



Study on Interactions of Some Metal Perchlorates Prevailing in Formamide by Physicochemical Approach

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ABSTRACT

The Physicochemical properties, electrolytic conductivities (Λ), densities (ρ), viscosities (η) and speed of sound (u) of lithium perchlorate, sodium perchlorate, and potassium perchlorate have been evaluated in pure formamide at 298.15 K. Association constants (K_A), limiting molar conductances (Λ_0), and co-sphere diameter (R) for ion-pair formation have been obtained from Fuoss conductance equation. Apparent molar volume (ϕ_V), viscosity B -coefficient and apparent molar adiabatic compressibility (ϕ_K) have been calculated from the measurement of density (ρ), viscosity (η) and speed of sound (u), respectively. Masson Equation has been employed to inspect the ion-solvent and ion-ion interactions from the values of limiting apparent molar volume (ϕ_V^0) and experimental slope (S_V^*), respectively. Ion-ion and ion-solvent interactions have been interpreted with the help of the derived parameters A and B obtained from Jones-Dole equations, respectively. The adiabatic compressibility (β_S) and limiting apparent molar adiabatic compressibility (ϕ_K^0) have been assessed using the u values.

Keywords: Metal perchlorates, Ion-solvent interaction, Conductance, Walden's Product, Adiabatic compressibility.

1. INTRODUCTION

Physicochemical study on the transport properties of electrolytes has been utilized to investigate the solvation and association behavior of ions in different solvent media. Electrolytic concentration and viscosity of the solvent play a vital part in deciding the electrical conductivity in solvents.

Perchlorates are the high melting point inorganic salts easily soluble in water and protic solvents. One of the most promising potential applications of lithium perchlorate is in the manufacture of chemical sources of energy (i.e., fuel cells) for electric cars [1]. Sodium perchlorate can be used to block iodine uptake for the patients with subclinical hyperthyroidism [2].

Study of conductance has been employed to measure association and solvation behavior of ions in solution. Techniques such as volumetric, viscometric, and interferometric studies help to investigate the molecular interactions in solution and to inspect the activities of the salt with different solvent concentrations. Study of apparent and limiting apparent molar volumes of the electrolyte along with the dependence of viscosity on the concentration of salt have been demonstrated as a function of ion-ion and ion-solvent interactions [3]. In conjunction with our investigation on electrical

conductance of electrolytes [4-6], the present work deals with the transport and thermodynamic properties of lithium perchlorate, sodium perchlorate and potassium perchlorate in formamide at 298.15 K.

2. EXPERIMENTAL METHODS

2.1. Source and Purity of the Sample

LiClO_4 , NaClO_4 and KClO_4 of puriss grade were obtained from Aldrich, Germany and used as purchased. Formamide of spectroscopy grade was procured from Sd. fine chemicals. standard methods [7] were applied for the purification of the solvent.

2.2. Apparatus and Procedure

A stock solution for each salt was prepared by mass, and the working solutions were obtained by mass dilution at 298.15 K. The conversion of molarity to molality was done using density values. The uncertainty of molarity of different solutions is found to be $\pm 0.0001 \text{ mol dm}^{-3}$. The density (ρ) was measured by a vibrating tube Anton Paar density-meter (DMA 4500M) with a precision of 0.0005 g/cm^3 . The calibration was done by double-distilled water and dry air, and uncertainty in density was $\pm 0.00005 \text{ g cm}^{-3}$.

Solvent viscosities were measured by means of a suspended Ubbelohde-type viscometer being calibrated

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at 298.15 K with doubly distilled water and purified methanol using density and viscosity values from the literature [8-10]. A perfectly dried and thoroughly cleaned viscometer filled with experimental solution was placed vertically in the glass-walled thermostat (Bose Panda Instruments Pvt. Ltd.) maintained to ± 0.01 K. After the attainment of thermal equilibrium, efflux times of flow were recorded with a stopwatch correct to ± 0.1 s. At least three repetitions of each data reproducible to ± 0.1 s were taken to average the flow times. Viscosity of the solution η can be determined using the following equation:

$$\eta = (Kt - L/t)\rho \quad (1)$$

Where K and L are the viscometer constants and t and ρ are the efflux time of flow in seconds and the density of the experimental liquid, respectively. The uncertainty in viscosity measurements is within ± 0.003 mPas.

