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Volumetric, Acoustic, and Fourier Transform Infrared Spectroscopic Study of Potassium Thiocyanate in Cyclohexanone, N, N-Dimethylformamide and Dimethyl Sulfoxide

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ABSTRACT

Partial molar volumes of potassium thiocyanate (KSCN) in cyclohexanone, N, N-dimethylformamide, and dimethyl sulfoxide have been determined from solution density measurements at various temperatures and various electrolyte concentrations. Furthermore, adiabatic compressibility of different solutions has been determined from the measurement of ultrasonic speeds of sound at 298.15 K. The experimental density data were evaluated by Masson equation, and the derived parameters were interpreted in terms of ion-solvent and ion-ion interactions. The structure making or breaking capacity of the electrolyte under investigation has been discussed. Analysis of compressibility data indicated the electrostriction of solvent molecules around the metal ion. The infrared spectra of different solutions at 298.15 K suggest the presence of "spectroscopically free" thiocyanate (CNS^-) ion in these solutions. The cation was found to be substantially solvated in these solvents, whereas the anion appeared to have a weak interaction with the solvent molecules. For KSCN, the solvent separated ion-pairs predominate over the contact species, and the observed process has been interpreted by an Eigen multistep mechanism.

Key words: *Partial molar volumes, Adiabatic compressibility, Ion-solvent and ion-ion interactions, Electrostriction, Infrared spectra.*

1. INTRODUCTION

Partial molar volumes of electrolytes provide valuable information about ion-ion, ion-solvent, and solvent-solvent interactions [1]. These informations are of fundamental importance for a proper understanding of the behavior of electrolytes in solution. Measurement of ultrasonic speeds of the solutions also helps in this regard. Such studies are complemented with structural studies of the metal ion solvates by different spectroscopic methods [2]. Recently, we have undertaken a comprehensive program to study the solvation and association behavior of some electrolytes in different aqueous and non-aqueous solvent media from the measurement of various transport, thermodynamic, and spectroscopic properties [3,4]. In this paper, an attempt has been made to reveal the nature of various types of interactions prevailing in solutions of potassium thiocyanate (KSCN) in cyclohexanone, N, N-dimethylformamide (DMF), and dimethyl sulfoxide (DMSO) from volumetric, acoustic, and Fourier transform infrared spectral (FTIR) measurements. The partial molar volumes have been fitted to a polynomial equation in terms of temperature and structure making or breaking capacities of the electrolyte has been inferred from the sign of $(\delta^2 V_\varphi^0 / \delta T^2)_P$.

2. EXPERIMENTAL

2.1. Materials

All the chemicals used were purchased from S. D. Fine Chemicals, India. Cyclohexanone was treated with dilute $KMnO_4$, dried with anhydrous $CaSO_4$ in a vacuum desiccator and then distilled [5]. DMF was purified by the method described by Roy *et al*. [6]. DMSO was kept several days over anhydrous $CaSO_4$, refluxed for 4 h over CaO. Finally, it was distilled at low pressure [7]. The purity of the solvents was ascertained by GLC and also by comparing experimental values of densities and ultrasonic speeds of sound with those reported in the literature [8] as recorded in Table 1. KSCN was purified by recrystallizing twice from conductivity water and was dried in a vacuum desiccator over P_2O_5 for 24 h before use.

2.2. Methods

The densities were measured with a vibrating-tube density meter (Anton Paar, DMA 4500M). The

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Table 1: Physical properties of pure solvents at different temperatures.

