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Determination of Thermodynamic Parameters of $VOSO_4$ in Different Organic Mixed Solvents

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Abstract

The measurements of vanadyl sulfate conductance have been in mixed solvent with methanol-H_2O (MeOH-H_2O), and N, N- Dimethyl formamide (DMF-H_2O) in different mass fraction 10%, 30% and 50% at four different temperatures from 298.15 to 313.15K. The experimental data have been analyzed by using the Fuoss-Shedlovsky extrapolation technique. Estimated thermodynamic parameters for association were calculated and discussed. These parameters such as molar conductance (Λ_m), limiting molar conductance (Λ_0), Walden product ($\Lambda_0 \eta_0$), ion-pair association constant (K_A), the activation energy for the transport process (E_a) and the standard thermodynamic parameters for association (ΔGo_A, ΔHo_A and ΔSo_A). The different thermodynamic parameters for Vanadyl sulfate were estimated and compared the result of organic solvent over wide range of temperature.

Keywords: Ion-pair association constants; Molar conductance; Organic mixed solvents; Walden product; Vanadyl sulfate

Introduction

Studying of thermodynamic properties of solution mixture has great important in theoretical and applied areas of research and useful in study of the intermolecular interaction and geometrical effects in the system, thermo-physical and bulk properties of solution and important in many other fields of industry [1-3]. The Fuoss-Shedlovsky is one of the mathematic equation of conductivity theories, which has been successfully used to investigate many electrolytes in solutions [4-9]. The physical properties of the binary mixed solvents like the viscosity and the relative permittivity can be varied over a wide range making them a favorite solvent system for the study of ion association and ion mobility. Recently, a study of the properties of vanadium compounds attracts tremendous attention due to its essential in many fields such as biochemistry, medical and in many industry [10-12]. It forms numerous inorganic compounds as vanadyl sulfate. Vanadyl sulfate is a component of food supplements and experimental drugs. Vanadyl sulfate exhibits insulin-like effects [13]. Vanadium compounds exhibit antitumor or anticarcinogenic properties. Vanadium (IV) oxide sulfate is a known inorganic compound of vanadium, which has a high physiological and industrial importance [14-20]. One of the importance of vanadyl sulfate used in industry as vanadium battery, its efforts such as longer life, better stability, higher efficiency and easy operation. N, N-dimethylformamide (DMF) is called super solvent, with all common polar and nonpolar solvents due to its miscibility. It is used in the manufacture of several chemical products and intermediates as a parent substance [21]. The binary mixed solvents (DMF-H_2O) are the favorite solvents can be used for studying of ion association and ion mobility, due to the change over a wide range of its physical properties. Investigate ion association by using conductivity technique enable us to know the factors affecting the thermodynamic and kinetic stability. These properties depend on the effective size of the ions, strong ion-solvent interactions as well as change the structure of the solvents. In this paper we found that the nature of ion-solvent interaction occurring in the medium depend on ion-pairing in salt solutions. Studying association phenomena show the effect of all parameters on the transport properties of vanadyl sulfate in a binary mixed solvent with alcohol mass fraction 10%, 30% and 50% (MeOH-H_2O) and (DMF-H_2O) at four different temperatures from 298.15 to 313.15K (with a step of 5K) by applying the Fuoss- Shedlovsky conductivity equation [22]. The association constant is higher in DMF-H_2O than that in MeOH-H_2O due to the formation of bulker DMF-water aggregates that hinder the movement of ions and lead to the formation of ion pairs.

Materials and Methods

Vanadyl sulfate ($VOSO_4$. 99.5%), methanol (MeOH, 99.6%), and (DMF 99.9%) were supplied from Sigma-Aldrich company and used without further purification. Bi-distilled water with a specific conductivity of 0.09 μS cm^{-1} at room temperature was furthermore used for binary solvents preparation. Specific conductance measurements of the salt solutions that previously prepared were measured using LF 191(Germany) conductivity meter (accuracy ± 0.01%) of a cell constant value 1 ± 0% cm^{-1}. The cell constant was determined with potassium chloride solutions [23-28]. MLW 3230 ultra-thermostat was connected to the Conductivity Bridge to give constant temperature (± 0.006 °C)

Binary mixed solvent of (methanol - water) and (DMF-water) with the alcohol mass fractions of 10%, 30%, and 50% were selected to be the solvent media for this study and were prepared by mixing required volume of methanol and dimethyl formamide with water (with error ± 0.01%) by applying the following equation:

$$\text{Alcohol percentage}=(V_1d_1)100/\,(V_1d_1+V_2d_2) \qquad (1)$$

Where d_1 and d_2 are the density of alcohol and water respectively. V_1 is the volume of alcohol which will be added to the volume V_2 of water to get the mixture of the required percentage. The physical properties, relative permittivity (ε), and viscosity (η^o) of (methanol -

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water) and (DMF- water) with the alcohol mass fractions of 10%, 30%, and 50% at temperatures from 298.15 to 313.15K (with a step of 5K) were tabulated in Table 1 [29-32].

