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Physico-chemical properties of binary systems involving modifier (methanol or propionic acid) and an acidic extractant di (2-ethylhexyl) phosphoric acid (DEHPA): volumetric, acoustic and viscometric routes

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ABSTRACT

In solvent extraction, a suitable modifier, basically polar liquid is used with extractant like DEHPA, TBP, TOPO and MIBK to enhance the efficiency in extraction processes. This paper is related to the study of physico-chemical properties of polar – polar binary mixtures at 303.15K and 0.1 MPa. Molar volume, free volume, isentropic compressibility, intermolecular free length, specific acoustic impedance, relaxation time, Rao's constant, Wada's constant, absorption coefficient have been calculated from the experimentally measured data of density, ultrasonic velocity and viscosity of pure components and binary mixtures of methanol/ propionic acid + DEHPA. In addition, excess molar volume, excess Gibb's energy of activation of viscous flow, deviations in viscosity, isentropic compressibility, free volume, intermolecular free length and acoustic impedance were also computed from the experimental data. The observed variations of excess/deviation functions with the composition of DEHPA have been discussed in terms of molecular interaction between unlike molecules in two binary mixtures due to chemical, physical and structural effects. It is found that the molecular interaction of methanol with extractant DEHPA is better than that of propionic acid and so methanol may be used as a suitable modifier with DEHPA in the solvent extraction process.

Keywords: *DEHPA; methanol; propionic acid; excess properties; molecular interaction*

1. INTRODUCTION

The study on physico-chemical properties of liquid mixtures is of considerable importance in several industrial, engineering and technological processes [1-3]. The knowledge of fundamental data, *viz.* density, viscosity and acoustic properties of liquid mixtures is of considerable importance in the illumination of the structural properties of molecules and helpful in industrial applications [4-6]. Several researchers have investigated the nature and strength of molecular interactions in liquid mixtures by employing various macroscopic and microscopic techniques, such as ultrasonic absorption, studying fluctuation in refractive indices, dielectric relaxation by using microwave techniques, Raman spectroscopy, Fourier Transform Infrared, ultraviolet visible and nuclear magnetic resonance [7-9]. This work presents a comparative study on physico-chemical properties of binary mixtures involving modifier (methanol and propionic acid) and acidic extractant, di (2-ethylhexyl) phosphoric acid (DEHPA) at temperature 303.15K and pressure of 0.1 MPa. Di-(2-ethylhexyl) phosphoric acid (DEHPA) is an acidic extractant, used in the chemical, hydrometallurgical and nuclear processing industries and is a highly effective extractant and used in the solvent extraction of uranium and rare-earth elements, zinc, copper, lead, vanadium, yttrium, cobalt, beryllium and other valuable metals [10-12]. The extraction efficiency of the extractant improves with addition of suitable diluents and modifiers and furthermore, studies on fundamental data such as density and viscosity of liquid mixtures are crucial in the extraction process on the molecular environment as well as molecular interaction [13-15].

In continuing effect to explore better polar modifier between two sets of modifiers [16, 17], i.e. 1- alkanols and monocarboxylic acids, various interaction parameters are reported in this paper. The discussions of these parameters are elucidated in terms of intermolecular interactions between components of binary liquid mixture. The degree and nature of the variation depend on the polarity and molecular size of the individual component. The results of our measurements of density (*ρ*), viscosity (*η*) and ultrasonic velocity(*U*) of DEHPA + 1-alkanols [16] and DEHPA + monocarboxylic acids [17] are used to calculate various interaction functions. From the measured values of U, ρ and η of binary mixtures of methanol/ propionic acid + DEHPA, various interaction parameters such as molar volume (V), free volume (V_f), isentropic compressibility (β_S), intermolecular free length (L_f), specific acoustic impedance (Z), relaxation time (τ), Rao's constant (R), Wada's constant (W), absorption coefficient (α / f^2), excess molar volume (V^E), excess Gibb's energy of activation of viscous flow (ΔG^{*E}), deviations in viscosity ($\Delta\eta$), isentropic compressibility ($\Delta\beta_S$), free volume (ΔV_f), intermolecular free length (ΔL_f) and acoustic impedance (ΔZ) have been computed. The variations in deviation/excess functions over the entire composition range of DEHPA have been discussed in terms of molecular interactions between unlike molecules. The main theme of the present article is a comparison study of molecular interactions between two polar modifiers (methanol and propionic acid) with polar acidic extractant (DEHPA).