Conductance measurements were performed in a systronic-308 conductivity bridge (accuracy $\pm 0.01\%$) using a dip-type immersion conductivity cell, CD-10, having a cell constant of approximately (0.1 ± 0.001) . A water bath maintained within $T = (298.15 \pm 0.01)$ K was used, and the cell was calibrated by the method proposed by Lind *et al.* [11]. The conductance data were reported at a frequency of 1 kHz and were uncertain to $\pm 0.3\%$.

Speeds of sound were recorded by multi frequency ultrasonic interferometer (Mittal Enterprise, New Delhi) working at 1 MHz with a precision of 0.2 m.s^{-1} . Its calibration was carried out with three solvents namely water, methanol, and benzene at 298.15 K. The full details of the methods and techniques have been revealed earlier [12-14]. The uncertainty of ultrasonic speed measurements was $\pm 0.2 \text{ m.s}^{-1}$. The uncertainty for the working temperature was ± 0.02 K.

3. RESULT AND DISCUSSION

The experimental values of physical properties of the pure solvent were in good agreement with those found in the literature, as in Table 1. The values of equivalent conductances (Λ) at various concentrations are exhibited in Table 2 and the conductance data for LiClO_4 , NaClO_4 and KClO_4 in Formamide have been examined with the help of the Fuoss conductance equation [15,16]. The limiting molar conductance (Λ_o), the association constant (K_A), and the distance of closest approach of ions (R) are derived for a given set of conductivity values ($c_j, \Lambda_j, j = 1, \dots, n$) from the following set of equations.

$$\Lambda = P\Lambda_o[(1 + R_X) + E_L] \quad (2)$$

$$P = 1 - \alpha(1 - \gamma) \quad (3)$$

$$\gamma = 1 - K_A c \gamma^2 f^2 \quad (4)$$

$$-\ln f = \beta \kappa / 2(1 + \kappa R) \quad (5)$$

$$\beta = e^2 / (\epsilon k_B T) \quad (6)$$

$$K_A = K_R / (1 - \alpha) = K_R / (1 + K_S) \quad (7)$$

Where the relaxation field effect is denoted by R_X , E_L corresponds to the electrophoretic counter current, κ^{-1} denotes the radius of the ion atmosphere, e is the electron charge, ϵ is the relative permittivity of the solvent mixture, k_B is the Boltzmann constant, c is the molarity of the solution, K_A is the overall pairing constant, K_R is the association constant of the solvent-separated pairs, K_S is the association constant of the contact pairs, γ is the fraction of solute present as unpaired ion, α is the fraction of contact pairs, β is twice the Bjerrum distance, f is the activity coefficient and T is the absolute temperature. The computations were performed by dint of using a program suggested by Fuoss. Shedlovsky extrapolation [17] of the data was used to obtain the initial Λ_o values for the iteration procedure. Input for the program is the set ($c_j, \Lambda_j, j = 1, \dots, n$), n, ϵ, η, T , initial values of Λ_o , and instruction to cover a pre-selected range of R values.

For the minimization of standard deviation, all the calculations are performed by finding the values of Λ_o and α .

$$\delta^2 = \sum [\Lambda_j(\text{cal}) - \Lambda_j(\text{obs})]^2 / (n - 2) \quad (8)$$

For a sequence of R values and then plotting δ against R, the minimum of the δ versus R curve represents the best-fit R. Hence, approximate runs are carried out over a fairly wide range of R values using 0.1 increments for the location of the minimum, but no significant minima were found in the δ -R curves for LiClO_4 , NaClO_4 and KClO_4 , in formamide; R values are assumed to be $R = (a + d)$, where the sum of the crystallographic radii of the ions is represented by a and the average distance corresponding to the side of a cell occupied by a solvent molecule is designated by d . This distance d is given by

$$d (\text{\AA}) = 1.183(M/\rho)^{1/3} \quad (9)$$

Where M is the molar mass of the solvent and ρ is its density. The values of Λ_o , K_A , and R attained by this procedure are reported in Table 3.