Solvent	T (K)	ρ_0 (kg.m^{-3})		u_0 (m.s^{-1})	
		Experimental	Literature	Experimental	Literature
Cyclohexanone	298.15	0.9412	0.9410	1847.2	1847.7
	308.15	0.9363	0.9365	-	-
	318.15	0.9241	0.9242	-	-
DMF	298.15	0.9440	0.9439	1461.6	1462.0
	308.15	0.9348	0.9349	-	-
	318.15	0.9249	0.9345	-	-
DMSO	298.15	1.0952	1.0954	1492.2	1493.0
	308.15	1.0854	1.0858	-	-
	318.15	1.0755	1.0757	-	-

DMF=Dimethylformamide, DMSO=Dimethyl sulfoxide

densitometer was calibrated at the experimental temperatures with doubly distilled, degassed water and dry air at atmospheric pressure. The temperature was automatically kept constant with an accuracy of $\pm 1 \times 10^{-2}$ K using the built-in Peltier technique. The stated repeatability and accuracy of the densities were $\pm 1 \times 10^{-5}$ g.cm^{-3} and $\pm 5 \times 10^{-5\,g}$.cm^{-3}, respectively. The mass measurements accurate to ±0.01 mg were made on a digital electronic analytical balance (Mettler, AG 285, Switzerland). The total uncertainty of density is ±0.0001 g.cm$^{-3,}$ and that of the temperature is ±0.01 K.

The various electrolyte solutions studied here were prepared by mass and the conversion of molality in molarity was accomplished [9] using density values. Ultrasonic speeds of sound for the solutions were determined at 298.15 K by a multifrequency ultrasonic interferometer working at 2 MHz with an uncertainty of ±0.2 ms^{-1}, as described earlier [10]. The experimental values of concentrations (c), densities (ρ),

All FTIR spectra were recorded in the range of 4000-400 cm^{-1}, at a resolution of 1 cm^{-1} using a Shimadzu 8300 FTIR spectrophotometer equipped with a Ge/KBr beam splitter and a DLATGS detector with 20 scans collected for each spectrum. The interferograms were apodized with the Happ-Genzel function. Demountable cells with KBr windows were used.

3. RESULTS AND DISCUSSION

The apparent molar volumes (V_ϕ) were determined from the solution densities using the following equation [11]:

$$V_\varphi = \frac{M}{\rho_0} - \frac{1000(\rho - \rho_0)}{c\rho_0} \tag{1}$$

Where, M is the molar mass of the solute, c is the molarity of the solution, ρ_0 and ρ are the densities of the solvent and solution, respectively. The limiting apparent molar volumes (V^0_ϕ) were calculated using a least-squares treatment to the plots of V_ϕ versus using the following Masson equation [12]:

$$\varphi_V = \varphi_V^0 + S_V^* \sqrt{c} \tag{2}$$

Where, V_φ^0 is the partial molar volume at infinite dilution and S_V^* is the experimental slope. The values of V_φ^0 and S_V^* along with the standard errors are reported in Table 2. Table 2 shows that V_φ^0 values are generally positive and decrease with a rise in temperature. This indicates the presence of strong ion-solvent interactions and these interactions are weakened with a rise in temperature. However, in the case of cyclohexanone solutions, V_φ^0 value increases initially at 308.15 K and then decreases with the increase in temperature (Figure 1). This may be attributed to larger electrostriction of the solvent molecules around the metal ion at 308.15 K and then a slow desolvation of it due to greater thermal agitation at a higher temperature. Similar results were reported earlier by some workers [13]. A perusal of Table 3 also reveals that S_V^* values are negative at 298.15 K for all the solutions of KSCN and S_V^* values increase as the temperature of the solutions increases. Since is a S_V^* measure of ion-ion interactions, the results indicate the presence of weak ion-ion interactions in the solutions at 298.15 K and these interactions increase with a rise in temperature. This is probably due to enhance interstitial accommodation of the cation in the void space left in the packing of solvent molecules.

