 D & ϵ = 46.6 at 298.15 K) Similarly DMF which is an aprotic polar solvent with a large dipole moment and relatively high dielectric constant. In pure state itself association through dipole-dipole interaction. DMF is biologically important and also very good solvent which is widely used in polymer science and pharmaceutical industry.

Alcohols are versatile solvent used a separation of saturated and unsaturated hydrocarbon and in pharmaceutical synthesis and also serve as solvents for many polymers. Alcohols are polar and self-associated through hydrogen bonding in the state.

Mixing properties such as excess molar volume deviation in viscosity have been used as qualitative and quantitative guide to understand molecular interaction between the components of the mixtures.

II. LITERATURE SURVEY

Densities, viscosities and their excess values of binary and multi-components have been investigated by many workers. The results have been interpreted in terms of differences in size of molecules and strength of specific or non-specific interactions taking place between the components of the mixtures.V^E, Δη, molar refraction, speed of sound and isentropic compressibility have computed by (Aminabhvi et al.1998) or the mixtures of ethyl benzene with dimethylamide, DMSO, DMF and tetrahydrofuran and dioxane at 298.03 K. V^E values were negative and these data vary with the second component of the mixtures. In case ethyl benzene + DMSO mixture, the Δη values were negative. For all the mixtures except ethyl benzene + 1,4-dioxane, Δη values were positive.

V^E, Δη, speed of sound, molar refractivity and isentropic compressibility have calculated by (Skrane et al.1995) for the mixtures of water with N, N-dimethylacetamide, DMSO, 1,4-dioxane, tetrahydrofuran, acetonitrile, ethylene glycol, diethylene glycol, 2-methoxyethanol 2-ethoxyethanol and DMF at 298.15 K. For all mixtures, the values of V^E were negative, suggested specific interactions between water and organic components of the mixture. The minima of the curves trend shifted towards the water-rich region of the mixtures. The values of Δη were positive for all the mixtures except at a few compositions in the case of mixtures of water with ethylene glycol, diethylene glycol, or acetonitrile. With these mixtures, the values of Δη were both positive and negative and magnitudes of Δη were small when Δη compared to those of other binaries. For a majority of the mixtures Δη shifted towards the water rich region.

V^E and excess refractive indices at 298.15 K for binary mixtures of DMF with benzene, toluene, cyclohexane, ethylbenzene, o-, m- and p-xylenes and styrene are reported

The V^E, Δη, excess free energy of activation flow and contact interaction parameter have been computed by Joshi et al. [4] for binary mixtures of nitrobenzene with cyclohexane, DMF from density and viscosity measurement in the temperature interval of 298.15 - 313.15 K. The V^E results for mixtures of nitrobenzene + cyclohexane was positive, suggested the existence of weak dispersion forces between the mixing components. This was further confirmed by negative values of Δη and ΔG. In case of mixture nitrobenzene + DMF, Δη were negative over the entire composition scale of the mixture. The negative V^E was attributed to dipole-dipole interactions between the components. The positive value of Δη and ΔG further confirmed specific interactions in the mixture.

Aminabhavi et al. measured densities and viscosities for fourteen binary liquid mixtures containing DMF, carbontetrachloride, cyclohexane, benzene, p-xylenes, methyethylketone and DMSO at 45^0C over the whole composition range.

The V^E, Δη, molar refraction, speed of sound and isentropic compressibility was calculated at 298.15, 303.15 and 308.15 K in binary mixtures of anisole with 2-chloroethanol, 1,4-dioxane, tetrachloroethylene, tetrachloroethane, DMF, DMSO and diethyl oxalate over the entire composition range. The positive V^E values indicated the repulsive forces operating between the component of the mixtures depending on their dipole moments and dielectric constants. The positive Δη value was observed for mixture of anisole + tetrachloroethane. It decreased as temperature increased. Molar refraction of anisole at 298.15 K was negative for all the mixtures. The negative values of speed of sound was observed in case of anisole + tetrachloroethylene, + tetrachloroethane, + diethyl oxalate and + 1,4-dioxane.

