

Density, Refractive Index, and Electrical Conductivity of Ternary Mixtures Containing 1,3-Butanediol + Sulfolane (TMS) + Perchlorate Salts at Different Temperatures and Atmospheric Pressure

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The density, refractive index, and electrical conductivity of binary and ternary mixtures containing 1,3-butanediol (1) + sulfolane (2) + lithium perchlorate or sodium perchlorate or ammonium perchlorate (3) were measured at temperatures between 293.15-313.15 K. From the density data, the excess molar volume V_m^E , the partial molar volume $\bar{V}_{m,i}$, the excess partial molar volume $\bar{V}_{m,i}^E$ were obtained. The excess molar volumes were correlated using the Redlich-Kister equation to estimate the respected coefficients. Using the Reis equation, the excess refractive index n_D^E was obtained and fitted by the Redlich-Kister equation. Moreover, the values of the specific electrical conductivity have been fitted by polynomial equation to calculate the adjustable parameters constants. The sign and magnitude of excess quantities have been discussed in terms of the nature of molecular interactions between the components. The impacts of the electrolyte and solvation effects on the volumetric, optical properties and conductivity behavior were also addressed.

Keywords: 1,3-Butanediol, Sulfolane, Perchlorate salt, Refractive indices, Density

INTRODUCTION

Attention in the nature of electrolytic solutions has been of great interest historically because of its importance in the development of currently held concepts of the ionic compounds nature and in electrochemistry. Observation, understanding, and description of electrolytic conductivity were principally important for the early development of solution theory. Depending on the type of salt added, the thermodynamic and excess thermo physical properties of the solutions can be increased or decreased. Tetramethylsulfone (TMS) or sulfolane is an important industrial solvent with physicochemical advantages like low vapor pressure, aprotic nature, easy recycling, relatively low toxicity, good miscibility with most of the common protic solvents. Also, it has reliable chemical and thermal stability,

very low autoprotolysis constant, and the capability to be used in many extraction processes. Particularly it is useful in the petrochemical industry because of its ability to extract aromatic hydrocarbons from aliphatic ones and to absorb waste gases [1,2]. Sulfolane is a globular molecule in which the negative end of its large dipole moment is exposed and cannot play as proton acceptor/donor [3]. Numerous applications have also appeared in the patent literatures for sulfolane in photographic emulsions, fabrics manufacturing, and polymer plasticizing. On the other hand, alkanediols are the class of materials with the ability to form intra molecular hydrogen bonding within their molecules and also the tendency to form hydrogen bonding with other molecules of component. Besides alkanediols play important roles in industry such as antifreezes, coolants, plasticizers, solvents and extractors in automotive, and also in cosmetic, pharmaceutical, food, and petroleum industrie [4]. The electrical conductivity investigation represents a

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simple, accurate and reliable method to study the solvents effects, modification in the structure of a solvent due to the presence of an ion, and ionic movement in a solution. Also, data from conductivity measurement can provide detailed information on ion-ion and ion-solvent interactions for different electrolyte systems [5-7]. The refractive index property can be measured for a small amount of sample at wide ranges of temperatures and pressures with high precisions and covers information about the intermolecular interactions [8-11]. Interactions between sulfolane and alcohols may be complex. Alcohols are self-associated liquids through H-bonding and possesses hydrophilic character. Sulfolane is a globular molecule in which the negative end of its large dipole moment is exposed and cannot act as proton acceptor/donor. Therefore, the mode of interactions of alcohols and sulfolane is of great importance in the field of solution chemistry as it can provide vital information regarding hydrophilic and hydrophobic interactions [12]. The experimental data were modeled with suitable equations for each property. Mixtures of sulfolane with other solvents are also of particular interest. In general, experimental data of densities and excess properties are used as tools to understand interactions that usually occur among molecules with diversified structural moieties and/or functional groups. While numerous articles have already been published, focusing on densities and excess properties of sulfolane mixtures [13-29]. There is still a lack of complete thermodynamic characterization data for sulfolane solutions having glycols. This paper reports the experimental values of density ρ , refractive indices n_D , and specific electrical conductivity k_s , of sulfolane + 1,3-butanediol + perchlorates salts (lithium perchlorate, sodium

perchlorate and ammonium perchlorate) at 0.1 mol per lit of (sulfolane + 1,3-butanediol) as a function of 1,3-butanediol mole fraction binary and ternary mixtures at different temperatures with 5 K interval and atmospheric pressure. The excess molar volume V_m^E partial molar volume $\bar{V}_{m,i}^E$, and excess partial molar volume $\bar{V}_{m,i}^E$, were calculated from the obtained experimental data. Moreover, the excess refractive index, n_D^E , of a given liquid mixture was calculated using the Reis et al equation. The values of V_m^E and the excess refractive index n_D^E have been fitted to the Redlich-Kister polynomial equation to drive binary coefficients and estimate their root mean square deviation. The experimental values of specific electrical conductivity have fitted by a polynomial model to obtain the related constants. Moreover, molar conductivities Λ , were calculated from specific conductivity k_s data.

EXPERIMENTAL

Materials

Sulfolane and perchlorate salts were purchased from Fluka while 1,3-butanediol was supplied by Merck. Purity of the used chemicals was higher than 98% and their water content was around 0.2%. The specifications of these chemicals were listed in Table 1 in details.

Sample Preparation

The binary and ternary mixtures were prepared by mass and stirred for 2-3 h to dissolve completely and produce a homogeneous mixture. They are stored in special air-tight bottles and kept safely before the experiments. The weighing was performed using a Precisa 240A balance

Table 1. Identification of Chemicals Used

Chemical name	Source	Formula	Mole fraction purity	CAS No.
Sulfolane	Fluka	C ₄ H ₈ O ₂ S	> 0.98	126330
Lithium perchlorate	Fluka	LiClO ₄	≥ 0.99	57556
Sodium perchlorate	Fluka	NaClO ₄	> 0.98	504632
Ammonium perchlorate	Fluka	NH ₄ ClO ₄	≥ 0.99	107880
1,3-Butanediol	Merck	C ₄ H ₁₀ O ₂	≥ 0.99	110634

Standard uncertainties are $u(T) = 0.01$ K, $u(x_i) = 0.0005$, $u(p) = 1$ kPa, relative standard uncertainties are $u_r(\rho) = 0.002$; and $u_r(n_D) = 0.01$.

(accuracy ± 0.1 mg). The uncertainty of the mole fraction was in the order of ± 0.0001 .