The partial molar volumes (V_φ^0) were fitted to a polynomial of the following type in terms of absolute temperature (T):

$$V_\varphi^0 = a_0 + a_1 T + a_2 T^2 \tag{3}$$

Values of coefficients of the above equation for different KSCN solutions are reported in Table 3. From

Table 2: Densities (ρ), partial molar volumes (V_ϕ), the experimental slopes (S_V^*), and apparent molar volumes (V_φ^0) of KSCN in solvents at different temperatures.

c ($mol.dm^{-3}$)	ρ ($g.cm^{-3}$)	V_ϕ ($cm^3.mol^{-1}$)	V_φ^0 ($cm^3.mol^{-1}$)	S_V^* ($cm^2. dm^{1/2}.mol^{-3/2}$)
Cyclohexanone+KSCN				
T=298.15 K				
0.0053	0.9414	60.60	79.11 (±1.34)	−265.79
0.0073	0.9415	56.00		
0.0136	0.9419	47.60		
0.0188	0.9423	42.20		
0.0230	0.9426	38.58		
0.0293	0.9431	34.35		
T=308.15 K				
0.0052	0.9364	73.00	90.21 (±1.53)	−241.93
0.0072	0.9365	69.70		
0.0135	0.9368	62.00		
0.0187	0.9371	56.70		
0.0229	0.9374	53.40		
0.0292	0.9378	49.70		
T=318.15 K				
0.0051	0.9242	76.60	88.32 (±1.37)	−168.79
0.0071	0.9243	73.50		
0.0134	0.9245	69.00		
0.0185	0.9248	65.10		
0.0226	0.9250	63.00		
0.0288	0.9253	59.92		
DMF+KSCN				
T=298.15 K				
0.0051	0.9441	82.16	99.64 (±2.02)	−245.02
0.0071	0.9442	78.20		
0.0131	0.9444	72.20		
0.0182	0.9446	68.00		
0.0223	0.9449	62.20		
0.0283	0.9452	58.02		
T=308.15 K				
0.0050	0.9351	43.90	31.58 (±1.03)	172.92
0.0070	0.9352	46.00		
0.0130	0.9354	51.40		
0.0180	0.9356	54.70		
0.0221	0.9358	56.90		
0.0280	0.9359	60.80		
T=318.15 K				
0.0049	0.9252	33.60	20.58 (±0.88)	176.28
0.0069	0.9253	35.20		
0.0129	0.9257	40.00		
0.0178	0.9259	43.60		

(Cond...)

Table 2: *(Continued...)*

c ($mol.dm^{-3}$)	ρ ($g.cm^{-3}$)	V_ϕ ($cm^3.mol^{-1}$)	V_φ^0 ($cm^3.mol^{-1}$)	S_V^* ($cm^2. dm^{1/2}.mol^{-3/2}$)
0.0219	0.9261	46.50		
0.0277	0.9263	50.60		
DMSO+KSCN				
T=298.15 K				
0.0056	1.09525	75.30	87.07 (±1.45)	−170.38
0.0079	1.09532	71.10		
0.0146	1.09552	66.60		
0.0201	1.09575	62.20		
0.0246	1.09594	60.00		
0.0314	1.09624	57.70		
T=308.15 K				
0.0055	1.0857	36.90	23.68 (±0.93)	179.57
0.0078	1.08588	39.50		
0.0144	1.08599	45.10		
0.0200	1.08616	49.50		
0.0244	1.08642	52.00		
0.0311	1.08665	54.90		
T=318.15 K				
0.0054	1.07559	35.20	19.98 (±0.56)	210.53
0.0077	1.07575	38.10		
0.0143	1.07606	45.30		
0.0198	1.07631	50.90		
0.0242	1.07647	53.70		
0.0308	1.0771	55.30		

Standard errors are given in parenthesis. DMF=Dimethylformamide, DMSO=Dimethyl sulfoxide, KSCN=Potassium thiocyanate

Table 3: Values of coefficients of Equation (3) for different KSCN solutions.