The densities and viscosities for binary mixtures methanol + DMF and ethanol + DMF were measured at 293.15 to 333.15 K and atmospheric pressure The results were used to calculate excess molar volume. Redlich-Kister equation was fitted to experimental data. (Bai et al.1999) reported V^E for binary and ternary mixtures of DMF +ME + water at 298.15 K as well as of DMF + ethanol+ water at 298.15 K.

The V^E values for mixtures of ME + DMF and the ethanol + DMF were reported by Zielkiewicz. For the system of ME with DMF, the absolute average deviation of the experimental V^E from values obtained with co-relation of the Redlich-Kister equation given by Wang et al. and Zielkiewicz under 9 mole fractions at 293.15 K is 12.6% and 2.1% respectively.

A perusal of literature reveals that the density, viscosity and spectroscopic studies of pharmaceutically important molecules in mixed solvents are scant, therefore in the present investigation I report the data for these systems over the entire composition range at room temperature.

The general objective of the work is to critically analyse some aspects of solution chemistry.

Specific objectives of the work are as follows:

- To study non-ideal behavior of liquid mixtures.
- To investigate kind of intermolecular interactions.
- Support conclusions drawn from the experimental data with theoretical.

III. EXPERIMENTAL SECTION

A. *Preparation of Binary Mixtures*

It is very important for viscosity measurements. Methanol (ME), DMSO and DMF were of standard grade (SRL) with purity near to 99% and used as such. Binary liquid mixtures of DMSO and DMF with Methanol (ME) (x_1) of compositions 0 – 100 mass % as well as $x_1 = 0.1$ to 0.9 were prepared by mixing a known mass of each liquid in an airtight stopper bottle. The masses were recorded on a digital balance (SHIMADZU AUX 220) to an accuracy of $\pm 1 \times 10^{-5}$ g. No attempt was made to monitor the relative humidity of the air to which the liquids were exposed for a short period of time during preparation. The mole fraction of each component was calculated. The estimated error in mole fraction was $< 1 \times 10^{-4}$. Care was taken to avoid contamination during mixing**.**

Table 1. Comparison of experimental data of ρ and η of solvents with literature at different temperatures.

Solvent	**Temp. K**	ρ		η	
		Expt.	**Lit.**	**Expt.**	**Lit.**
ME	298.15	0.7863	0.7863[a], 0.7864[b], 0.7866[c]	0.547	0.549[a], 0.551[b]
	303.15	0.7820	0.7820[d,] 0.7819[e], 0.7816[f]	0.513	0.516[d], 0.515[f]
	308.15	0.7769	0.7771[b], 0.7770[g]	0.472	0.469[b], 0.479[g]
DMSO	298.15	1.0956	1.0953[h], 1.0959[i], 1.0956[j,] 1.0957 [k, l] 1.0958[m, n]	1.994	1.991[h] ,1.991[i], 1.996[j], 1.991[k, l], 1.996[m, n]
	303.15	1.0885	1.0853[h], 1.0896[o]	1.626	1.654[h], 1.798[o]
	308.15	1.0854	1.0853[h], 1.0855[i], 1.0855o, 0856[p]	1.650	1.654[h], 1.644[i],1.654[o], 1.645[p]
DMF	298.15	0.9444	0.9442[m] ,0.9449[n], 0.9442[q], 0.9445[s], 0.9445[t]	0.803	0.802[m], 0.805[n]0.801[r], 0.802[s], 0.802[t]
	303.15	0.9396	, 0.9398[s,] 0.9397[t]	0.758	0.756[s,] 0.762[t]
	308.15	0.9351	0.9356[s], 0.9356[t]	0.709	0.707[s], 0707[t]

([a]Tu C.H., et al.,2001; [b]Marigliano, A.C.et al.,2002; [c]Yang, C.et al.,2008; [d]Nikam, P. S., et al.2000; [e]Yang, C.et al.,2006; [f]Rajwade, R. P., et al.,2008; [g]Pal, A., et al.,2005; [h]Nikam, P. S., et al.,2003; [i]Nikam, P. S., et al.,2000)