Density Measurements

An Anton Paar DMA 500 digital vibrating tube densitometer was used to measure density of the neat compounds and their binary mixtures. The instrument was calibrated with double-distilled water and air. Each value is an average of at least two measurements. The claimed uncertainty of density was $\pm 10^{-4}$ g cm⁻³. The temperature was recorded by the help of a thermocouple with uncertainty estimated to be within ± 0.01 K.

Refractive Index Measurements

Refractive indices for the sodium D line were obtained using a thermostatically controlled and digital Abbe-type refractometer AR2008 (Krüss-Germany). The temperature

was controlled using a constant temperature bath (JULABO model F 34, Germany). The refractometer calibration was carried out using twice distilled and deionized water. A minimum of three independent readings were recorded for each measurement. The accuracy of refractive index measurements was estimated to be less than 0.0002 units.

Electrical Conductivity Measurements

Electrical conductivity was measured with a Jenway conductometer model 4020, UK. The accuracy of electrical conductivity measurements is $\pm 10^{-2}$ μ S cm⁻¹. The cell constant is 0.95 cm⁻¹, which was calculated by repeated measurements of KCl solutions.

RESULTS AND DISCUSSION

Table 2 compares the measured density and refractive

Table 2. Comparison of the Experimental Values of Density (ρ) and Refractive Index (n_D) of Pure Liquids with the Available Corresponding Literature Values at Different Temperatures and Pressure of 101.325 kPa

T (K)	ρ (g cm ⁻³)		n_D	
	Experimental	Literature	Experimental	Literature
Sulfolane				
293.15	1.2696	1.2708 ^[29]		1.48541 ^[29]
298.15	1.2651	1.26654 ^[30] 1.2640 ^[31] 1.26705 ^[20] 1.2665 ^[29]	1.4841	1.4836 ^[29]
303.15		1.2623 ^[28]	1.4817	1.4819 ^[32] 1.4818 ^[29]
	1.2621			
308.15	1.2576	1.2570 ^[31]	1.4792	1.47965 ^[29]
313.15	1.2545	1.2540 ^[28]		1.47800 ^[29]
1,3-Butanediol				
298.15	1.0003	1.00008 ^[33]	1.4387	1.439 ^[29] 1.4382 ^[34]
303.15	0.9973	0.99716 ^[33]	1.4378	1.4376 ^[34]
308.15	0.9944	0.99423 ^[33]	1.4367	1.4363 ^[34]

Standard uncertainties are $u(T) = 0.01$ K, $u(x_1) = 0.0005$, $u(p) = 1$ kPa, relative standard uncertainties are $ur(\rho) = 0.002$; and $ur(n_D) = 0.01$.

index data of the pure liquids with those reported in the literatures at different temperatures [30-33]. From Table 2, the quality of the experimental data could be confirmed as well.

Volumetric Properties

The obtained experimental values of densities and calculated volumetric properties comprising excess molar volume V_m^E , partial molar volume $\bar{V}_{m,i}$, excess partial molar volume $\bar{V}_{m,i}^E$, for binary (sulfolane + 1,3-butanediol) and ternary { sulfolane + 1,3-butanediol + (lithium perchlorate, sodium perchlorate and ammonium perchlorate)} mixtures at 0.1 mol per lit of (sulfolane + 1,3-butanediol) as a function of 1,3-butanediol mole fraction at different temperatures are reported in Table 3. As the extension of strength and type of bonding between the components is extremely important, the experimental V_m^E values were obtained based on the following equation:

$$V_m^E = V_m - V_m^{id} \quad (1)$$

Where, the molar volume (V_m) was calculated as:

$$V_m = \frac{x_1 M_1 + x_2 M_2 + x_3 M_3}{\rho} \quad (2)$$

Typically, the molar volume of an ideal solution (V_m^{id}) could be obtained as:

$$V_m^{id} = \frac{x_1 M_1}{\rho_1} + \frac{x_2 M_2}{\rho_2} + x_3 \bar{V}_{m,3}^\infty \quad (3)$$

Where, x_1 , x_2 , and x_3 are the mole fraction of 1,3-butanediol (1), sulfolane (2), and the electrolyte (3), respectively; M_1 , ρ_1 , M_2 , ρ_2 are molar mass and densities of 1,3-butanediol and sulfolane, respectively, ρ is the density of ternary mixtures and $\bar{V}_{m,3}^\infty$ is partial molar volume values of the electrolytes at infinite dilution can be expressed by Eq. (6). The salt concentration (0.1 M) was constant in the all experiments. For determination of the experimental V_m^E data for binary mixtures containing (sulfolane + alkanediols), Eqs. (2) and (3) are used, considering null the electrolyte contribution. Noticeably, perchlorate salts can dissolve in

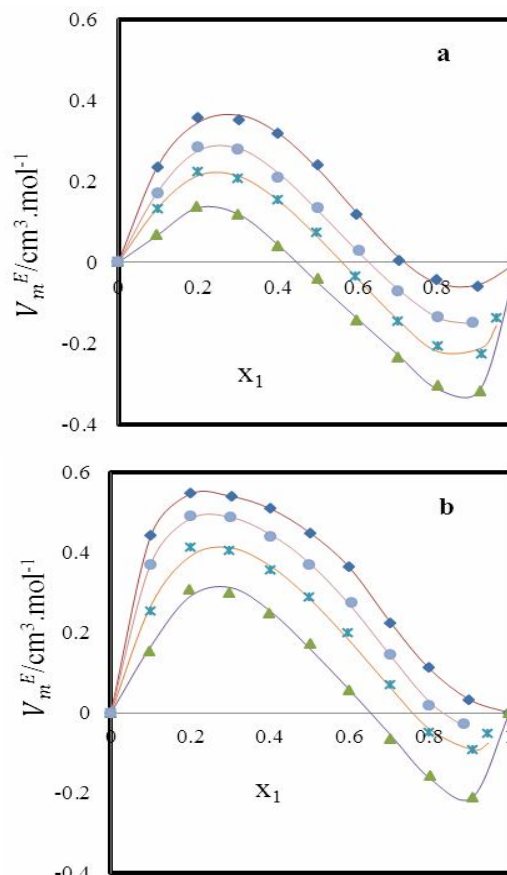


Fig. 1. Dependence of the excess molar volume for the binary and ternary mixtures of sulfolane + 1,3-butanediol (■) + perchlorates salts (LiClO₄ (▲) or NaClO₄ (*) or NH₄ClO₄ (●)) as a function 1,3-butanediol mole fraction at temperatures of 293.15 K (a) and 313.15 K (b). The lines represent V_m^E calculated from Redlich Kister equation.