2. MATERIALS AND METHODS

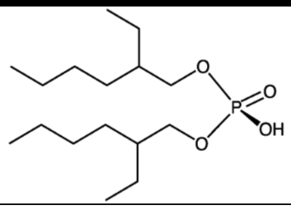
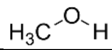
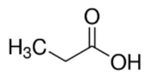
2.1. Materials.

The chemicals used are of analytical reagent (AR) grade and reported in Table 1. The measured values of ultrasonic velocity (U), viscosity (η) and density (ρ) of the pure samples were checked by comparing with the literature values [18-21] and show fairly well with literature data.

2.2. Properties measurements.

The binary liquid mixtures of methanol / propionic acid + DEHPA over the entire mole fraction range of DEHPA were prepared in air-tight bottles by mass measurement. Adequate precautions were taken to avoid evaporation and environmental damages. The details concerning the apparatus, experimental set-up and operational procedures for measurements of ultrasonic velocity, density and viscosity of liquid mixtures are described previously [22, 23]. In all measurements, temperature was maintained within ± 0.1 K by an electronically controlled thermostatic water bath. The measurements of ρ, U, η for each sample were measured thrice at 303.15K and 0.1 Mpa and average values were taken.

Table 1. Provenance, purity, CAS number, molecular mass and structure of the samples

Chemicals used	Provenance	Mass fraction purity	CAS number	Molecular mass (g mol^{-1})	Structure
DEHPA ($C_{16}H_{35}O_4P$)	Spectrum	0.98	298-07-7	322.43	
Methanol (CH_4O)	Aldrich	0.99	67-56-1	32.04	
Propionic acid ($C_3H_6O_2$)	E- Merck	0.99	79-09-4	74.08	

3. RESULTS AND DISCUSSION

In continuation to our earlier works [16, 17], the interaction parameters such as isentropic compressibility (β_S), intermolecular free length (L_f), specific acoustic impedance (Z), molar volume (V), free volume (V_f), relaxation time (τ), Rao's constant (R), Wada's constant (W) and absorption coefficient (α / f^2) have been computed from the experimental data of density (ρ), viscosity (η) and ultrasonic velocity (U) of binary mixtures of methanol/ propionic acid with di(2-ethylhexyl) phosphoric acid (DEHPA) by using standard relations [23-26] and are presented in Table 2.

The deviations in isentropic compressibility $(\Delta\beta_s)$, intermolecular free length (ΔL_f), acoustic impedance (ΔZ), free volume (ΔV_f) and viscosity $(\Delta\eta)$ data have been calculated using the following relation and displayed graphically in Figs. 1 to 5.

$$\Delta Y = Y_{mix} - Y_{ideal} \quad (1)$$

where Y_{mix} corresponds to the values of different acoustic parameters, i.e. β_s, L_f, Z, V_f and η of binary liquid mixtures and $Y_{ideal} = \sum_{i=1}^{2} X_i Y_i$ represents the value of components of the binary mixtures, X_i is the mole fraction and Y_i is the acoustic parameter of i^{th} components.

Excess molar volume (V^E) and excess Gibbs free energy of activation (ΔG^{*E}) have been calculated by using the following equations [23-26] and graphically represented in Figs. 6 and 7.