Solution	a_0 ($cm^3.mol^{-1}$)	a_1 ($cm^3.mol^{-1}$)	a_2 ($cm^3.mol^{-1}$)
Cyclohexanone+ KSCN	−6219.128	40.489	−0.065
DMF+KSCN	28340.840	−179.784	0.285
DMSO+KSCN	29397.191	−187.290	0.298

DMF=Dimethylformamide, DMSO=Dimethyl sulfoxide, KSCN=Potassium thiocyanate

the values of coefficients, the following equations are obtained:

For cyclohexanone solutions:

$$V_\varphi^0 = -6219.128/\text{cm}^3.\text{mol}^{-1} + 40.489\,\text{T cm}^{-3}.\text{mol}^{-1} -0.065\,\text{T}^2\ \text{cm}^{-3}.\text{mol}^{-1} \tag{4}$$

For DMF solutions:

$$V_\varphi^0 = 28340.840\,\text{cm}^{-3}.\text{mol}^{-1} - 179.784\,\text{T cm}^{-3}.\text{mol}^{-1} +0.285\,\text{T}^2\ \text{cm}^{-3}.\text{mol}^{-1} \tag{5}$$

For DMSO solutions:

$$V_\varphi^0 = 29397.191\,\text{cm}^{-3}.\text{mol}^{-1} - 187.290\,\text{T cm}^{-3}.\text{mol}^{-1} +0.298\,\text{T}^2\ \text{cm}^{-3}.\text{mol}^{-1} \tag{6}$$

The partial molar expansibilities (φ_E^0) can be obtained by the following equation**:**

$$\varphi_E^0 = \left(\delta V_\varphi^0 / \delta \text{T}\right)_\text{P} = \text{a}_1 + 2\text{a}_2\text{T} \tag{7}$$

The values φ_E^0 of for different solutions of the studied electrolyte at 298.15, 308.15, and 318.15 K are reported in Table 4.

Table 4: The values of limiting partial molar expansibilities (φ_E^0) for different solutions of KSCN different temperatures.

Solution	φ_E^0 (cm^3.mol^{-1}.K^{-1}) 298.15 K	φ_E^0 (cm^3.mol^{-1}.K^{-1}) 308.15 K	φ_E^0 (cm^3.mol^{-1}.K^{-1}) 318.15 K	$\left(\frac{\delta\varphi_E^0}{\delta T}\right)_p$
Cyclohexanone+ KSCN	1.729	0.429	−0.870	Negative
DMF+KSCN	−9.838	−4.138	1.561	Positive
DMSO+KSCN	−9.593	−3.636	2.327	Positive

DMF=Dimethylformamide, DMSO=Dimethyl sulfoxide, KSCN=Potassium thiocyanate

According to Hepler [14], the sign of $(\delta^2 V_\varphi^0/\delta T^2)_P$ is a better criterion than S_V^* in characterizing the long-range structure making and breaking capacity of the electrolytes in solution. The general thermodynamic expression used is as follows:

$$\left(\delta C_P/\delta P\right)_T = -\left(\delta^2 V_\varphi^0/\delta T^2\right)_P \tag{8}$$

If the sign of $(\delta^2 V_\varphi^0/\delta T^2)_P$ is positive the electrolyte is a structure maker, else it is a structure breaker. As evident from Table 4, the electrolyte under investigation is a structure maker in all the experimental solvents except cyclohexanone.

The adiabatic compressibility (β) was evaluated from the following equation:

$$\beta = u^{-2}\rho^{-2} \tag{9}$$

Where, ρ is the solution density and u is the sound speed in the solution. The partial molal adiabatic compressibility (ϕ_K) of the solutions was determined from the relation [10],

$$\varphi_K = \frac{M\beta}{\rho_0} + \frac{1000(\beta\rho_0 - \beta_0\rho)}{m\rho\rho_0} \tag{10}$$

Where, β_0, β are the adiabatic compressibility of the solvent and solution, respectively, and m is the molality of the solution. The limiting partial molal adiabatic compressibility (φ_K^0) and the experimental slope (S_K^*) were obtained by fitting ϕ_K against the square root of molality of the electrolyte ($\sqrt{m}$) by the method of least-squares.

$$\varphi_K = \varphi_K^0 + S_K^*\sqrt{m} \tag{11}$$

The values of m, u, β, ϕ_K, S_K^*, and are presented in Table 5. Figure 2 shows the relation between m and u. A perusal of Table 5 shows that φ_K^0 values are positive and S_K^* values are negative for all the solutions. Since the values φ_K^0 of and S_K^* are measures of the ion-solvent and ion-ion interactions, respective, the results are in good agreement with those drawn from the values V_φ^0 of and S_V^* discussed earlier.