sulfolane and alcohols as well. Of the three salts, lithium perchlorate dissolves completely in sulfolane and 1, 3-butanediol. To determine the experimental V_m^E data for the binary mixture (sulfolane + 1,3-butanediol), Eqs. (2) and (3) were also used. Figure 1 and Table 3 illustrate the excess molar volume behavior obtained by the Redlich-Kister equation as well as the experimental V_m^E data, as a function of 1,3-butanediol concentration, for ternary mixtures containing sulfolane + 1,3-butanediol + (LiClO₄, or NaClO₄, or NH₄ClO₄) systems (0.1 M) and for salt-free

Table 3. Experimental Density (ρ), Excess Molar Volume V_m^E , Partial Molar Volume \bar{V}_m and Excess Partial Molar Volume $\bar{V}_{m,i}^E$ for the Binary and Ternary Mixtures of Sulfolane + 1,3-Butanediol + Perchlorates Salts (LiClO_4 or NaClO_4 or NH_4ClO_4) as a Function 1,3-Butanediol Mole Fraction at 298.15 K

x_1	ρ (g cm^{-3})	V_m^E ($\text{cm}^3 \text{mol}^{-1}$)	$\bar{V}_{m,1}$ ($\text{cm}^3 \text{mol}^{-1}$)	$\bar{V}_{m,2}$ ($\text{cm}^3 \text{mol}^{-1}$)	$\bar{V}_{m,1}^E$ ($\text{cm}^3 \text{mol}^{-1}$)	$\bar{V}_{m,2}^E$ ($\text{cm}^3 \text{mol}^{-1}$)
Sulfolane + 1,3-butanediol 298.15 K						
0.0000	1.2651	0.0000	94.9784	94.9885	4.8674	0.0000
0.1003	1.2359	0.2946	91.8919	95.1175	1.7809	0.1289
0.1995	1.2092	0.4073	90.8619	95.3102	0.7509	0.3217
0.3028	1.1827	0.4017	90.4218	95.4297	0.3108	0.4411
0.4003	1.1578	0.3654	90.1422	95.5771	0.0312	0.5886
0.5004	1.1322	0.3085	89.9260	95.7915	-0.1850	0.8029
0.5979	1.1078	0.1855	89.7992	95.9136	-0.3118	0.9251
0.7031	1.0811	0.0529	89.8431	95.8013	-0.2679	0.8127
0.7976	1.0562	-0.0141	89.9928	95.3842	-0.1182	0.3957
0.9001	1.0282	-0.0309	90.1023	94.7575	-0.0087	-0.2310
1.0000	1.0001	0.0000	90.1110	94.8651	0.0000	-0.1234
Sulfolane + 1,3-butanediol + LiClO_4 298.15 K						
0.0000	1.2651	0.0000	90.5679	94.9885	0.4569	0.0000
0.0984	1.2391	0.0867	91.1297	94.9735	1.0187	-0.0150
0.1977	1.2127	0.1707	90.5275	95.0987	0.4165	0.1102
0.2992	1.1869	0.1417	89.9080	95.2774	-0.2029	0.2889
0.3992	1.1617	0.0763	89.6638	95.4126	-0.4472	0.4241
0.5006	1.1360	-0.0065	89.6495	95.4382	-0.4615	0.4497
0.5989	1.1110	-0.1033	89.6567	95.4094	-0.4543	0.4209
0.7019	1.0844	-0.1981	89.6433	95.4252	-0.4677	0.4367
0.8007	1.0583	-0.2695	89.7030	95.2755	-0.4080	0.2870
0.9076	1.0290	-0.2820	89.9225	93.7877	-0.1884	-1.2008
1.0000	1.0001	0.0000	90.1110	89.2782	0.0000	-5.7103
Sulfolane + 1,3-butanediol + NaClO_4 298.15 K						
0.0000	1.2651	0.0000	91.4537	94.9885	1.3427	0.0000
0.1008	1.2376	0.1539	91.5750	95.0277	1.4640	0.0392
0.1995	1.2110	0.2663	90.7967	95.2002	0.6857	0.2117
0.2992	1.1855	0.2539	90.1187	95.3993	0.0077	0.4108
0.4013	1.1596	0.1992	89.8265	95.5656	-0.2845	0.5771
0.4972	1.1353	0.1243	89.7698	95.6287	-0.3412	0.6402
0.5944	1.1107	0.0213	89.7366	95.6473	-0.3744	0.6588
0.7012	1.0834	-0.0986	89.6954	95.6940	-0.4156	0.7055
0.8002	1.0573	-0.1713	89.7803	95.5012	-0.3307	0.5127
0.9096	1.0275	-0.1990	90.0268	93.6754	-0.0842	-1.3131
0.9476	1.0159	-0.1060	90.1575	92.1525	0.0465	-2.8360
Sulfolane + 1,3-butanediol + NH_4ClO_4 298.15 K						
0.0000	1.2651	0.0000	92.1788	94.9885	2.0678	0.0000
0.1009	1.2370	0.1985	91.8781	95.0512	1.7671	0.0627
0.1995	1.2102	0.3285	90.9534	95.2550	0.8424	0.2664
0.3006	1.1842	0.3267	90.2153	95.4710	0.1043	0.4824
0.4013	1.1588	0.2652	89.8950	95.6383	-0.2160	0.6498
0.4992	1.1340	0.1875	89.8241	95.7132	-0.2869	0.7247
0.6042	1.1073	0.0860	89.8077	95.7271	-0.3033	0.7385
0.7010	1.0826	-0.0248	89.7860	95.7194	-0.3250	0.7308
0.8000	1.0565	-0.0985	89.8788	95.4710	-0.2322	0.4825
0.8880	1.0325	-0.1146	90.1207	93.9288	0.0097	-1.0597

Standard uncertain ties $u(T) = 0.01$ K, $u(x_1) = 0.0005$, $u(p) = 1$ kPa, relative standard uncertainties $ur(\rho) = 0.002$; $ur(\bar{V}_m^E) = 0.006$ and $ur(n_D) = 0.01$.