Solvent	T/K	ε	η/m Pa.s
10% (MeOH-H_2O)	298.15	74.31	0.7459
	303.15	72.33	0.7018
	308.15	70.44	0.6508
	313.15	68.23	0.6107
30% (MeOH-H_2O)	298.15	65.4	0.7982
	303.15	63.75	0.7528
	308.15	61.79	0.6924
	313.15	59.67	0.6541
50% (MeOH-H_2O)	298.15	55.32	0.841
	303.15	53.56	0.8321
	308.15	51.54	0.8278
	313.15	49.76	0.8115
10% (DMF-H_2O)	298.15	76.1	0.7899
	303.15	74.12	0.7451
	308.15	72.21	0.691
	313.15	70.31	0.6512
30% (DMF-H_2O)	298.15	67.05	0.8436
	303.15	65.11	0.8036
	308.15	63.65	0.7562
	313.15	61.13	0.7123
50% (DMF-H_2O)	298.15	58.34	0.8765
	303.15	56.76	0.8345
	308.15	54.78	0.8012
	313.15	52.56	0.7988

Table 1: The relative permittivity (ε), and viscosity (η, m Pa.s) at different temperatures of the used solvents.

Results and Discussion

The molar conductance (Λ_M) for $VOSO_4$ solutions in binary mixed solvents at different temperatures was calculated by applying Equation (2).

$$\Lambda_m = \frac{\left(K_s - K_{solv}\right).K_{cell}.1000}{C} \quad (2)$$

Where K_s and K_{solv} are the specfic conductance of the solution and the solvent respectively, Kcell is the cell constant and C is the molar concentration of the metal salt solution.

The experimental conductance data were analyzed by using Fuoss-Shedlovsky conductivity equations. The limiting molar conductance (Λ_o) were estimated for vanadyl sulfate solutions in H_2O, (DMF-H_2O) and (MeOH-H_2O) mixed solvents at different temperatures by extrapolating the linear Onsager plot [33] between (Λ_m) and (C)½ as shown in Figure 1.

$$\frac{1}{\Lambda_m S(Z)} = \frac{1}{\Lambda_0} + \left(\frac{K_\Lambda}{\Lambda_0}\right)\left(C\,\Lambda_m\,\gamma_{\pm}^2 S(Z)\right) \quad (3)$$

S (Z) = 1 + Z + Z2/2 + Z3/8 + …… etc. (4)

$$Z = \frac{S(\Lambda_m C)^{1/2}}{\Lambda_0^{3/2}} \quad (5)$$

Where S, Z, and S(Z) are all Fuoss-Shedlovsky parameters.

The value of (Λ_o) was used to calculate the Onsager slope (S) from the Equation (6).

$S = a\Lambda_o + b$ (6)

$a = 8.2 \times 10^5 / (\varepsilon T)^{3/2}$ (7)

$b = 82.4/ \eta((\varepsilon T)^{1/2}$ (8)

Where (ε) is the dielectric constant of the solvent, (η) is the viscosity of solvent and (T) is the temperature. Using the data of (Λm), S (z) and (Λo), the values of degree of dissociation (α) were calculated by using the following equation:

$$(\alpha) = \frac{\Lambda_m S(Z)}{\Lambda_o} \quad (9)$$

Using these (α) and (ε) values, the mean activity coefficients (γ±) were evaluated by means of Equation (10).

$$\log\gamma = -\frac{Z_+ Z_- A\sqrt{I}}{I + Br^0\sqrt{I}} \quad (10)$$

Where Z_-, Z_+ are the charges of ions in solutions A, B are the Debye-Hückel constant.

$A = 1.824 \times 10^6 (\varepsilon T)^{-3/2}$ (11)

$B = 50.29 \times 10^8 (\varepsilon T)^{-1/2}$ (12)

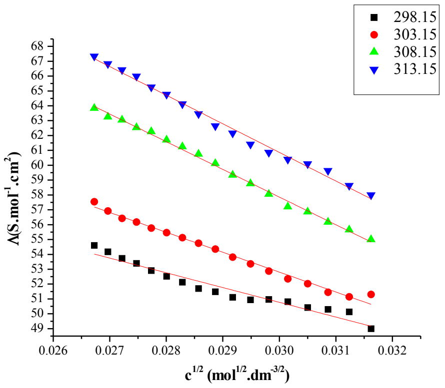

Figure 1: The plot of (Λ) versus ($C^{1/2}$, $mol^{1/2}$.$dm^{-3/2}$) in 30% (DMF-H_2O) solvents for vanadyl sulphate at different temperatures.