Inspection of Table 3 reveals the limiting molar conductance Λ_o is found to have an increasing order whilst moving from LiClO_4 , NaClO_4 followed by KClO_4 . It also implies that the association in formamide is highest for LiClO_4 and lowest for KClO_4 and intermediate in the case of NaClO_4 . Hence, the

Table 1: Density (ρ), viscosity (η) and dielectric constant (ϵ) of pure formamide at 298.15 K.

Solute	$\rho \times 10^{-3}$ (kg m ⁻³)		η (mPa. s.)		u (ms ⁻¹)		ϵ
	Expt.	Lit.	Expt.	Lit.	Expt.	Lit.	
Formamide	1.12908	1.1291 [23]	3.299	3.302 [23]	1596.1	1597.8 [24]	111.0

Table 2: The concentration (c) and molar conductance (Λ) of LiClO₄, NaClO₄ and KClO₄ in formamide at 298.15 K.

LiClO ₄		NaClO ₄		KClO ₄	
$c \times 10^4$ (mol.dm ⁻³)	$\Lambda \times 10^4$ (S. m ² . mol ⁻¹)	$c \times 10^4$ (mol.dm ⁻³)	$\Lambda \times 10^4$ (S. m ² . mol ⁻¹)	$c \times 10^4$ (mol.dm ⁻³)	$\Lambda \times 10^4$ (S. m ² . mol ⁻¹)
1.3363	10.31	1.9825	12.38	5.0535	13.05
1.5725	10.16	2.7456	12.24	5.9000	12.85
1.9349	9.98	3.4040	12.01	6.8069	12.76
2.1815	9.88	3.9760	11.73	7.4966	12.56
2.4555	9.75	4.6010	11.53	7.9017	12.45
2.6830	9.64	5.0940	11.35	8.4042	12.38
2.9173	9.51	5.6882	11.23	8.8506	12.36
3.2005	9.37	6.5331	10.98	9.6783	12.14
3.4077	9.27	7.1770	10.86	10.3362	12.08
3.7288	9.10	7.7451	10.62	10.9362	12.01
3.9323	8.99	8.0770	10.64	11.8267	11.79
4.2230	8.85	8.4390	10.53	12.5670	11.73
4.5199	8.72	8.9820	10.31	13.3079	11.51
4.7263	8.63	9.6970	10.16	13.7789	11.44

Table 3: Limiting molar conductivity (Λ°), association constant (K_A), co-sphere diameter (R) and standard deviations (δ) of experimental Λ of LiClO₄, NaClO₄ and KClO₄ at 298.15 K.

Salts	Solvent	Λ° (S·m ² ·mol ⁻¹)	K_A (dm ³ ·mol ⁻¹)	R (Å)	δ
LiClO ₄	Formamide	11.529	921.322	7.6262	5.294
NaClO ₄		13.533	446.743	7.9561	6.168
KClO ₄		14.567	248.296	8.3161	4.895

ion-solvent interaction in formamide is maximum in the case of LiClO₄ and minimum for KClO₄. The ion-solvent interaction of NaClO₄ exists between LiClO₄ and KClO₄. This indicates that lowering of conductance of KClO₄ is found to be highest and least for LiClO₄. The lowest viscosity of Formamide for KClO₄ also supports the above observation because with lowering of viscosity the Λ_o value should increase [18].

The above trend in Λ_o values can be verified through a characteristic function called the Walden product, $\Lambda_o \eta$ given in Table 4. Even though the viscosity of formamide for LiClO₄ is more than that of NaClO₄ and KClO₄, the $\Lambda_o \eta$ is found to attain an increasing order from LiClO₄ to KClO₄ which points out the predominance of Λ_o over η .

ΔG° is given by the following relationship [19] and is given in Table 4.

$$\Delta G^\circ = -RT \ln K_A \quad (10)$$

The explanation of negative values of ΔG° can be given considering the participation of specific covalent interaction in the ion association process. The decrease in the value of ΔG° in formamide for LiClO₄ than KClO₄ indicates the highest degree of association in LiClO₄. High K_A values indicate strong ion association in the media. The ion-association process is exothermic. As a result, gibbs free energy change is negative, and the ion association becomes favorable.