binary mixtures, both at two temperatures of 293.15 K and 313.15 K. Results showed a positive and negative trend for excess molar volume in all mixtures. The curves are skewed towards the side of 1,3-butanediol rich region. From Fig. 1, it can be observed that the maximum and minimum values of excess volume in terms of mole fraction of the 1,3-butanediol (x_1) are close to $x_1 \approx 0.2$ and $x_1 \approx 0.9$ respectively. However, the maximum amount of V_m^E for the ternary mixtures of {sulfolane + 1,3-butanediol + (LiClO₄, or NaClO₄, or NH₄ClO₄)} are 0.1369, 0.2239, and 0.2856 cm³ mol⁻¹, respectively. Correspondingly, the minimum value of V_m^E for {sulfolane + 1,3-butanediol + (LiClO₄, or NaClO₄, or NH₄ClO₄)} ternary systems are found to be -0.3170, -0.2261, and -0.1495 cm³ mol⁻¹ at 293.15 K. It is worthy to note that the values of V_m^E increases with the increase in temperature in all mixtures. The magnitude of V_m^E of the mixtures is due to the contributions from several opposing effects such as extends of specific interaction, structural contributions, complex formation, formation or breaking of hydrogen bond, London forces, and *etc.* The obtained negative excess molar volumes may be attributed to the specific interactions between the -OH group of 1,3-butanediol and S=O bond of sulfolane molecules and structural contributions arising from the geometric coupling of 1,3-butanediol molecules into sulfolane. The obtained positive excess molar volume reveals the weak interaction among the mixture components or due to the breaking of bonds formed in the solution those results in an expansion of the mixture volume. The main reason that cause expansion of volume on mixing of the components might be described according to the dissociation of one or two component/s, steric hindrance owing to chain branching, geometrical mismatch of molecules, and formation of weaker solute-solvent bond compared to other interactions like solute-solute or solvent-solvent bonds [34]. It seems that the main contribution to the changes in the excess molar volume comes from the self-hydrogen bonding of 1,3-butandiol. For the sulfolane rich zones (typically $x_1 < 0.5$) it acts as structural breaker and accordingly the V_m^E values are more positive. Obviously, as the concentration of the used diol increased the interactions between the like molecules, *i.e.* 1,3-butandiol can be enhanced and therefore the V_m^E values

become negative at $x_1 > 0.7$. Compared to the binary solution, the presences of perchlorates salts in the ternary mixtures improve the interactions between the diols molecules as the maximum values of the V_m^E decreased. In other words, the perchlorate salts play as structural maker by improving the diol-diol interaction through hydrogen bonding. Among the used salts, this is more significant for the lithium perchlorate at all temperatures. Anyways, the magnitude of the positive deviations in V_m^E can be ordered as: sulfolane + 1,3-butanediol > sulfolane + 1,3-butanediol + ammonium perchlorate > sulfolane + 1,3-butanediol + sodium perchlorate > sulfolane + 1,3-butanediol + lithium perchlorate. It seems that the solvation is more remarkable in the mixtures containing LiClO₄ than those solutions having NH₄ClO₄ and NaClO₄ salts. This fact could be also associated to the effect of electrolyte cation size since their size reduces as: $r_{Li^+} < r_{Na^+} < r_{NH_4^+}$. Accordingly, the molecules and ions arrangement are favored toward an increase in the volumetric contraction. On the other hand, the experimental V_m^E data sound that the electron density on oxygen atoms of the hydroxyl groups of 1,3-butanediol is comparatively greater in the case of the lithium salt leading to stronger bonding between sulfolane and 1,3-butanediol molecules. Observation of data indicates that the addition of an electrolyte in the binary mixture of sulfolane and 1,3-butanediol increases the contraction effect, which represent a greater decrease in the excess molar volume values than in that of the salt free mixtures. The expansion of molecular agitation in solution at higher temperatures leads to weakening the strength of intermolecular bonding and attraction between like and unlike molecules. Therefore, the total volume of mixture is expanded and the density of solution is reduced. The order of strength of interaction follows the order: T = 293 > 298 > 303 > 308 > 313 K. Anyways, the presence of the electrolytes improves the solvation effects in the mixtures of sulfolane + 1,3-butanediol as evidenced in Table 3.

The composition dependence of V_m^E was correlated by the conventional curve-fitting approach using the Redlich-Kister polynomial equation [35]:

$$V_m^E = x_1 x_2 \sum_{i=0}^n A_i (x_1 - x_2)^i \quad (4)$$

Where, Y is the excess quantity (V_m^E), x_1 , x_2 are the mole

Table 4. Coefficients of the Redlich-Kister Equation along with their Root Mean Square Deviation (*RMSD*). for the Binary and Ternary Mixtures of Sulfolane + 1,3-Butanediol + Perchlorates salts (LiClO_4 or NaClO_4 or NH_4ClO_4) as a Function 1,3-Butanediol Mole Fraction at 298.15 K

Property	A_4	A_3	A_2	A_1	A_0	<i>RMSD</i>
Sulfolane + 1,3-butanediol						
V_m^E						
298.15	1.8904	-0.5247	-0.7124	-1.9707	1.1954	0.0109
n_D^E						
298.15	-0.0034	0.0012	0.0070	-0.0006	-0.0109	0.00009
Sulfolane + 1,3-butanediol + LiClO_4						
n_D^E						
298.15	-2.7540	-1.2740	0.1710	-1.8140	-0.0437	0.0173
n_D^E						
298.15	-0.0021	0.0012	0.0052	-0.0035	-0.0166	0.00013
Sulfolane + 1,3-butanediol + NaClO_4						
V_m^E						
298.15	-1.0797	-0.6656	-0.2645	-2.0179	0.1967	0.0124
n_D^E						
298.15	-0.0163	-0.0087	0.0091	0.0001	-0.0213	0.00005
Sulfolane + 1,3-butanediol + NH_4ClO_4						
V_m^E						
298.15	-0.9274	-0.3571	0.2187	-2.0189	0.7353	0.0137
n_D^E						
298.15	-0.0156	-0.0111	0.0075	-0.0003	-0.0255	0.0014

fraction of 1,3-butanediol (1) and sulfolane (2) respectively, and the A_i s are adjustable binary coefficients which are estimated *via* multi-parametric regression analysis based on the least-squares method. In each case, the optimum number of A_i coefficients were obtained from an examination of root mean square deviation (*RMSD*, as given by [36]:

$$RMSD = \left[\sum_{i=1}^n (x_{c,i} - x_{e,i})^2 / n \right]^{1/2} \quad (5)$$

Where, x_c and x_e are the calculated and experimental

properties, n is the number of data points. The calculated values of A_i and root mean square deviation of V_m^E in 298.15 K are listed in Table 4. The other calculated values of A_i in different temperatures are presented in supplementary data: Table S2.