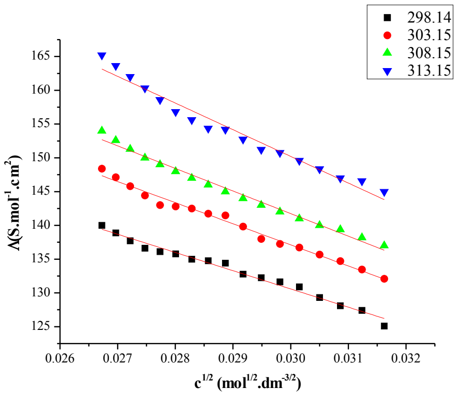

Figure 2: The plot of (Λ) versus ($C^{1/2}$, $mol^{1/2}\ dm^{-3/2}$) in 30%(MeOH-H_2O) solvents for vanadyl sulphate at different temperatures.

From Figures 1 and 2 we calculated data of limiting molar conductance as a function of concentration and compared the value of 30% (DMF-H_2O) and 30% (MeOH-H_2O) observed that limiting molar conductance in case of methanol is greater than that in case of dimethyl formamide according to the following trend (MeOH-H_2O)>(DMF-H_2O) this due to higher intermolecular hydrogen bonding in DMF.

The obtained results of limiting molar conductance and degree of dissociation for vanadyl sulfate Table 2 in case of increasing percentage of a binary mixed solvent of both (MeOH-H_2O) and (DMF-H_2O) 50% and 30% is lower than in case of 10% at different temperature, this due to conductivity values decrease when mixed solvent percentage increase due to formation of intermolecular and intramolecular hydrogen bonding which make hindrance to the ions mobility, in case of DMF-H_2O limiting molar conductance value decrease than in case of methanol-H_2O solvent due to presence of (-OH group) in methanol which give degree of polarity than in DMF in addition to effect of higher dielectric constant and viscosity values. Although degree of dissociation inversely proportional with limiting molar conductance as in equation (9) and the calculated value proved that as shown in Table 2.

Solvent	T/K	Λ_o(S cm^2 mol^{-1})	α
10% (MeOH-H_2O)	298.15	225.45	0.735
	303.15	245.95	0.682
	308.15	271.27	0.621
	313.15	284.55	0.598
30% (MeOH-H_2O)	298.15	211.22	0.635
	303.15	230.85	0.606
	308.15	242.22	0.547
	313.15	268.33	0.491
50% (MeOH-H_2O)	298.15	98.93	0.590
	303.15	110.79	0.545
	308.15	122.63	0.537
	313.15	136.48	0.502
10% (DMF-H_2O)	298.15	99.23	0.636
	303.15	101.32	0.627
	308.15	114.39	0.589
	313.15	128.22	0.552
30% (DMF-H_2O)	298.15	80.54	0.649
	303.15	94.14	0.581
	308.15	113.73	0.527
	313.15	118.63	0.520
50% (DMF-H_2O)	298.15	51.83	0.696
	303.15	59.45	0.634
	308.15	69.02	0.591
	313.15	80.36	0.536

Table 2: The limiting molar conductances (Λ_o, S cm^{-2} mol^{-1}), and the degree of dissociation (α) of vanadyl sulfate in the used solvents at different temperatures.

Ion-pair association constant: The mean values of the ion-pair association constant (K_A) of all systems under study were represented in Table 3. It was found that the association constant for vanadyl sulfate in the used solvents, increases with increasing temperature which indicates an endothermic association process. This temperature dependence of the association process of ions can be explained from the relation between desolvation and association of ions whereas the temperature increases, the desolvation process of ions take place, the ions will become too closer together, therefore the association of ions increases [34]. The obtained result show that the association constant increase as the proportions of organic solvent increase and increase more in case of DMF-H_2O solvent than in MeOH-H_2O. The association constant values for vanadyl sulfate in 50% (MeOH-H_2O) is higher than values of association constant in 10% and the same for DMF-H_2O. This may be related to the interaction between methanol and water, DMF and water at a low concentration affected by the formation of cage-like structures, commonly known as (icebergs) around the hydrophobic ends of the methanol, and DMF which effect on the mobility of ions and decrease association constant in case of lower concentration [35] as tabulated in Table 3.