Excess molar Volume,

$$V^E = X_1 M_1 \left(\frac{1}{\rho} - \frac{1}{\rho_1} \right) + X_2 M_2 \left(\frac{1}{\rho} - \frac{1}{\rho_2} \right) \quad (2)$$

where X_1 and X_2 are mole fractions, M_1 and M_2 are the molar masses and ρ_1 and ρ_2 are the densities of pure components 1 and 2 of binary mixtures respectively.

Excess Gibbs free energy of activation,

$$\Delta G^{*E} = RT \, [\ln \eta V - \sum_{i=1}^{2} X_i \ln \eta_i V_i] \quad (3)$$

where R is universal gas constant. η_i and V_i are the viscosity and molar volume of i^{th} components, respectively.

From Table 2, it is observed that the values of isentropic compressibility (β_s), intermolecular free length (L_f), free volume (V_f) have been decreased in both methanol and propionic acid systems. Furthermore, specific acoustic impedance (Z), molar volume (V), relaxation time (τ), molar sound velocity/ Rao's constant (R), molar compressibility / Wada's constant (W) and absorption coefficient (α / f^2) have been increased in both systems with an increase in DEHPA concentration. The decreasing values of L_f, β_s , V_f and increasing values of Z, W, R, α / f^2, τ with the mole fraction of DEHPA suggest that there is a significant interaction between the modifier (methanol/ propionic acid) and DEHPA molecules and shows a structure promoting behaviour and suggest that the molecular association between unlike molecules is stronger than that of like molecules [4-6].

Table 2. Mole fraction of DEHPA (X_2), isentropic compressibility (β_S), intermolecular free length (L_f), specific acoustic impedance (Z), molar volume (V), free volume (V_f), Rao's constant (R), Wada's constant (W), absorption coefficient (α/f^2) and acoustic relaxation time (τ) of binary mixture {methanol / propionic acid (X_1 = 1-X_2) + DEHPA (X_2)} at T = 303.15 K and at p = 0.1 MPa.

X_2	$\beta_S . 10^{10}$ m^2N^{-1}	$L_f . 10^{11}$ m	$Z . 10^{-6}$ $N\ m^{-3}\ s$	$V . 10^6$ $m^3\ mol^{-1}$	$V_f . 10^8$ $m^3\ mol^{-1}$	$R . 10^3$ $m^3\ mol^{-1}$ $(m\ s^{-1})^{1/3}$	$W . 10^3$ $m^3\ mol^{-1}$ $(m^2N^{-1})^{-1/7}$	$\alpha/f^2 . 10^{13}$	$\tau . 10^{12}$ s
methanol + DEHPA									
0	10.839	6.833	0.845	0.410	6.327	0.421	0.782	0.134	0.740
0.0413	10.020	6.572	0.911	0.529	2.413	0.546	1.022	0.325	1.803
0.0751	9.606	6.433	0.945	0.628	1.727	0.649	1.22	0.476	2.658
0.1088	9.287	6.325	0.971	0.727	1.361	0.752	1.418	0.637	3.582
0.1723	8.835	6.169	1.009	0.913	0.972	0.949	1.794	0.978	5.566
0.2681	8.331	5.991	1.051	1.194	0.839	1.248	2.366	1.362	7.886
0.3639	7.934	5.846	1.085	1.475	0.808	1.551	2.944	1.667	9.821
0.4602	7.597	5.721	1.114	1.758	0.828	1.859	3.531	1.89	11.330
0.5442	7.348	5.626	1.135	2.005	0.896	2.13	4.045	2.051	12.470
0.6161	7.177	5.561	1.148	2.219	0.884	2.367	4.517	2.175	13.391
0.7023	6.929	5.464	1.171	2.473	0.942	2.652	5.05	2.252	14.070
0.8292	6.623	5.342	1.201	2.849	1.011	3.075	5.856	2.376	15.161
0.928	6.392	5.248	1.224	3.141	1.059	3.409	6.479	2.455	15.910
1	6.222	5.177	1.243	3.354	1.134	3.654	6.93	2.442	16.002
propionic acid + DEHPA									
0	8.105	5.909	1.101	0.755	10.01	0.784	1.501	0.171	0.973
0.07	7.724	5.769	1.126	0.934	7.355	0.979	1.872	0.247	1.442
0.13	7.467	5.672	1.144	1.089	5.55	1.148	2.192	0.335	1.991
0.23	7.131	5.543	1.169	1.347	4.294	1.432	2.73	0.469	2.853
0.32	6.913	5.457	1.186	1.58	3.248	1.688	3.216	0.641	3.963
0.47	6.659	5.356	1.206	1.969	2.233	2.119	4.03	0.984	6.215
0.51	6.601	5.332	1.211	2.073	2.063	2.234	4.248	1.082	6.864
0.59	6.505	5.294	1.219	2.282	1.79	2.465	4.685	1.288	8.239
0.66	6.44	5.267	1.225	2.464	1.623	2.667	5.067	1.469	9.446
0.71	6.405	5.253	1.227	2.595	1.525	2.812	5.34	1.602	10.331
0.76	6.371	5.239	1.231	2.726	1.43	2.957	5.614	1.746	11.301
0.81	6.335	5.224	1.233	2.857	1.353	3.102	5.888	1.887	12.250
0.88	6.292	5.206	1.237	3.04	1.275	3.305	6.272	2.073	13.510
0.94	6.257	5.192	1.240	3.197	1.207	3.479	6.6	2.246	14.680
1	6.222	5.177	1.243	3.354	1.134	3.654	6.93	2.442	16.002