The experimental value of densities of LiClO₄, NaClO₄, and KClO₄ in formamide, as a function of concentration at 298.15 K is listed in Table 5. Limiting apparent molar volume is essential to study the interactions of LiClO₄, NaClO₄, and KClO₄ in the pure solvent. The apparent molar volumes ϕ_V given

in Table 5 were obtained from the solution densities using the following equation.

$$\phi_V = M/\rho - (\rho - \rho_o)/c\rho\rho_o \quad (11)$$

Where M is the molar mass of the solute, c is the molarity of the solution, ρ and ρ_o are the densities of the solution and pure solvent, respectively. The limiting apparent molar volumes ϕ_V^0 were evaluated in accordance with a least-squares treatment to the plots of ϕ_V versus \sqrt{c} using the following Masson equation [20].

$$\phi_V = \phi_V^0 + S_V^* \cdot \sqrt{c} \quad (12)$$

Where ϕ_V^0 is the limiting apparent molar volume at infinite dilution and S_V^* is the experimental slope.

Table 4: Walden product ($\Lambda_o\eta_o$) and Gibb's energy change (ΔG°) of LiClO₄, NaClO₄ and KClO₄ in formamide at 298.15 K.

Solute	$\Lambda_o\eta_o$ (Sm ² mol ⁻¹ mPas. s.)	$\Delta G^\circ \times 10^{-4}$ (J mol ⁻¹)
LiClO ₄	38.034	-1.6939
NaClO ₄	44.645	-1.5125
KClO ₄	48.056	-1.3669

Table 5: Experimental values of densities (ρ), apparent molar volume (ϕ_V), limiting apparent molar volume (ϕ_V^0) and experimental slope (S_V^*) of LiClO₄, NaClO₄ and KClO₄ in formamide at 298.15 K.

c (mol.dm ⁻³)	$\rho \times 10^{-3}$ (kg m ⁻³)	$\phi_V \times 10^6$ (m ³ mol ⁻¹)	$\phi_V^0 \times 10^6$ (m ³ mol ⁻¹)	$S_V^* \times 10^6$ (m ³ mol ^{-3/2} kg ^{1/2})
LiClO ₄ in formamide				
0.0133	1.12940	75.33272	82.3	-59.68
0.0265	1.12981	72.67569		
0.0398	1.13029	70.41229		
0.0532	1.13082	68.54253		
0.0665	1.13140	66.83022		
0.0799	1.13201	65.39346		
NaClO ₄ in formamide				
0.0132	1.12968	72.7480	79.42	-58.74
0.0263	1.13037	69.8869		
0.0397	1.13114	67.6158		
0.0529	1.13194	65.8661		
0.0663	1.1328	64.2178		
0.0798	1.13368	62.9683		
KClO ₄ in formamide				
0.0134	1.13000	68.7985	75.39	-56.46
0.0266	1.13099	66.2733		
0.0399	1.13205	64.1524		
0.0533	1.13317	62.2918		
0.0665	1.13430	60.8549		
0.0801	1.13551	59.4029		

The plots of ϕ_V against the square root of the molar concentration \sqrt{c} were found to be linear with negative slopes which are shown in Figure 1.

The values of ϕ_V^0 and S_V^* along with the standard errors are reported in Table 5. The extent of ion-solvent interaction can be examined by taking the ϕ_V^0 values. A perusal of Table 5 indicates that the ϕ_V^0 values are positive and is highest in case of LiClO₄ in formamide. This indicates that maximum ion-solvent interaction in formamide is found in the case of LiClO₄, and the same is minimum for KClO₄. The said

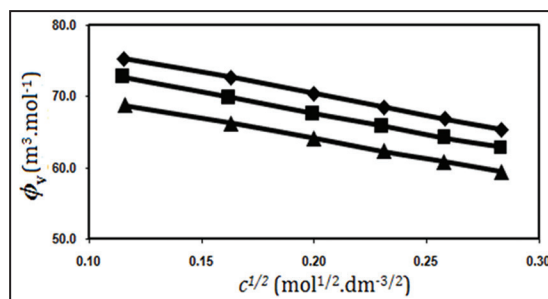


Figure 1: Apparent molar volume (ϕ_V), and the square root of concentrations (\sqrt{c}) for LiClO₄ (—◆—), NaClO₄ (—■—) and KClO₄ (—▲—) in formamide at 298.15 K.