Solvent	T/K	K_A	$\Lambda_o\eta$
10% (MeOH-H_2O)	298.15	639.78	17.81
	303.15	787.88	16.96
	308.15	1275.4	16.43
	313.15	1458.28	16.04
30% (MeOH-H_2O)	298.15	798.07	16.84
	303.15	819.14	16.52
	308.15	1418.22	15.97
	313.15	1583.96	15.21
50% (MeOH-H_2O)	298.15	919.31	10.90
	303.15	1021.32	9.822
	308.15	1559.58	9.614
	313.15	1696.03	9.258
10% (DMF-H_2O)	298.15	1127.06	7.883
	303.15	1253.58	7.547

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	308.15	1524.1	7.309
	313.15	1895.50	7.111
30% (DMF-H_2O)	298.15	1098.10	6.753
	303.15	1638.41	6.521
	308.15	2204.45	6.311
	313.15	2218.63	6.175
50% (DMF-H_2O)	298.15	1861.86	6.419
	303.15	2198.54	6.120
	308.15	2228.15	5.923
	313.15	2525.47	5.791

Table 3: The mean ion pair association constant (K_A, mol. dm^{-3}), Walden product ($\Lambda_o\eta$, S. mol^{-1} cm^2 Pa.s) and the hydrodynamic radii (R_H) of vanadyl sulfate in the used solvents at different temperatures.

Walden product: The Walden product (Λ_0 η_0 or λ_0 η_0) is very important from the point of view of ion-solvent interaction [36,37].

Walden has written his rule in the form as in Equation (13).

$$\Lambda_0 \eta_0 = 0.82\left[\frac{1}{r_s^+} + \frac{1}{r_s^-}\right] \quad (13)$$

The factor $\left(\frac{1}{[1/r_s^+ + 1/r_s^+]}\right)$ is a measure of the hydrodynamic radii (RH) of the ions, r is the radius of a hypothetical sphere that diffuses with the same speed as the particle under study. In practice, the solute molecules in solution are solvated and dynamic. So, the radius calculated from the diffusion properties of the particle is indicative of the apparent size of the dynamic hydrated/solvated particle, and the hydrodynamic radius. The inverse behavior of the hydrodynamic radii with Walden product was reported by many authors [38-41].Walden product inversely proportional with temperature as it decreases with increasing temperature. This may be related to the increase in the size of solvated ions in the mixtures as we raise the temperature which occur due to the variation of the proportion of the methanol- H_2O molecules and the same for DMF-H_2O in the sheath of solvation with temperature. Walden product are affected by two factors, limiting molar conductance which is directly proportional to temperature, and viscosity which is inversely proportional to temperature. From this observation, we can conclude that the viscosity value is the most effective factor. The decrease in values of Walden product with increasing proportion of methanol and DMF in the order: 10%>30%>50% can be explained from the point of view, increasing the content of organic solvent leads to a bigger size of solvation shells around cation and this reduces its mobility as well as Walden product [42]. Many authors reported the change in Walden product with temperature, solvent composition and nature of electrolyte [43] (Figure 3).

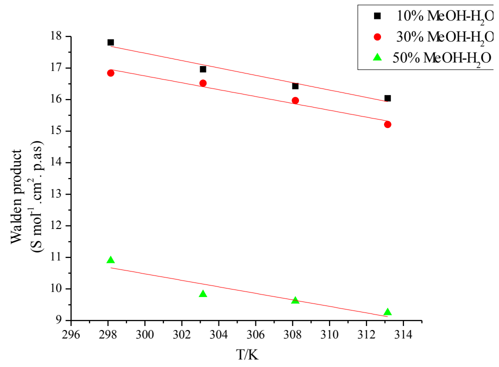

Figure 3: The plot of Walden product (Λ_0 η) vs. temperature (T/K) for vanadyl sulfate in the (methanol-H_2O) solvents.

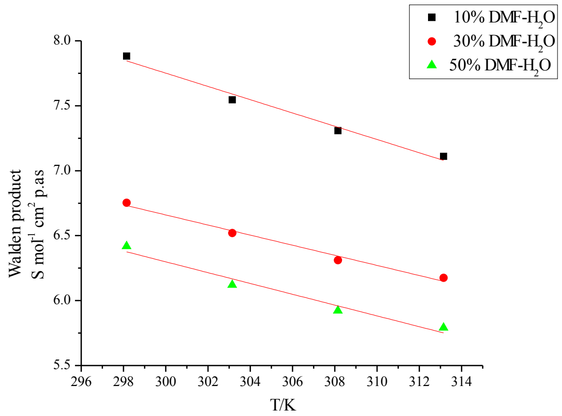

Figure 4: The plot of Walden product (Λ_0 η) vs. temperature (T/K) for vanadyl sulfate in the (DMF-H_2O) solvents.