When two miscible liquids are mixed together, the volume of mixtures may not equal that of the sum of the volumes of the individual components. This difference in the total volume is called partial molar volume. The partial molar volumes are not constant and vary with the

composition of the mixture. because the environment of themolecules in the mixture changes with the composition. It is the changing molecular environment, and the consequent alteration of the interactions between molecules that results in change in the volume occupied by a given number of molecules. This change depends upon the identity of the surrounding molecule such as Li^+ , rNa^+ , or $< \text{rNH}_4^+$. In addition, partial molar quantities are important in the study of the dependence of an extensive property in the phase composition at constant pressure and temperature. The partial molar volume of component i , in the binary mixtures \bar{V}_i , is defined as:

$$\bar{V}_i = \left(\frac{\partial V}{\partial n_i} \right)_{T,P,n_j} \quad (6)$$

Where, n_i is the number of moles of the i th component added to the mixture and n_j is the number of moles of the other components. Here, with the use of excess molar volume and molar volume of the neat components, the partial molar volume of component 1 and 2, $\bar{V}_{m,1}$ and $\bar{V}_{m,2}$, were calculated using the following equations [37]:

$$\bar{V}_{m,1} = V_m^E + V_1^* + (1 - x_1) \left(\frac{\partial V_m^E}{\partial x_1} \right)_{T,P} \quad (7)$$

$$\bar{V}_{m,2} = V_m^E + V_2^* - x_1 \left(\frac{\partial V_m^E}{\partial x_1} \right)_{T,P} \quad (8)$$

Where, V_1^* and V_2^* devotes to the molar volume of neat components 1,3-butanediol (1), sulfolane (2) respectively.

The derivative of $\left(\frac{\partial V_m^E}{\partial x_1} \right)_{T,P}$ was estimated by differentiation of the polynomial fitting of V_m^E equation (Eq. (4)) with respect to x_1 .

$$\bar{V}_{m,1} = V_1^* + (1 - x_1)^2 \sum_{i=0}^m A_i (1 - 2x_1)^i - 2x_1 (1 - x_1)^2 \sum_{i=0}^m A_i i (1 - 2x_1)^{i-1} \quad (9)$$

$$\bar{V}_{m,2} = V_2^* + x_1^2 \sum_{i=0}^m A_i (1 - 2x_1)^i - 2x_1 (1 - x_1) \sum_{i=0}^m A_i i (1 - 2x_1)^{i-1} \quad (10)$$

The obtained values of partial molar volume for all mixtures just at 298.15 K are listed in columns 4 and 5 of Table 3.

The other obtained values of partial molar volume in different temperatures between 293.15-313.15 K are presented in supplementary data: Table S1. The data indicates that $\bar{V}_{m,1}$ and $\bar{V}_{m,2}$ are positive for all of the experimental temperatures. From Table 3, the partial molar volumes of 1,3-butanediol decrease by increasing the partial molar volume of sulfolane. The Gibbs-Duhem equation is defined as follows:

$$\sum_i x_i d\bar{m}_i = 0 \quad (11)$$

Where, \bar{m}_i can be any partial molar property. According to this equation, the partial molar quantities are not independent from other components of the mixture. By adding salt the values of partial molar volumes $\bar{V}_{m,1}$ and $\bar{V}_{m,2}$ decreases and for solution contain lithium perchlorate is the smallest amount.

Using partial molar volumes, the corresponding excess values ($\bar{V}_{m,1}^E$ and $\bar{V}_{m,2}^E$) have been calculated according to the following equation:

$$\bar{V}_{m,1}^E = \bar{V}_{m,1} - V_1^* \quad (12)$$

$$\bar{V}_{m,2}^E = \bar{V}_{m,2} - V_2^* \quad (13)$$

The numerical values of $\bar{V}_{m,1}^E$ and $\bar{V}_{m,2}^E$ are reported in Table 3. Positive and negative values are observed for the amounts of $\bar{V}_{m,1}^E$ data of mixtures over the whole composition range.

Upon adding the perchlorate salts, the values of $\bar{V}_{m,1}^E$ became more negative and $\bar{V}_{m,2}^E$ become more positive than the neat system, *i.e.* sulfolane + 1,3-butanediol. Again, as the size of cation in the used salts increased, the contraction in volume decreased as well. The ammonium salt exhibits the smallest changes in $\bar{V}_{m,1}^E$ values. The $\bar{V}_{m,2}^E$ value for the lithium salt becomes completely positive. These observations indicate that the molar volumes of 1,3-butanediol + sulfolane in the ternary mixtures are less than their respective values in the pure state demonstrating that there is a contraction in volume with the presence of the electrolytes.

Optical Properties

Attempts have been paid in this section to measure the

refractive indices at different temperatures owing to continuous interest in optical properties of the electrolyte solutions. Normally, refractive index could be defined as the ratio of the light speed in a vacuum to the speed of light through the medium of interest. Variation of the refractive index in the mixture provides valuable information about the structural accommodation and accessible the free volume. By increasing free volume of the mixture, light transmission occurs with higher speed and the refractive index decreases. The refractive indices of 1,3-butanediol + sulfolane + perchlorate salt ternary mixtures were obtained over the temperature range of 298.15 to 308.15 K and the data is represented in Table 5. Decreased values in the refractive indices were observed by increasing the mole fraction of 1,3-butanediol. The same trend was seen with the rise in temperature as evidenced clearly in Table 5. Obviously, presence of the perchlorate salts results in reduction of the refractive index.

From the obtained data of refractive index, the deviations in refractive index, Δn_D were calculated. Reis *et al.* [38] have proposed an expression to assess the refractive index of thermodynamically ideal liquid mixtures. According to this formulation, it is possible to estimate the Δn_D of a given liquid mixture by using the following equation:

$$\Delta n_D = n_D - \left[\varphi_1 (n_{D_1})^2 + \varphi_2 (n_{D_2})^2 \right]^{\frac{1}{2}} \quad (14)$$

Where, n_D is the refractive index of the mixture, and $n_{D,i}$ the refractive index of component i . Moreover φ is the volume fraction before mixing and calculated with the following equations:

$$\varphi_i = \frac{x_i V_i}{(x_1 V_1 + x_2 V_2)} \quad (15)$$

where x_i and V_i are the mole fraction and the molar volume of pure components. Figure 2 and Table 5 show the graphical and numerical changes in excess refractive indices along with the calculated values by the Redlich-Kister equation for the binary and ternary mixtures. The A_i parameters obtained by using the Redlich-Kister equation and the respected values of root mean square are given in Table 4. The excess refractive indices for all the systems are negative across the whole range of compositions and increased by temperature increment which is the result of an