The isentropic compressibility, intermolecular free length, free volume decreases and specific acoustic impedance increases with increase in DEHPA concentration (X_2) suggests that (i) there is a significant interaction between components of binary mixtures and (ii) a structure promoting behavior with the increasing mole fraction of DEHPA.

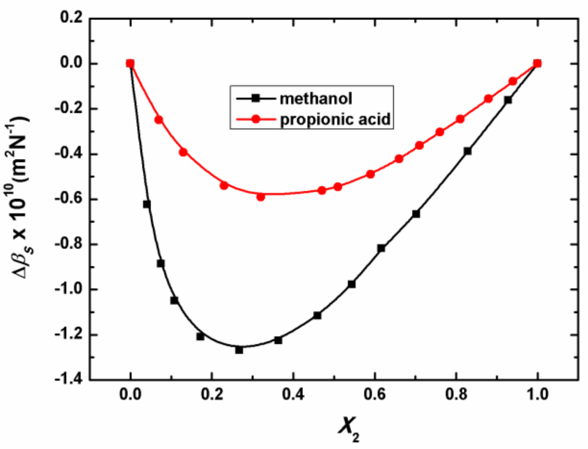


Figure 1. Deviation in isentropic compressibility ($\Delta\beta_s$) versus mole fraction (X_2) of DEHPA.

This is supported by other mentioned parameters discussed herewith. It is seen from Table 2 that the variation in molar sound velocity, molar compressibility and acoustic relaxation time is linearly increasing with increasing concentration of DEHPA for both binary mixtures. The increasing trends of the three parameters with an increase in concentration suggest the availability of more components in a given region that leads to close packing of the medium, eventually increases the molecular interaction [23, 24].

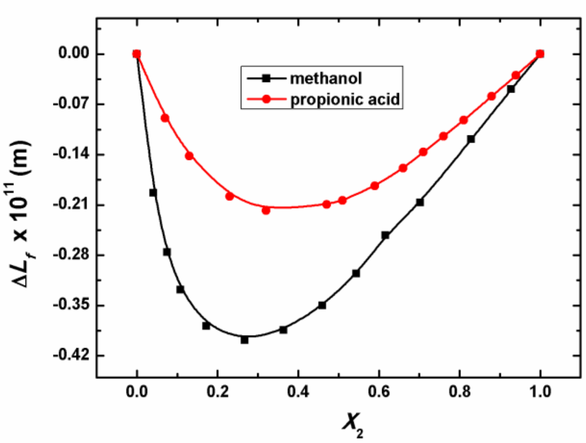


Figure 2. Deviation in intermolecular free length (ΔL_f) versus mole fraction (X_2) of DEHPA.

