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Conductometric study of potassium chloride in ethylene glycol + water mixtures at different temperatures

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Molar conductivity of KCl solutions in ethylene glycol + water mixtures (w_{EG} = 0.10, 020, 0.30, and 0.40) was measured at T = (278.15, 288.15, 298.15, and 308.15) K. The values of limiting molar conductivity (Λ^o), association constant (K_A) and distance parameter (R) were calculated using the low concentration Chemical Model (lcCM). In this paper the values of Walden product ($\Lambda^o \cdot \eta$), as well as the standard thermodynamic quantities for the association reaction at T = 298.15 K were presented. Changes in these quantities as a function of changes in the composition of the mixed solvent have been discussed.

Keywords: potassium chloride, ethylene glycol + water mixtures, conductivity, thermodynamic quantities, Walden product, association distance.

1. INTRODUCTION

The mixed solvents are interesting for investigation of ion association reactions by conductometry because the addition of even small amounts of organic component to water changes significantly physical properties of the mixed solvents (for ethylene glycol (EG) + water (W) mixtures see Table 1.). EG is similar to some other aprotic solvents with regard to its relative permittivity (ε_r = 41.4 at 20°C) and molecular dipole moment (μ = 2.28 D) [1]. Physico-chemical properties of EG structure is a spatial net of hxdrogen bonds, which is very sensitive to the influence of temperature and electrolytes [2]. Respectively, EG is similar to W. Solvent W is also polar (μ = 1.87 D, ε_r = 80.37 at 20°C) [2]. Thermodynamic properties of (EG + W) mixtures (excess volumes, viscosity deviation, excess refraction, and surface excess) have been reported by Tsierkezos and Molinou [3]. The same autors [2] have measured transference numbers of $CuSO_4$ in (EG + W) mixtures at 20°C by potential difference (pd) method, the limiting molar (Λ^o) and ionic conductances (λ_+^o, λ_-^o) as well as the ion association constants (K_A) and viscosity. The potential difference measurements for KCl in (EG + W) mixtures at 278.15, 288.15, 298.15, and 308.15 K have been reported by Ma *et al*. [4].

In this work we report conductometric data for electrolyte KCl in (EG + W) mixtures with mass fraction, w_{EG} = 0.10, 0.20, 0.30, and 0.40 at 278.15, 288.15, 298.15, and 308.15 K. The Λ^o, the K_A, and Walden product ($\Lambda^o\ \eta$) were derived. Standard Gibbs energy (ΔG^o), enthalpy (ΔH^o) and entropy (ΔS^o) changes for the association reaction of K^+ and Cl^- were calculated from the temperature dependence of K_A. These measurements are unknown in the literature. It would be interesting to carry out the investigation for these systems with the pd measurements in the concentration cell with transference [5] as a function of EG mass fraction in the mixtures (EG + W) at different temperatures. Namely, the limiting molar conductivity of individual ions (λ_+^o, λ_-^o) in this case can be determined without any assumptions by combining limiting transference numbers of the ions (t_+^o, t_-^o) (our future study) with the limiting molar conductivity of electrolyte (Λ^o) (our present study) [5].

Table 1. Density, viscosity, and relative permittivity of ethylene glycol (EG) + water (W) mixtures at different temperatures[a]

T	278.15 K	288.15 K	298.15 K	308.15 K
w_{EG} = 0.10				
d / g cm^{-3}	1.01399[b]	1.01236[b]	1.00972[b]	1.00624[b]
$10^3\eta$ / Pa s	1.9047[c]	1.4875[c]	1.1487[c]	0.9212[c]
ε_r	82.98[d]	79.07[d]	75.23[d]	71.62[d]
w_{EG} = 0.20				
d / g cm^{-3}	1.02862[b]	1.02606[b]	1.02270[b]	1.01865[b]
$10^3\eta$ / Pa s	2.5732[c]	1.9554[c]	1.4816[c]	1.1754[c]
ε_r	79.60[d]	75.83[d]	72.30[d]	68.90[d]
w_{EG} = 0.30				
d / g cm^{-3}	1.04371[b]	1.04016[b]	1.03603[b]	1.03133[b]
$10^3\eta$ / Pa s	3.4498[c]	2.5857[c]	1.9213[c]	1.4972[c]
ε_r	76.92[d]	73.47[d]	70.09[d]	66.87[d]
w_{EG} = 0.40				
d / g cm^{-3}	1.05845[b]	1.05401[b]	1.04907[b]	1.04343[b]
$10^3\eta$ / Pa s	4.5236[c]	3.3704[c]	2.4625[c]	1.8828[c]
ε_r	73.24[d]	69.87[d]	66.47[d]	63.39[d]

[a] w_{EG} is the mass fraction of EG in the mixture.