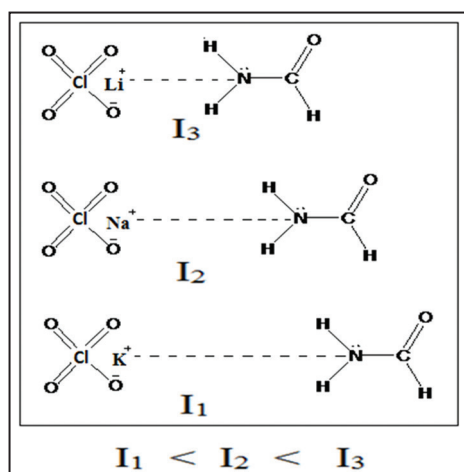
interaction of NaClO_4 becomes in between LiClO_4 and KClO_4 . These effects are further represented in the scheme 1 where I_1 , I_2 , and I_3 are the extents of ion-solvent interaction of KClO_4 , NaClO_4 , and LiClO_4 in formamide, respectively, shown in scheme 1. Li^+ ion with highest charge density and smallest in size compared to Na^+ and K^+ favors the accessibility towards formamide rendering the highest ion solvent interaction exhibited by the following scheme. So the interaction of formamide with Li^+ ion is maximum for LiClO_4 and minimum in the case of K^+ in KClO_4 .

On the other hand, the S_V^* indicates the extent of ion-ion interaction. The S_V^* values are negative due to the disappearance of ion-ion attractive force at infinite dilution. The ion-ion interactions decrease with dilution which can be attributed to increase in distance between ions at infinite dilution. The values of S_V^* show that the extent of ion-ion interaction in Formamide is maximum for KClO_4 and minimum for LiClO_4 . From the comparison of the magnitude of ϕ_V^0 values with that of S_V^* , it is evident that ion-solvent interactions predominate over ion-ion interactions in all the solutions. Furthermore, the values of ϕ_V^0 lead to the fact that the lowest ion-solvent interaction of KClO_4 results in higher conductance than NaClO_4 whereas NaClO_4 exhibits higher conductance than LiClO_4 in Formamide.

The viscosity data has been examined using Jones-Dole equation [21].

$$(\eta/\eta_0 - 1)/\sqrt{c} = A + B\sqrt{c} \quad (13)$$

Where η and η_0 are the viscosities of the solution and solvent respectively. The values of A and B are acquired by a computerized least-square method and recorded in Table 6. This can be achieved from the straight line by plotting $(\eta/\eta_0 - 1)/\sqrt{c}$ against \sqrt{c} as depicted in Figure 2.



Scheme 1: Interaction of LiClO_4 , NaClO_4 and KClO_4 with formamide.

Perusal of Table 6 reveals the negative values of a coefficient are generally negative for all of the solutions under investigation and point toward weak ion-ion interactions. The viscosity B coefficient [22] describes the effects of ion-solvent interactions. Positive values of the viscosity B coefficients for LiClO_4 , NaClO_4 and KClO_4 in formamide reflect the presence of strong ion-solvent interactions. The viscosity B-coefficient value is found to be lowest for KClO_4 supporting the fact that the conductance is highest KClO_4 (Scheme 2).