In order to have a more clear picture of the strength of molecular interactions between components of the liquid mixtures, it is of interest to discuss the deviation/excess properties. The deviation of a parameter from ideality is an indication of the presence of interaction between components of liquid mixture and the magnitude with sign of the parameter is a measure of interaction [7-9].

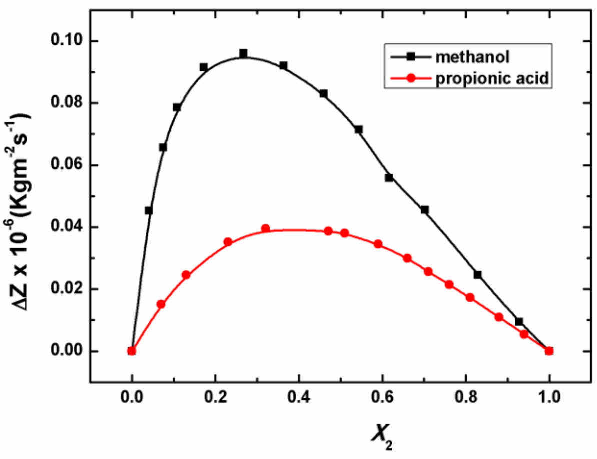


Figure 3. Deviation in acoustic impedance (ΔZ) versus mole fraction (X_2) of DEHPA

The magnitude of deviation depends upon the nature and concentration of the constituents of the mixtures. The deviations in isentropic compressibility $(\Delta\beta_s)$, intermolecular free length (ΔL_f), acoustic impedance (ΔZ), free volume (ΔV_f), viscosity $(\Delta\eta)$ and excess volume (V^E), excess Gibb's energy of activation flow (ΔG^{*E}) data have been calculated using the standard relations stated above and displayed graphically in Figs. 1 - 7.

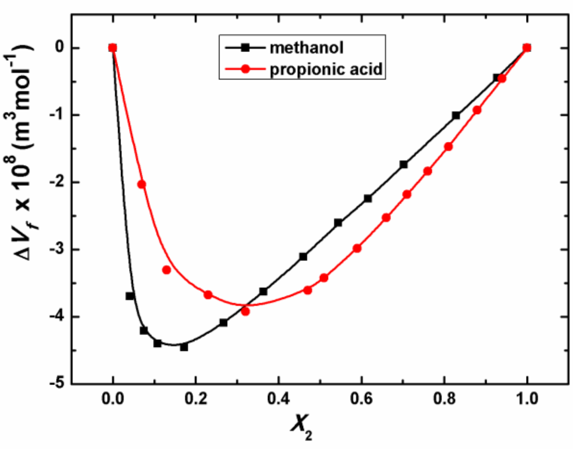


Figure 4. Deviation in free volume (ΔV_f) versus mole fraction (X_2) of DEHPA

Figs. 1-4 show that the trend of isentropic compressibility, intermolecular free length, free volume are negative and acoustic impedance are positive for both systems under study over the whole composition range, indicate the presence of specific interactions [1-5]. The negative values of $\Delta\beta_s$, ΔL_f, ΔV_f and positive value of ΔZ indicate that the liquid mixture is less compressible than the pure liquids forming the complex and molecules in the mixture are more tightly bound than in pure liquids. This corroborates the presence of relatively strong molecular interaction, possibly through hydrogen bonding between unlike molecules [24, 25]. The strength of the interaction between the component molecules of the mixtures as observed: methanol + DEHPA > propionic acid + DEHPA.