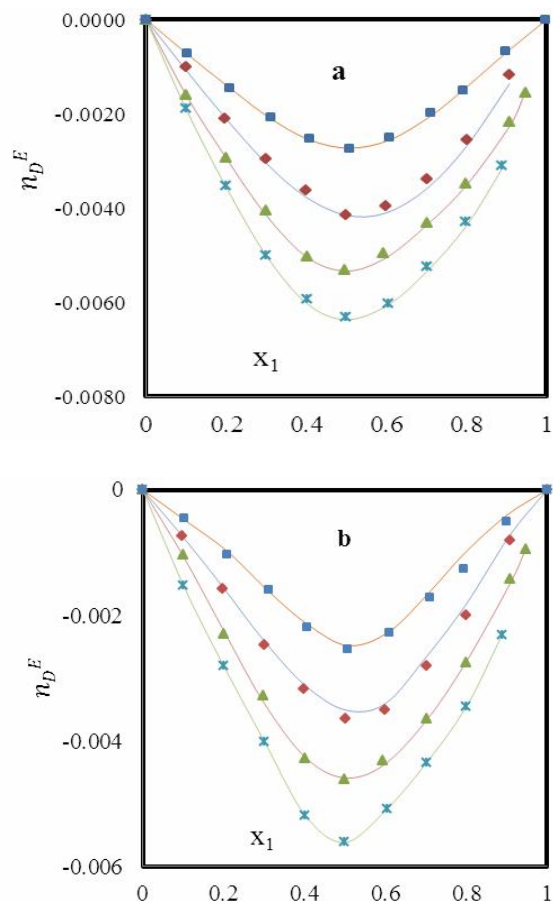


Fig. 2. Dependence of the excess refractive index for the binary and ternary mixtures of sulfolane + 1,3-butanediol (■) + perchlorates salts (LiClO_4 (◆) or NaClO_4 (▲) or NH_4ClO_4 (*)) as a function 1,3-butanediol mole fraction at temperature of 298.15 K (a) and 308.15 K (b). The lines represent n_D^E calculated from the Redlich Kister equation.

isobaric contraction process. As indicated in Fig. 2, the mixtures containing the ammonium salt exhibit the lowest n_D^E amounts at different temperatures. As the temperature increased the values of the excess refractive index became more positive for all mixtures. The minima of Fig. 2 are located in the composition $x_1 \approx 0.5$ in terms of 1,3-butanediol mole fraction. No considerable changes in the place of minima were observed by changing the perchlorate salt. Additionally, the minimum values of the excess refractive index in ternary mixtures are the following:

Table 5. Refractive Indices and the Respective Excess Values for the Binary and Ternary Mixtures of Sulfolane + 1,3-Butanediol + Perchlorates Salts (LiClO₄ or NaClO₄ or NH₄ClO₄) as a Function 1,3-Butanediol Mole Fraction at Temperatures of 298.15-308.15 K

x_j	n_D T = 298.15 K	n_D T = 303.15 K	n_D T = 308.15 K	n_D^E T = 298.15 K	n_D^E T = 303.15 K	n_D^E T = 308.15 K
Sulfolane + 1,3-butanediol						
0.0000	1.4841	1.4817	1.4792	0.0000	0.0000	0.0000
0.1027	1.4790	1.4770	1.4748	-0.0007	-0.0005	-0.0003
0.2080	1.4737	1.4720	1.4701	-0.0015	-0.0010	-0.0007
0.3126	1.4685	1.4670	1.4653	-0.0021	-0.0016	-0.0012
0.4078	1.4638	1.4623	1.4607	-0.0025	-0.0022	-0.0018
0.5076	1.4591	1.4576	1.4561	-0.0027	-0.0025	-0.0022
0.6086	1.4547	1.4534	1.4521	-0.0025	-0.0023	-0.0019
0.7106	1.4505	1.4494	1.4482	-0.0020	-0.0017	-0.0014
0.7935	1.4471	1.4461	1.4450	-0.0015	-0.0013	-0.0010
0.8991	1.4429	1.4420	1.4409	-0.0007	-0.0005	-0.0004
1.0000	1.4387	1.4378	1.4367	0.0000	0.0000	0.0000
Sulfolane + 1,3-butanediol + LiClO ₄						
0.0000	1.4841	1.4817	1.4792	0.0000	0.0000	0.0000
0.0984	1.4789	1.4769	1.4747	-0.0010	-0.0007	-0.0006
0.1977	1.4735	1.4719	1.4701	-0.0021	-0.0016	-0.0011
0.2992	1.4682	1.4667	1.4652	-0.0030	-0.0025	-0.0019
0.3992	1.4631	1.4617	1.4603	-0.0036	-0.0032	-0.0026
0.5006	1.4580	1.4568	1.4554	-0.0041	-0.0037	-0.0032
0.5989	1.4537	1.4526	1.4515	-0.0040	-0.0035	-0.0029
0.7019	1.4495	1.4486	1.4477	-0.0034	-0.0029	-0.0023
0.8007	1.4457	1.4450	1.4442	-0.0026	-0.0020	-0.0014
0.9076	1.4420	1.4413	1.4403	-0.0012	-0.0008	-0.0006
1.0000	1.4389	1.4378	1.4367	0.0002	0.0000	0.0000
Sulfolane + 1,3-butanediol + NaClO ₄						
0.0000	1.4841	1.4817	1.4792	0.0000	0.0000	0.0000
0.1008	1.4782	1.4765	1.4744	-0.0016	-0.0010	-0.0008
0.1995	1.4726	1.4711	1.4696	-0.0029	-0.0023	-0.0016
0.2992	1.4671	1.4659	1.4646	-0.0041	-0.0033	-0.0025
0.4013	1.4616	1.4605	1.4594	-0.0050	-0.0043	-0.0034
0.4972	1.4570	1.4560	1.4548	-0.0053	-0.0046	-0.0040
0.5944	1.4529	1.4520	1.4509	-0.0050	-0.0043	-0.0037
0.7012	1.4486	1.4479	1.4470	-0.0043	-0.0036	-0.0030
0.8002	1.4448	1.4443	1.4435	-0.0035	-0.0027	-0.0022
0.9096	1.4409	1.4406	1.4400	-0.0022	-0.0014	-0.0008
0.9476	1.4397	1.4393	1.4387	-0.0015	-0.0010	-0.0004
Sulfolane + 1,3-butanediol + NH ₄ ClO ₄						
0.0000	1.4841	1.4817	1.4792	0.0000	0.0000	0.0000
0.1009	1.4779	1.4760	1.4741	-0.0019	-0.0015	-0.0011
0.1995	1.4720	1.4706	1.4690	-0.0035	-0.0028	-0.0022
0.3006	1.4661	1.4651	1.4639	-0.0050	-0.0040	-0.0031
0.4013	1.4607	1.4596	1.4587	-0.0059	-0.0052	-0.0041
0.4992	1.4559	1.4549	1.4539	-0.0063	-0.0056	-0.0048
0.6042	1.4514	1.4508	1.4498	-0.0060	-0.0051	-0.0044
0.7010	1.4477	1.4472	1.4464	-0.0052	-0.0043	-0.0036
0.8000	1.4440	1.4436	1.4430	-0.0043	-0.0035	-0.0027
0.8880	1.4410	1.4407	1.4402	-0.0031	-0.0023	-0.0015