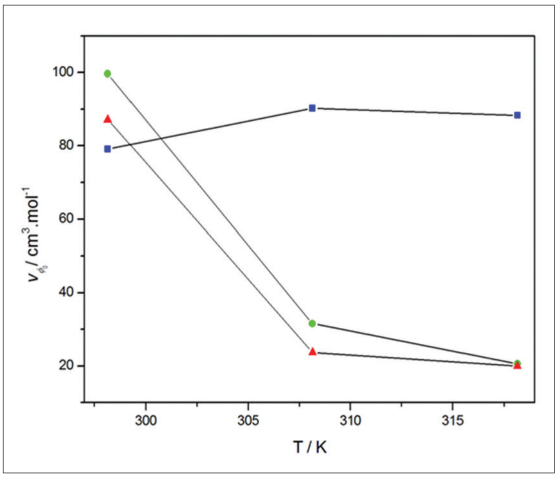

Figure 1: Plots of partial molar volumes (V_φ^0) against temperatures: Cyclohexanone (■); dimethylformamide (●); and dimethyl sulfoxide (▲).

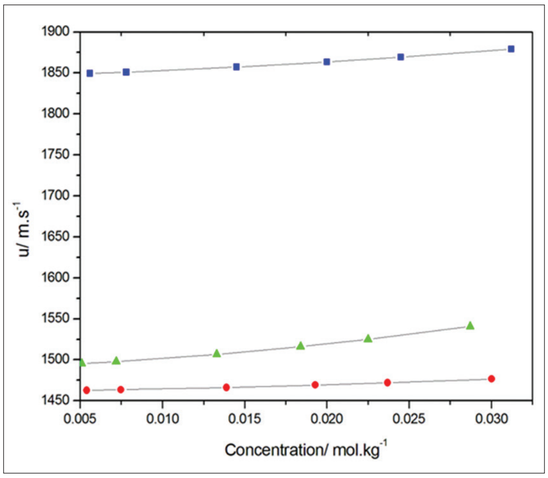

Figure 2: Plots of sound velocity against molality of potassium thiocyanate solutions: cyclohexanone (■); dimethyl sulfoxide (●); and dimethylformamide (▲).

IR studies have been done in DMF, DMSO, and cyclohexanone solvents. In the IR spectra of DMF [15], the band at 2931 cm^{-1} is assigned for C-H stretching frequency of cis methyl group. The band corresponds to 1390, 1255, and 1098 cm^{-1} are assigned for C-N stretching, N-CH_3 stretching, and NC_mH deformation, respectively. The band at 661 cm^{-1} is assigned for O=CNC torsion, and C=O stretching frequency appeared at 1664 cm^{-1}. The addition of KSCN to this solvent would result in the frequency shift in the IR spectra that could be assigned to a DMF-K^+ interaction. On the addition of KSCN to DMF, the band at 2931 and 1255 cm^{-1} did not show any noticeable frequency

Table 5: The values of m, u, β, φ_K, φ_K^0, and S_K^* of KSCN in different solvents.

m (mol.kg^{-1})	u (m.s^{-1})	β×10^{10} (Pa^{-1})	φ_K ×10^{10} (m^3.mol^{-1}.Pa^{-1})	φ_K^0 ×10^{10} (m^3.mol^{-1}.Pa^{-1})	S_K^* ×10^{10} (m^3.mol$^{-3/2}$.Pa^{-1}.kg$^{1/2}$)
Cyclohexanone+KSCN					
0.0056	1849.5	3.1053	−1.42	0.241	−22.28
0.0078	1851.0	3.0999	−1.71		
0.0145	1857.2	3.0782	−2.46		
0.0200	1863.4	3.0565	−2.92		
0.0245	1869.4	3.0357	−3.27		
0.0312	1879.0	3.0033	−3.66		
DMF+KSCN					
0.0054	1462.6	4.9516	−0.99	0.793	−24.77
0.0075	1463.3	4.9464	−1.38		
0.0139	1466.1	4.9262	−2.13		
0.0193	1469.3	4.9040	−2.67		
0.0237	1472.0	4.8841	−3.04		
0.0300	1476.5	4.8528	−3.46		
DMSO+KSCN					
0.0051	1495.5	4.0822	−3.00	0.605	−50.44
0.0072	1497.8	4.0693	−3.71		
0.0133	1506.5	4.0216	−5.17		
0.0184	1516.0	3.9708	−6.20		
0.0225	1525.0	3.9233	−6.97		
0.0287	1540.6	3.8433	−7.97		