Activation energy: Since the mobility of the ion has a great effect on the conductance, it is important to treat the rating process taking place with the change of temperature [6] based on Equation (14)

$$\Lambda_0 = A\ e^{-Ea./RT} \quad (14)$$

Where "E_a" is the activation energy of the transfer process, "A" is the frequency factor, and "R" is the gas constant. From the plot of (ln Λ_0) vs. (1/T), (as in Figure 2) the E_a values can be estimated by elevation of the fraction of organic solvent the activation energy E_a of transport process was found to increase and increase in the following direction: (Methanol-H_2O)<(DMF-H_2O). The observed order of Walden product was observed also for E_a which may be attributed to the solvents viscosity (Figure 4).

Thermodynamics of association: The standard Gibbs free energy of association ($\Delta G°_A$) was calculated by using Equation (15) for all salts under study in all solvent mixtures at all temperatures and its values were tabulated in Table 4.

$$\Delta G°_A = -\ RT\ \ln K_A \quad (15)$$

Where R is the gas constant and equal (8.314 $J.mol^{-1}.K^{-1}$). The values of the standard enthalpy ($\Delta H°_A$) and the standard entropy ($\Delta S°$ A) of association process were obtained from van't Hoff equation $\left(\frac{dlnk}{dt}\right)=\left(\frac{\Delta H^0_A}{RT^2}\right)$by plotting (log K_A) versus (1/T), where the slope is equal the value of ($-\Delta H_A/2.303R$) while the entropies of association ($\Delta S°A$) were calculated by the use of Gibbs–Helmohltz equation (Equation 15). $\Delta G°_A = \Delta H°_A - T\Delta S°_A$ (16).

The thermodynamic parameters of association values showed the effect of temperature from increasing in the negative values of the associating free energy ($\Delta G°_A$) as the temperature rise from 298.15 to 313.15K. It was found that the association processes in all systems are spontaneous processes and the associating free energy becomes more negative with increase in temperatures. This indicates that ion-pair association is favored with lowering of dielectric constant of the medium.

The positive value of ($\Delta H°_A$), indicates the ion association processes are endothermic in nature. A positive entropy values ($\Delta S°_A$) can be explained on the assumption that iceberg structure around the cation is broken when association takes place leading to an increase in the degree of disorderliness and the positive ($\Delta H°_A$) and ($\Delta S°_A$) values are in a good agreement with several theories in many solvents [44].

Solvent	**T/K**	**$\Delta H°_A$** (kJ.mol-1)	**$\Delta S°_A$** (J.mol-1)
10% ($MeOH-H_2O$)	298.15	24.02	145.23
	303.15		146.31
	308.15		146.98
	313.15		148.01
30% ($MeOH-H_2O$)	298.15	36.32	196.62
	303.15		197.18
	308.15		197.91
	313.15		198.01
50% ($MeOH-H_2O$)	298.15	46.90	213.94
	303.15		214.13
	308.15		214.99
	313.15		215.01
10% ($DMF-H_2O$)	298.15	23.64	136.44
	303.15		136.92
	308.15		137.84
	313.15		138.55
30% ($DMF-H_2O$)	298.15	37.07	184.69
	303.15		185.12
	308.15		185.91
	313.15		186.36
50% ($DMF-H_2O$)	298.15	33.16	168.21
	303.15		168.95
	308.15		169.46
	313.15		170.02

Table 4: The thermodynamic parameters of association ($\Delta H°_A$) Enthalpy change of association and ($\Delta S°_A$) which is the entropy of association for vanadyl sulfate in used mixed solvents.

Conclusions

Conductivity measurements for vanadyl sulfate in a binary mixed solvent with alcohol mass fraction of 10%, 30%, and 50% ($MeOH-H_2O$) and ($DMF-H_2O$) at different temperatures have been reported. The conductivity data have been analyzed using Fuoss-Shedlovsky equation.

The extent of ion-pairing in vanadyl sulfate solutions under study depends on the nature of the (ion-solvent) and (solvent-solvent) interaction taking place in the solution. Furthermore, it depends on the dielectric constant and the properties of different solvents. The association constant increases as the temperature increases and association constant is higher in ($DMF-H_2O$) than in ($MeOH-H_2O$) this due to formation of bulker ($DMF-H_2O$) aggregates which hinder movement of ions than in ($MeOH-H_2O$) and this lead to formation of ion pairs.

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