The adiabatic compressibility (β_S) was calculated from the following equation:

$$\beta_S = 1/u^2\rho \quad (15)$$

Where u is the speed of sound in the solution and ρ is the density of the solution. The apparent molar adiabatic compressibility (ϕ_K) of the solutions was found out from the relation,

$$\phi_K = M\beta_S/\rho + (\beta_S\rho_o - \beta_o\rho) / c\rho\rho_o \quad (16)$$

Where β_S , β_0 are the adiabatic compressibilities of the solution and solvent, respectively, and c is the molarity of the solution. Limiting apparent molar adiabatic compressibilities (ϕ_K^0) and experimental slopes (S_K^*) were achieved by fitting ϕ_K against the square root of molarity (\sqrt{c}) of the electrolyte employing the method of least squares.

$$\phi_K = \phi_K^0 + S_K^* \cdot \sqrt{c} \quad (17)$$

The values of β_S and ϕ_K are recorded in Table 7. Since the values of ϕ_K^0 and S_K^* are the measure of ion-solvent and ion-ion interactions, respectively, a perusal of Table 7 and Figure 3 reveals the values are in agreement with results drawn from the values of ϕ_V^0 and S_V^* discussed earlier.

4. CONCLUSION

Extensive investigation of LiClO_4 , NaClO_4 , and KClO_4 in formamide reveals that LiClO_4 is more associated in formamide than the other two perchlorates, and it remains as ion-pairs. The ion-association is found minimum in

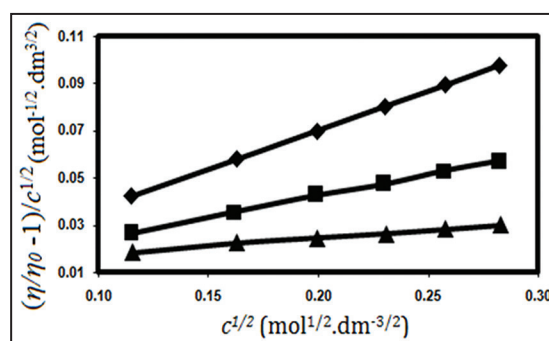
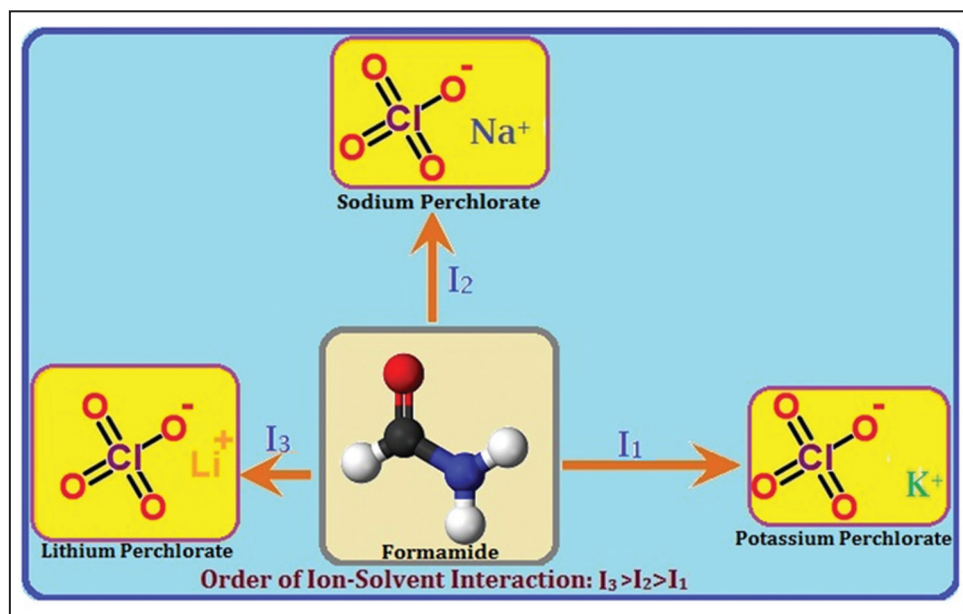


Figure 2: Plots of $(\eta/\eta_0 - 1)/\sqrt{c}$ versus \sqrt{c} for LiClO_4 (—◆—), NaClO_4 (—■—) and KClO_4 (—▲—) in formamide at 298.15 K.

Table 6: Experimental values of concentration (c), viscosity (η) ($\eta/\eta_0-1/2)/c^{1/2}$, viscosity A, B- coefficients of LiClO₄, NaClO₄ and KClO₄ in formamide at 298.15 K.