DMF=Dimethylformamide, DMSO=Dimethyl sulfoxide, KSCN=Potassium thiocyanate

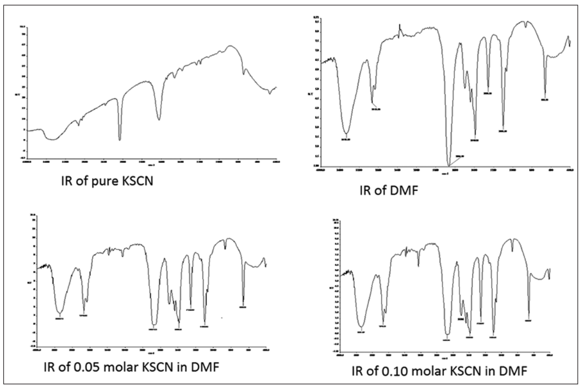

Figure 3: Infrared spectrum of pure potassium thiocyanate (KSCN), dimethylformamide (DMF) and KSCN in DMF.

shift, but the peak at 1664, 1390, and 661 cm^{-1} showed an appreciable frequency shift in the IR spectra. The C=O frequency at 1664 cm^{-1} of pure DMF has shifted to 1677 and 1672 cm^{-1} for 0.05M KSCN and 0.1 M KSCN, respectively. This shift in frequency may be attributed for the interaction or solvation of K$^+$ ion by C=O group of DMF. Different band values of KSCN in DMF are shown in Table 6 (Figure 3).

DMSO is a sulfur analog of acetone where a sulfur atom replaces the carbonyl carbon. In IR spectra of DMSO [16], the peak at 1435.85 cm^{-1} and 1311.89 cm^{-1} are attributed for the anti-symmetric bending of CH_3 and symmetric deformation of CH_3 group, respectively. The peak at 1052.88 cm^{-1} can be assigned as S=O stretching. On the addition of KSCN to DMSO solvent, the peak at 1435 cm^{-1} did not show any remarkable change but the peak at 1311 cm^{-1} got appreciably shifted to 1313.66 cm^{-1}. Again the stretching frequency ay 1053 cm^{-1} corresponding to S=O shifted to 1027.22 cm^{-1}. This shifting of frequency may be attributed for the interaction between the cation (K$^+$) and the S=O bond of DMSO solvent. Furthermore, the C-S stretching frequency at 701 cm^{-1} of pure DMSO has been shifted to 703.29 cm^{-1} on the addition of KSCN. Different band values of KSCN in DMSO are shown in Table 6 (Figure 4).

In the IR spectra of cyclohexanone [17], the peak at 2939 and 2864 cm^{-1} corresponds to C-C ring stretching vibration and the peak at 1712 cm^{-1} is exhibited for C=O stretching frequency. When KSCN is added to this solvent, the IR spectra did not show any

Table 6: Different band values for IR of different pure solvents and KSCN with solvents.