{sulfolane + 1,3-butanediol + (LiClO₄, or NaClO₄, or NH₄ClO₄)} = -0.0041, -0.0053, - 0.0063. However, the positive excess molar volume establishes the poor interaction between the mixture components leading to an increase in the mixture volume known as packing effect. Accordingly, in the real mixtures, the free volume is more available than in the ideal state and the light passes at a higher speed, so the excess refractive indices of the mixture become negative.

Electrical Conductivities

Electrical conductivity is one of the most important transport properties frequently demanded by chemists and engineers dealing with electrolyte solutions. The conductivity of solution is determined by the concentration, charge, and mobility of the dissolved ions. Conductometry provides us most precise valuable information concerning ion association and ion solvation interactions in solution. Widespread ranges of interacting forces like solvent-solvent, ion-solvent and ion-ion interactions prevail in the electrolyte solutions in different concentration regions and leave their mark on the transport properties. The measured specific electrical conductivities values for sulfolane (2) + 1, 3-butanediol (1) + lithium perchlorate or sodium perchlorate or ammonium perchlorate at temperatures 298.15-313.15 K are listed in Table 6. The specific electrical conductivities values for the mixtures were determined from:

$$k_s = kL/A \quad (16)$$

Where, k_s is the specific electrical conductivity, k is the experimental electrical conductivity, L is the distance between the electrodes, and A is the effective area of the electrode. The ratio of (L/A) is called the cell constant. For all the systems the specific conductivities have an uncertainty of ± 0.05 . The maximum specific electrical conductivity is in $x_1 \approx 0.1$ at temperature 298.15 K. According to the solvation and change in specific electrical conductivity data in ternary mixtures are following: {sulfolane + 1,3-butanediol + (LiClO₄, or NaClO₄, or NH₄ClO₄)} = 0.70, 0.95, and 28.69 ms cm⁻¹ at 298.15 K. As expected, and shown in Fig. 3, the conductivity of each composition increases with the temperature. The specific electrical conductivity values decrease with an increase in

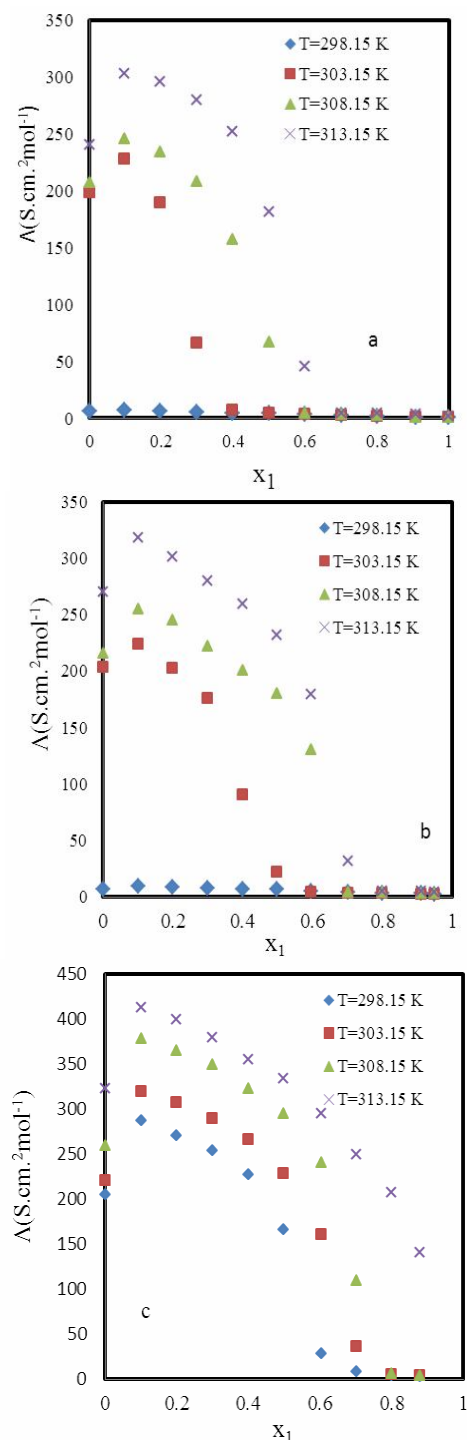


Fig. 3. Molar electrical conductivity (Λ) values for ternary mixtures of sulfolane + 1,3-butanediol + LiClO₄ (a), NaClO₄ (b), NH₄ClO₄ (c) as a function 1,3-butanediol mole fraction at different temperatures.

Table 6. Specific Electrical Conductivity (k_s) Values for the Binary and Ternary Mixtures of Sulfolane + 1,3-Butanediol + Perchlorates Salts (LiClO_4 or NaClO_4 or NH_4ClO_4) as a Function 1,3-Butanediol Mole Fraction at Temperatures of 298.15-313.15 K