Densities of liquids/mixtures were measured by weighing a definite volume of the liquid in specific gravity bottle having a volume about of10 c.c. The specific gravity bottle was calibrated with distilled water at room temp. The estimated uncertainty in density measurement of pure liquids and binary mixtures was $\pm 1 \times 10^{-4}$ gcm^{-3}. From the known density of water at room temperature and the weight of the water taken in specific gravity bottle, corresponding volume of water was calculated. It was then filled with experimental liquids. Masses of liquids under investigation were obtained and the corresponding densities thus determined (Nikam P.S.et al.,1998).

B. *Viscosity measurements by capillary flow method*

The time required when liquid flow through capillary under specified condition can be used to determine the viscosity of the liquid. This method is based on Poiseuille's principle. In the present study, this method was employed to determine the viscosity of fluid at various temperatures. The usual form of this simple, yet accurate, apparatus known as Ostwald viscometer was used for comparing viscosities of different liquids. The absolute viscosity of the liquid can then be obtained knowing the viscosity of the standard liquid. The viscometer was calibrated with triple distilled water at room temperature (vary with seasons) using the relation (Nikam, P. S et al.,1998) shown in Eq. (1)

$$\eta = \rho(At - B/t) \qquad (1)$$

Where A and B are the viscometer constants and t is the time of flow. All flow times were greater than 100s. The force driving the liquid through the capillary in Ostwald viscometer is equal to $\rho_1 hg$ where 'h' is mean difference of level of liquid in the two limbs of the tube, 'ρ_1' is the density of the liquid and 'g' is gravitational constant. The resistance of flow depends on the dimensions of the capillary which are constant and on the viscosity of the liquid.

If now, the same volume of a second liquid of known viscosity is introduced in the tube, the mean difference of level of the two liquid surfaces will also be h, so that the driving force is now $\rho_2 hg$.

Thus, the driving force is proportional to their viscosities. Since the rate of flow is proportional to force/resistance, the times of outflow (t_1 and t_2) for the same volume of the two liquids are in the inverse ratio as shown in Eq. (2)

$$t_1 / t_2 = (\eta_1/\rho_1) / (\eta_2/\rho_2) \text{ OR } \eta_1/\eta_2 = (\rho_1/t_1) / (\rho_2/t_2) \qquad (2)$$

Thus, if the absolute viscosity of one liquid is known, that of the given liquid can be determined. Since the rate of flow through a capillary tube depends on ‘r’ (internal radius of capillary) can vary from 0.2 to 2.0 mm, Ostwald viscometer can be made to cover a range of 10^4 in viscosities. A small error may arise in these measurements due to the change of volume of the liquid owing to expansion. But this may be neglected provided that during the out-flow period, the lower meniscus lies inside the bulb ‘E’ so that the change of level is small. The estimated error of viscosity measurement was ± 0.1 or 0.2%. The uncertainties in dynamic viscosities were of the order ± 0.003 m.Pas. The difference reading did not deviate from the mean by more than 0.2 s. Experimental densities and viscosities of solvents were compared with those reported in the literature. From the observation of Table I, it could be said that, our experimental values matched very well with those of literature.

The experimental data on δ & η for binary mixtures of DMSO & DMF with Methanol **(**ME), at room temperatures were used to determine the excess mole volume V^E using the relation shown in Eq. (3)

$$V^E = x_1 M_1 [1/\rho_m - 1/\rho_1] + x_2 M_2 [1/\rho_m - 1/\rho_2] \qquad (3)$$

ρ_1 and ρ_2 are densities of pure components and Pm = densities of mixture, x_1, x_2, M_1, M_2 are mole fraction and molecular weight.