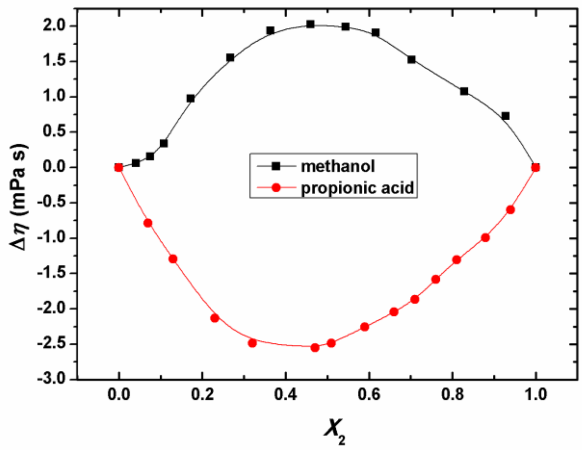


Figure 5. Deviation in viscosity $(\Delta\eta)$ versus mole fraction (X_2) of DEHPA

The trend of deviation in viscosity (Fig. 5) for methanol + DEHPA is positive whereas propionic acid + DEHPA is negative. In general, this function depends on two factors: (i) specific interactions between unlike components such as hydrogen bond formation and charge transfer complexes may cause for an increase in viscosity in mixtures than in pure components and (ii) geometrical difference in size and shape of component molecules and loss of dipolar association in a pure component may contribute decrease in viscosity in mixtures than in pure components. The first factor produces positive deviation in viscosity and second factor leads to negative deviation in viscosity [5-8, 25]. Thus it reveals that the molecular interaction in methanol + DEHPA is higher than propionic acid + DEHPA.

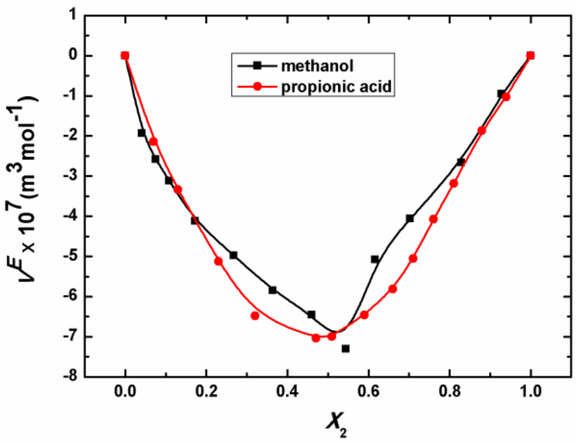


Figure 6. Excess molar volume (V^E) versus mole fraction (X_2) of DEHPA.

The plot of V^E with mole fraction X_2 for the binary mixtures under study is depicted in Fig. 6 and the variation is negative in both mixtures over the whole composition range. In general the excess molar volume is negative when the interactions between unlike molecules are stronger than the interaction between like molecules [24-26]. In both binary systems, on mixing of DEHPA with methanol and propionic acid may induce mutual dissociation of the H-bonded structures present in both modifiers with subsequent formation of new H-bonds (P = O---H – O) between phosphoryl group of DEHPA and hydroxyl group of both modifiers resulting in negative values of V^E in methanol (1) + DEHPA (2) and propionic acid (1) + DEHPA (2) mixtures. The magnitude of maximum value of V^E is slightly more in methanol + DEHPA from propionic acid + DEHPA. The excess molar volumes are influenced by mainly two factors [2-5, 24]: (i) loss of dipolar association and differences in size and shape and (ii) dipole-dipole, dipole – induced dipole interaction and charge transfer complex formation between unlike molecules. The former effect leads to expansion in volume and the later contributes to contraction in volume [2-5, 24]. However the value of V^E depends on the balance effect between these two opposing contributions. The experimental data in the present study suggest that the above second effect is dominant and makes contraction in volume in both binary mixtures.