x_1	k_s (ms cm^{-1}) \pm 0.05	k_s (ms cm^{-1}) \pm 0.05	k_s (ms cm^{-1}) \pm 0.05	k_s (ms cm^{-1}) \pm 0.05
	T = 298.15 K	T = 303.15 K	T = 308.15 K	T = 313.15 K
Sulfolane + 1,3-butanediol				
0.0000	0.00204	0.00216	0.00220	0.00231
0.1003	0.00258	0.00271	0.00304	0.00331
0.1995	0.00254	0.00261	0.00278	0.00314
0.3028	0.00209	0.00216	0.00245	0.00277
0.4003	0.00162	0.00165	0.00200	0.00238
0.5004	0.00125	0.00128	0.00155	0.00198
0.5979	0.00095	0.00101	0.00124	0.00160
0.7031	0.00067	0.00074	0.00090	0.00117
0.7976	0.00042	0.00050	0.00060	0.00084
0.9001	0.00024	0.00027	0.00030	0.00051
1.0000	0.00012	0.00017	0.00020	0.00023
Sulfolane + 1,3-butanediol + LiClO_4				
0.0000	0.62	19.80	20.70	24.00
0.0984	0.70	22.80	24.55	30.30
0.1977	0.61	18.92	23.41	29.60
0.2992	0.53	6.60	20.80	28.00
0.3992	0.47	0.66	15.70	25.20
0.5006	0.42	0.44	6.70	18.10
0.5989	0.35	0.38	0.39	4.55
0.7019	0.25	0.26	0.32	0.44
0.8007	0.16	0.16	0.26	0.41
0.9076	0.08	0.07	0.12	0.35
1.0000	0.04	0.05	0.09	0.21
Sulfolane + 1,3-butanediol + NaClO_4				
0.0000	0.71	20.33	21.57	26.98
0.1008	0.95	22.42	25.46	31.83
0.1995	0.88	20.24	24.51	30.12
0.2992	0.80	17.58	22.23	28.03
0.4013	0.70	9.07	20.05	25.94
0.4972	0.63	2.16	17.99	23.18
0.5944	0.52	0.44	13.05	17.90
0.7012	0.44	0.33	0.45	3.14
0.8002	0.34	0.29	0.43	0.46
0.9096	0.27	0.25	0.35	0.45
0.9476	0.23	0.24	0.33	0.43
Sulfolane + 1,3-butanediol + NH_4ClO_4				
0.0000	20.52	22.04	25.94	32.30
0.1009	28.69	31.92	37.81	41.23
0.1995	27.08	30.69	36.48	39.90
0.3006	25.37	28.98	34.96	37.91
0.4013	22.71	26.60	32.30	35.53
0.4992	16.63	22.80	29.45	33.35
0.6042	2.85	16.06	24.04	29.45
0.7010	0.80	3.61	10.93	24.99
0.8000	0.39	0.53	0.60	20.71
0.8880	0.33	0.36	0.40	14.06

Standard uncertainties are $u(T) = 0.01$ K, $u(x_1) = 0.0005$, $u(p) = 1$ kPa, $u_r(k_s) = 0.1$.

Table 7. Calculated Constants from the Experimental Data of the Polynomial Model for Specific Electrical Conductivity (k_s)

k_s	β_1	β_2	β_3	β_4	β_5	β_6	R^2
Sulfolane + 1,3-butanediol							
T = 298.15 K	0.0109	-0.0009	-0.062	0.0977	-0.0582	0.0106	0.9996
T = 303.15 K	0.03355	0.0606	-0.0077	0.0788	-0.0565	0.0105	0.9998
T = 308.15 K	-0.0480	0.1974	-0.3161	0.2514	-0.1027	0.0159	0.9989
T = 313.15 K	-0.1162	0.4006	-0.5443	0.3714	-0.1335	0.0199	0.9998
Sulfolane + 1,3-butanediol + LiClO ₄							
T = 298.15 K	-45.703	144.09	-174.53	101.06	-28.347	2.7374	0.9998
T = 303.15 K	1955	-3885	1567.50	1182.30	-943.58	121.31	0.9905
T = 308.15 K	-1650.50	3417.50	-2314.60	725.11	-272.83	55.373	0.9956
T = 313.15 K	-2259.40	5921.50	-5534.30	2384.30	-634.15	97.741	0.9889
Sulfolane + 1,3-butanediol + NaClO ₄							
T = 298.15 K	-58.35	187.56	-236.1	147.42	-47.546	6.4948	0.9987
T = 303.15 K	-523.63	990.69	-545.86	204.24	-192.94	44.639	0.9984
T = 308.15 K	-4783.40	13827	-14853	7357.30	-1742.90	166.30	0.9939
T = 313.15 K	-4157.60	12390	-13727	7031.90	-1744.50	26.929	0.9920
Sulfolane + 1,3-butanediol + NH ₄ ClO ₄							
T = 298.15 K	-8952.70	23623	-23184	10578	-2376.10	234.50	0.9933
T = 303.15 K	-4484.60	13711	-15656	8354.20	-2196.80	252.11	0.9952
T = 308.15 K	51.844	2666.50	-5967.40	4671.60	-1645.30	240.42	0.9984
T = 313.15 K	2974.80	8667.80	-9870.40	5523.20	-1591.50	201.86	0.9998

mass fraction of 1,3-butanediol and increase with temperature increment. From specific conductivity data, molar conductivities, Λ , were extracted using the following equation:

$$\Lambda = k_s / c \quad (17)$$

Figure 3 show values of the molar conductivities of sulfolane + 1,3-butanediol + (LiClO₄, or NaClO₄, or NH₄ClO₄) mixtures. Data indicated that by increasing mass fraction of 1,3-butanediol in the binary mixtures, the electrical conductivity decreased as well. Addition of the perchlorate salts considerably enlarged k_s values at the same temperature. This increment is more considerable in the case of ammonium salt possibly owing to the lesser contribution of the ammonium ions solvation in the sulfolane + 1,3-butanediol binary mixtures.

Also, specific conductivity data as a function of mole fraction of 1,3-butanediol were fitted to polynomial

Eq. (18):

$$y = \beta_6 x^6 + \beta_5 x^5 + \beta_4 x^4 + \beta_3 x^3 + \beta_2 x + \beta_1 \quad (18)$$

Where, generally y is the physical property and $\beta_1, \beta_2, \beta_3, \beta_4, \beta_5$, and β_6 are constants determined from the experimental data. Table 7 contains the coefficients obtained from the polynomial model (Eq. (16))

CONCLUSIONS

Density, refractive index, and electrical conductivity of binary and ternary mixtures comprising (sulfolane + 1,3-butanediol + perchlorate salt) were measured over the whole composition range at different temperatures. The experimental values of density were used to compute the volumetric parameters including the excess molar volume, V_m^E , partial molar volume, $\bar{V}_{m,i}$, and excess partial molar volume $\bar{V}_{m,i}^E$. The excess molar volumes were observed to be

positive and negative values which could be attributed to the predominance of dispersion forces and a contractive trend in terms of molecular interactions. The addition of a perchlorate salt to the binary mixture having sulfolane (2) + 1,3-butanediol (1) increased the contraction effect, which represents a decrease in the excess molar volume values. The values of excess refractive indices were negative and decreased when temperature increased. These observations were in agreement with the changes in values of excess molar volume. Also, the excess refractive indices and excess molar volume were correlated with Redlich-Kister equation. The signs and magnitude of various quantities have been discussed in terms of dipole-dipole interactions, structural effects and complex formation between the component molecules. From the experimental measurements of electrical conductivity, it was observed that the specific electrical conductivity is highest for the solutions containing ammonium perchlorate with the largest cation size.

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