Deviation in viscosity ($\Delta\eta$) were obtained as follows in Eq. (4)

$$\Delta\eta = \eta_{mix} - (\eta_1 x_1 + \eta_2 x_2) \qquad (4)$$

C. IR measurements

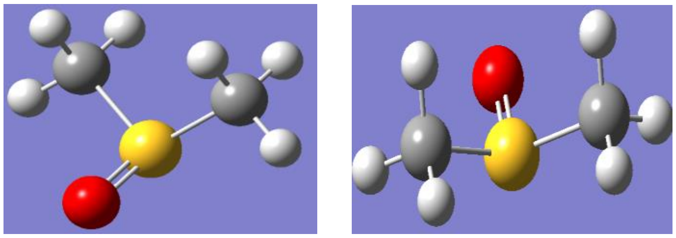
FTIR spectra of experimental solvent systems were recorded on a FTIR spectrometer (Model: SIMADZU 8400S PC) by using KBr pellet in the region 400-4000 cm^{-1} with 4.0 cm^{-1} resolution. The transmission values were read in steps of 5%. The spectrometer possesses out to aligned energy optimization and dynamically aligned interferometer. It is fitted with KBr beam splitter, a DLATGS detector. A base line correction was made for the spectra recorded. The FTIR – 8400S relies on an interferometer to perform infrared measurement and a personal computer to control the interferometer and process the data. Selected stretching frequencies of solvents used in this work are listed in Table II.

Table II. Selected neat IR frequencies of solvents

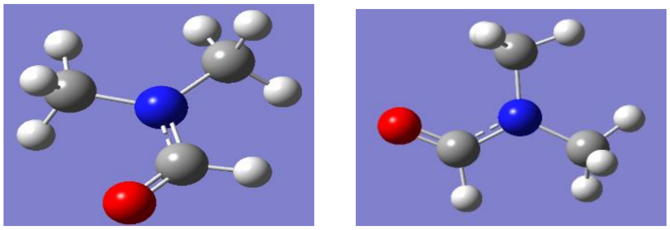
ME		**DMSO**		**DMF**	
Group	**ν**	**Group**	**ν**	**Group**	**ν**
O-H	3570, 3450	S=O	1051.19	C=O	1752
C-H	2833.00	C-H	2995 , 2963	C-H	2944.35
C-H	2945.40	--	--	--	--
C-O	1030.02	--	--	--	--

D. Optimization of geometries using Gaussian Package

Geometry optimizations of heterocyclic compounds, pure solvents and their 1:1 and 1:1:1 complex were carried out using the DFT/B3LYP method with medium size 6-31G (d) basis set available in G03 (W) series of programs. All optimized geometries were viewed through Gauss View 4.1 software. Fig.1 includes optimized geometries of solvents and solutes under investigation.



DMSO



DMF

Fig. 1. Optimized geometries of DMSO and DMF using B3LYP/6-31G(d) basis set

IV. RESULT AND DISCUSSION

Methanol **(**ME) is self-associated through hydrogen bonding, thus on mixing Methanol **(**ME), with DMSO/DMF, interesting properties may be observed and hence densities and viscosities studies of binary liquids mixtures of ME with these co-solvents are presented. Experimental results of densities and viscosities for the binary mixtures of Methanol **(**ME) with DMSO and DMF are included in Table III.

Excess molar volumes (V^Es) and deviation in viscosities ($\Delta\eta$s) were calculated from the measured densities and viscosities of the pure components and the binary mixtures using Eq. (3) and (4) respectively. The variations of V^Es with x_1 for ME (x_1) + DMSO ($1 - x_1$) and ME (x_1) + DMF ($1 - x_1$) are represented in Fig. 2 and 3 respectively. The value of V^E for binary mixtures are either positive or negative depends upon physical and chemical interactions (Nikam, P. S., et al.,2000).

Physical interactions make positive contribution mainly consisting of dispersion forces. Chemical or specific interactions which include charge transfer, geometrical fitting between components and hydrogen bonding formation making negative contribution. Mixing of DMSO/DMF with Methanol **(**ME) tends to breaks the association present in Methanol **(**ME) molecule with a subsequent increase in V^E. However, because of simultaneous interaction mainly due to hydrogen bonding between DMSO/DMF and Methanol **(**ME) molecules, in addition to the interstitial accommodation due to difference in shape and size of component molecule, resulting in overall decrease in the value of V^E with mole fraction of x_1.