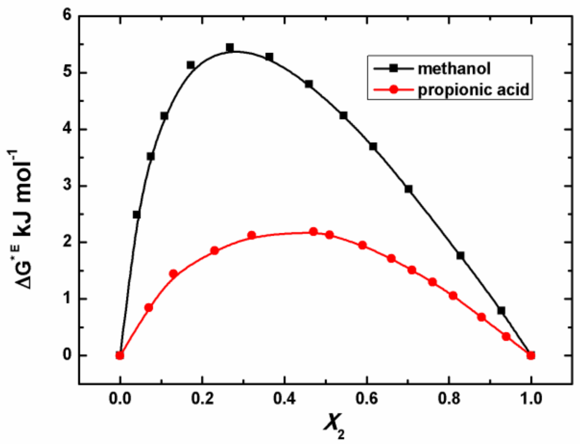


Figure 7. Excess Gibb's energy of activation flow (ΔG^{*E}) versus mole fraction (X_2) of DEHPA.

In the studied binary mixtures, the values of excess Gibb's energy of activation flow (ΔG^{*E}) (Fig.6) are positive for two binary systems over the whole composition range. This function is a more adequate property for the study of molecular interactions and positive trends indicate closer packing of the molecules due to H -bonding of unlike molecules in the binary systems. Positive values ΔG^{*E} for both mixtures are considered due to chemical interactions, formation of hydrogen bonds and other complex forming interactions [23-25]. It depends on the structural characteristics of liquid components arising from the geometrical fitting of one component into the structure of another component. The molar volume of methanol, propionic acid and DEHPA are 0.4096 x10^{-6} m^3 mol^{-1}, 0.7545 x10^{-6} m^3 mol^{-1} and 3.3541x10^{-6} m^3 mol^{-1} respectively. DEHPA molecule is bigger in size as compared to methanol and propionic acid under study. Positive values of ΔG^{*E} agree with the analysis of interactions in cases of deviation in isentropic compressibility, intermolecular free length, acoustic impedance, free volume, viscosity and excess volume.

A plausible interpretation of various excess/deviation properties can be suggested from the above results for different components in both liquid mixtures containing DEHPA. Trends of deviation/excess properties indicate the presence of molecular interaction in the present binary mixtures under study. It may be qualitatively inferred that the interaction between unlike molecules is mainly due to hydrogen bonding through a highly polar lone pair oxygen atom of phosphoryl group of DEHPA and hydrogen atom of hydroxyl group of methanol and propionic acids. The effective dipole moment and dielectric constant [21-23] are useful parameters to investigate the impact of polarity on bulk properties of components of liquid mixtures. The present investigation deals with polar-polar binary mixtures whose component dipole moments and dielectric constants have the respective values as mentioned herewith:

(i) DEHPA (μ_D ~ 2.74D) > propionic acid (μ_P ~ 1.76D) > methanol (μ_M ~ 1.70 D)

(ii) methanol (ε_m ~ 33) > DEHPA (ε_D ~ 4.46) > propionic acid (ε_D ~ 3.4)

Comparison of various studied properties reveals that dipole-dipole type of molecular interaction between unlike molecules in DEHPA and methanol system predominates over propionic acid and degree of interaction is relatively stronger in methanol may be due to its smaller carbon chain length, lower dipole moment and higher dielectric constant.

4. CONCLUSIONS

This paper is a part of our continuing studies to select a suitable modifier among 1-alkanols and monocarboxylic acids with DEHPA for solvent extraction process. The macroscopic properties such as relaxation time, Rao's constant, Wada's constant, absorption coefficient, deviation in isentropic compressibility, intermolecular free length, acoustic impedance, free volume, viscosity, excess volume and excess Gibb's energy of activation flow for the binary mixtures of methanol / propionic acid and DEHPA have been determined from experimental values of density, viscosity and ultrasonic velocity. The result of the above said functions suggest the presence of stronger molecular interaction between unlike molecules in DEHPA and methanol system predominates over propionic acid system. The degree of interaction is relatively stronger in methanol may be due to its smaller carbon chain length, lower dipole moment and higher dielectric constant.

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