C (mol dm ⁻³)	η (mPa s)	$(\eta/\eta_0-1/2)/c^{1/2}$ (kg ^{1/2} mol ^{-1/2})	A (kg. mol ⁻¹)	B (kg ^{1/2} mol ^{-1/2})
LiClO ₄ in formamide				
0.0133	3.315	0.042	0.003	0.331
0.0265	3.330	0.058		
0.0398	3.345	0.070		
0.0532	3.360	0.080		
0.0665	3.375	0.089		
0.0799	3.390	0.098		
NaClO ₄ in formamide				
0.0132	3.309	0.026	0.005	0.182
0.0263	3.318	0.036		
0.0397	3.327	0.043		
0.0529	3.335	0.047		
0.0663	3.344	0.053		
0.0798	3.352	0.057		
KClO ₄ in formamide				
0.0134	3.306	0.018	0.010	0.067
0.0266	3.311	0.022		
0.0399	3.315	0.024		
0.0533	3.319	0.026		
0.0665	3.323	0.028		
0.0801	3.327	0.030		



Scheme 2: Extent of ion-solvent interactions of formamide with Alkali Metal Perchlorates.

the case of KClO₄ in formamide. The said interaction of NaClO₄ becomes in between LiClO₄ and KClO₄. The volumetric, viscometric, and interferometric studies reveal the predominance of ion-solvent interaction over the ion-ion interaction in all the studied solutions.

5. ACKNOWLEDGMENTS

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Table 7: Experimental values of speed of sound (u), adiabatic compressibility (β_s) and apparent molar adiabatic compressibility (ϕ_K), limiting apparent molar adiabatic compressibility (ϕ_K^0), and experimental slopes (S_K^*) of LiClO_4 , NaClO_4 and KClO_4 in formamide at 298.15 K.

C (mol dm^{-3})	u (m s^{-1})	$\beta_s \times 10^{10}$ (Pa^{-1})	$\phi_K \times 10^{10}$ ($\text{m}^3 \text{mol}^{-1} \text{Pa}^{-1}$)	$\phi_K^0 \times 10^{10}$ ($\text{m}^3 \text{mol}^{-1} \text{Pa}^{-1}$)	$S_K^* \times 10^{10}$ ($\text{m}^3 \text{mol}^{-3/2} \text{Pa}^{-1} \text{kg}^{1/2}$)
LiClO₄ in formamide					
0.0133	1596.1	3.4766	-0.174	3.372	-31.26
0.0265	1600.2	3.4578	-1.751		
0.0398	1612.4	3.4045	-2.926		
0.0532	1631.9	3.3222	-3.869		
0.0665	1657.5	3.2188	-4.642		
0.0799	1688.5	3.1001	-5.449		
NaClO₄ in formamide					
0.0132	1601.3	3.4764	-0.561	2.665	-28.46
0.0263	1613.3	3.4522	-1.963		
0.0397	1632.4	3.3989	-3.048		
0.0529	1657.2	3.3176	-3.932		
0.0663	1687.1	3.2168	-4.648		
0.0798	1724.1	3.1014	-5.328		
KClO₄ in formamide					
0.0134	1601.4	3.4508	-2.567	1.148	-15.74
0.0266	1609.2	3.4145	-9.320		
0.0399	1620.4	3.3643	-14.833		
0.0533	1634.9	3.3016	-19.531		
0.0665	1651.9	3.2308	-23.329		
0.0801	1672.1	3.1498	-26.924		

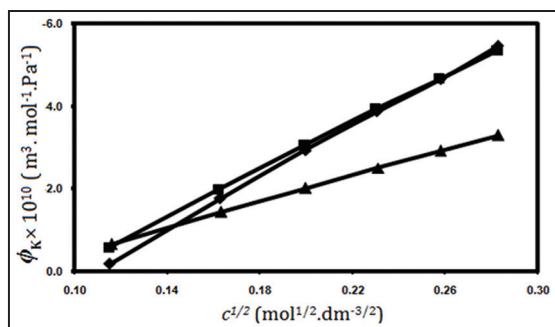


Figure 3: Apparent molar adiabatic compressibilities (ϕ_K) and the square root of concentrations (\sqrt{c}) for LiClO_4 (—◆—), NaClO_4 (—■—) and KClO_4 (—▲—) in formamide at 298.15 K.

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