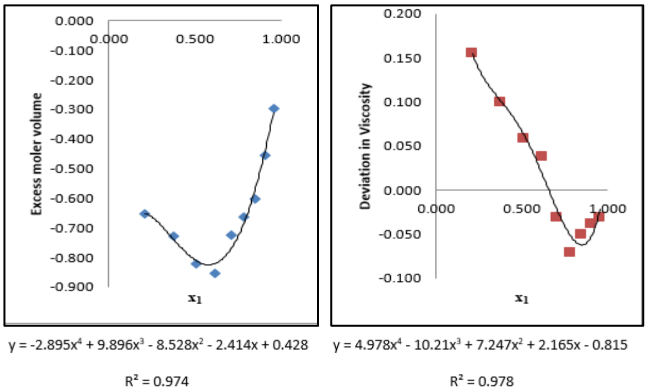


Fig. 2. Excess functions of ME + DMSO at 303.15 K

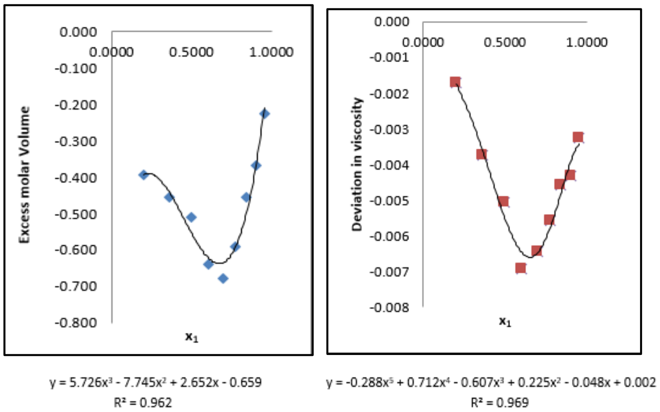


Fig. 3. Non-ideal behaviours of ME + DMF at 308.15 K

The interaction between Methanol **(**ME) and DMSO/DMF most obvious lead to weak dispersion type and hydrogen bond effects possibly due to S=O /C=O and OH groups. Rather large negative V^E for Methanol **(**ME) +DMSO between $x_1 \approx 0.5\text{-}0.6$ indicates the existence of strong intermolecular interaction between DMSO and Methanol **(**ME). With increasing x_1, the interaction between unlike molecules becomes less important and so V^E is less negative. A correlation between signs of $\Delta\eta$ and V^E has been observed for a number of binary solvent systems i.e., $\Delta\eta$ being negative where V^E is positive or vice-versa (Comelli, F., et al.,2006).

In general, for systems, where dispersion and dipolar interaction are operating η^E values are found to be negative. Negative $\Delta\eta$ may be attributed to unequal molecular sizes of the constituent molecules of the mixture where dispersion forces are dominant. Variation of $\Delta\eta$ with x_1 (ME + DMSO) yields a sigmoid nature. The sigmoid nature of the graph is attributed to the equilibria of state effect and steric factor arising from the change of orientation of DMSO with increase in the mole fraction of ME. For system ME + DMF, $\Delta\eta$ values are negative for the whole composition range, in general for systems where dispersion and dipolar interactions are operating. However, the magnitude of this deviation is very small i.e., negligible interactions of this type are operating in the mixture. Similar results are reported in the literature for ME + DMF and other systems.

Table III. Experimental ρ and η of binary systems

ME + DMSO at 303.15

x_1	ρ	η
0.212	1.0580	1.541
0.379	1.0214	1.297
0.510	0.9883	1.107
0.619	0.9560	0.964
0.709	0.9231	0.793
0.785	0.8932	0.667
0.850	0.8650	0.614
0.907	0.8367	0.562
0.956	0.8096	0.514

ME + DMF at 308.15 K

x_1	ρ	η
0.202	0.9174	0.655
0.363	0.9020	0.618
0.495	0.8887	0.587
0.603	0.8749	0.561
0.695	0.8603	0.541
0.774	0.8442	0.525
0.842	0.8275	0.511
0.901	0.8121	0.498
0.954	0.7961	0.487