Band cm^{-1}			Assigned mode
DMF pure	**DMF+0.05 m KSCN**	**DMF+0.1 m KSCN**	
661.55	660.70	661.97	O=CNC tor
1098.33	1096.62	1097.02	NC_mH def
1255.53	1255.90	1255.97	NCH_3 str
1390.70	1389.81	1389.98	CN str
1664.50	1677.25	1672.91	C=O str
2931.56	2930.62	2931.21	CH str (cis methyl)
661.55	660.70	661.97	O=CNC tor
701.09	710.71	701.12	C-S str
1052.88	1046.42	1046.02	S=O str
1311.89	1312.20	1312.21	$-CH_3$ symmetric def
1435.85	1435.90	1433.72	$-CH_3$ asymmetric bending
2907.09	2908.51	2908.37	C-H asymmetric str
2990.52	2991.48	2990.54	C-H asymmetric str
701.09	710.71	701.12	C-S str
Cyclohexanone pure	**Cyclohexanone+0.05 m KSCN**	**Cyclohexanone+0.1 m KSCN**	
1119.39	1119.39	1119.41	
1222.81	1222.78	1222.80	
1712.70	1712.54	1712.26	C=O stretching
1864.54	2864.54	2864.54	C-C ring str
2939.65	2939.55	1939.59	C-C ring str

KSCN=Potassium thiocyanate, IR=Infrared

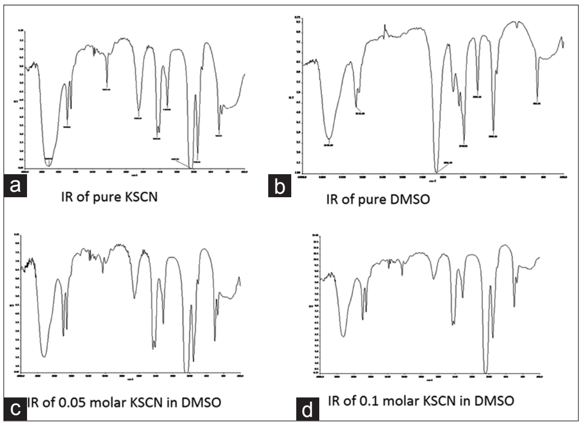

Figure 4: Infrared (IR) spectrum of pure potassium thiocyanate (KSCN), dimethyl sulfoxide (DMSO) and KSCN in DMSO. (a) IR of pure KSCN, (b) IR of pure DMSO, (c) IR of 0.05 molar KSCN in DMSO, (d) IR of 0.1 molar KSCN in DMSO.

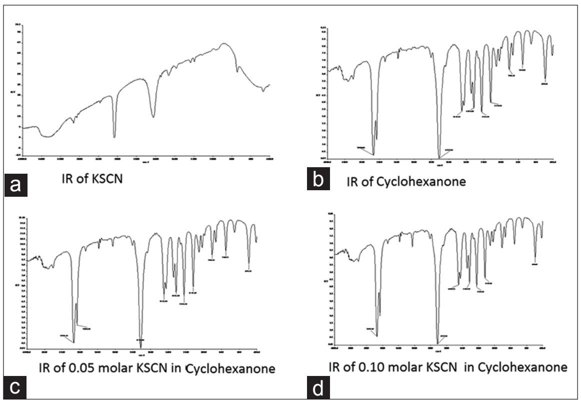

Figure 5: Infrared (IR) spectrum of pure potassium thiocyanate (KSCN), cyclohexanone and KSCN in cyclohexanone. (a) IR of KSCN, (b) IR of cyclohexanone, (c) IR of 0.05 molar KSCN cyclohexanone, (d) IR of 0.10 molar KSCN cyclohexanone.

appreciable frequency shift in the C-C ring stretching region. Only small frequency shift is observed in the carbonyl (C=O) stretching region. This small frequency shift indicates that very weak interaction exists between solvent and the solute (KSCN). The band values are shown in Table 6 (Figure 5).

4. CONCLUSION

In summary, partial molar volumes (φ_V^0) and adiabatic compressibilities of KSCN different solvents indicate the presence of strong solute-solvent interactions. Furthermore, the trends in the sign of $(\delta^2 V_\varphi^0 / \delta T^2)_P$ for the electrolyte show that it is a structure maker in all the experimental solvents except cyclohexanone

5. ACKNOWLEDGMENTS

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