A. *IR and theoretical studies*

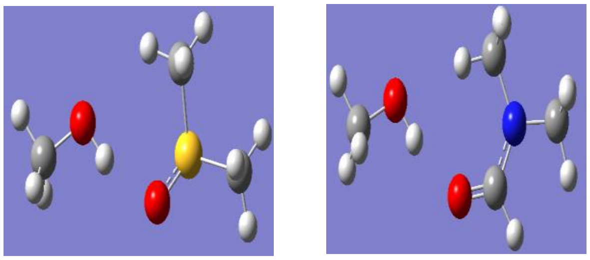
FTIR stretching frequencies of some functional groups of mixtures of Methanol (ME), with DMSO and DMF are listed in Table IV for ME + DMSO, maximum νOH (at low x_1) decreased upon dilution ($x_1 \approx 0.2\text{-}0.9$). The magnitude of all these frequencies is higher than that for pure Methanol (ME), this may be due to the breaking of Methanol (ME) aggregates making more free monomers of it in the system. The νS=O of DMSO disappeared at 1051.19 cm^{-1} in all compositions and appeared at a lower but almost constant value suggesting hetero association between Methanol (ME) and DMSO. In case of ME + DMF, both νOH and νC=O decreased with the increase in the amount of x_1 in the binary system suggesting breaking of aggregates of Methanol (ME) as well as DMF. An aldehyde νC-H at 2944 cm^{-1} moved down upon addition of co- solvent indicating decreased double bond character of C=O in presence of ME. The hydrogen bond formation between hydroxy proton of ME and oxygen of O=S (DMSO) / O=C (DMF) is further confirmed by the theoretical investigation. Fig. 4 depicts minimum energy structures for 1:1 complex of components of binaries using our legal licensed copy G03 (W) software. From above studies, hetero intermolecular interactions are in the following order: ME + DMSO > ME + DMF.

Table IV. Selected IR frequencies for binaries
ME (x_1) + DMSO (1-x_1)

x_1	OH	S=O
0	--	1051.19
0.1	3387.11	1031.95
0.2	3381.33	1031.95
0.3	3379.4	1031.95
0.4	3379.4	1030.02
0.5	3377.47	1030.02
0.6	3373.61	1030.02
0.7	3358.18	1030.02
0.8	3346.61	1030.02
0.9	3346.61	1030.02
1	3352.39	--

ME (x_1) + DMF (1- x_1)

x_1	OH	C=O	C-H (Aldehyde)
0	--	1675.00	2944.35
0.1	3458.48	1674.27	2931.90
0.2	3439.19	1674.27	2933.83
0.3	3433.41	1672.69	2935.76
0.4	3419.9	1672.34	2935.76
0.5	3421.83	1664.62	2935.76
0.6	3387.11	1668.48	2939.61
0.7	3365.9	1668.48	2941.54
0.8	3352.39	1670.41	2943.47
0.9	3352.39	1648.48	2943.47
1	3352.39	--	--



ME + DMSO ME + DMF

Fig. 4. Optimized geometry of Hydrogen bond interactions: ME with DMSO .

CONCLUSION

Experimental data of density and viscosity of pure liquids and in their binary mixtures have been measured at room temp. The data have been used to compute excess properties of systems. Positive and negative deviations are observed for V^E and $\Delta\eta$ respectively.

This work reports the experimental data δ and η for two binary systems (ME + DMSO & ME + DMF) at room temp.

A good agreement was found between our data and literature results of density and viscosity of pure liquids, V^E and viscosity deviation ($\Delta\eta$).

FTIR spectra of were recorded on a FTIR spectrometer (Model: SIMADZU 8400S PC) by using KBr pellet in the region 400-4000 cm^{-1} with 4.0 cm^{-1} resolution. The DFT (B3LYP) calculations were performed using Gaussian 03W program package. Geometry of the individual, two and three molecules together was optimized at 6-31G (d) basis set to know the exact nature of intermolecular interactions.

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