[b] Measured in present work.

[c] Interpolated or extrapolated data from literature [3].

[d] Values from Ref. [4].

2. EXPERIMENTAL

All chemicals are obtained from Merck and are *p.a.* quality. Water was distilled twice (specific conductivity ~ 10^{-6} S cm^{-1}) and KCl was dried for several hours at 393.15 K. Ethylene glycol was used without further purification.

The conductance measurements were performed in the solutions containing 0.10, 0.20, 0.30, and 0.40 mass fraction of EG in W, usually for 12 molar concentration of KCl salt.

Conductivity was measured using automatic bridge (Precision Component Analyzer) type 6430B production of Waynr Kerr companies (Great Britain) with a very low uncertainty of 0.02%. For more details see Refs. [6-9]. The measuring cell made of pyrex glass was calibrated using KCl [10]. Barthel used a similar measuring cell in his works. The cell was shielded and placed in a calibration thermostat of Bu20F type (Lauda, Germany). The thermostat stability was estimated at 0.005 K. The temperature was controlled by the Amarell 3000TH AD thermostat. The DLK 25 type flow cooler (Lauda, Germany) was also used in the measurement. The conductivity was measured in the following frequency ranges: ν = 0.2, 0.5, 1.0, 1.5, 2.0, 3.0, 5.0, 10.0 and 20.0 kHz. It should be noted that all measured conductivity values, $\lambda = 1/R_\infty$, were the results of extrapolation of cell resistance, $R_\infty(\nu)$, to infinite frequency $R_\infty = lim_{\nu\to\infty}R(\nu)$ using the empirical function $R(\nu) = R_\infty + A/\nu$. The standard values for temperature and pressure are u(T) = 0.01 K, u(p) = 0.05 MPa, respectively. The expanded uncertainty was $U_c(\Lambda) = 0.0005\,\Lambda$ at a pressure p = 0.1 MPa.

The density (Table 1.) of (EG + W) mixtures was determined at 278.15, 288.15, 298.15, and 308.15 K using Anton Paar oscillating U-tube densimeter (model DMA 4500M) with precision of ± 1 × 10^{-5} g cm^{-3}, and accuracy ± 5 × 10^{-5} g cm^{-3}. The densimeter was calibrated (according to the manufacturer's recommendations) to air and distilled water (this water was distilled several times). All calibration was carried out at atmospheric pressure [5].

3. RESULTS AND DISCUSSION

The experimental values, *i.e.* conductances (Λ) in dependence of molalities (m), for investigated KCl in (EG + W) mixtures are compiled in Tables 2 a-d.

The conductivity data were analyzed using the Fuoss-Justice equation according to the low concentration Chemical Model (lcCM) [11]. This approach uses the following set of equations:

$$\Lambda = \left[\Lambda^{\mathrm{o}} - S(\alpha c)^{1/2} + E(\alpha c)\ln(\alpha c) + J(\alpha c) - J_{3/2}(\alpha c)^{3/2}\right] \tag{1}$$

$$K_{\mathrm{A}} = \frac{1-\alpha}{\alpha^2 c y_{\pm}^2} \tag{2}$$

$$\ln y_{\pm} = -\frac{A\alpha^{1/2}c^{1/2}}{1 + BR\alpha^{1/2}c^{1/2}} \tag{3}$$

The meaning of individual parameters in Equations 1-3 is discussed in our previous work [8].

Table 2a. Molar conductances (Λ) and corresponding molalities (m) for KCl in w_{EG} = 0.10 mass fraction of EG in the mixture (EG + W) at different temperatures at pressure p = 0.1 MPa

$w_{EG} = 0.10$					
nr.	$\frac{m \cdot 10^3}{mol \cdot kg^{-1}}$	$\frac{\Lambda}{S \cdot cm^2 \cdot mol^{-1}}$ 278.15 K	$\frac{\Lambda}{S \cdot cm^2 \cdot mol^{-1}}$ 288.15 K	$\frac{\Lambda}{S \cdot cm^2 \cdot mol^{-1}}$ 298.15 K	$\frac{\Lambda}{S \cdot cm^2 \cdot mol^{-1}}$ 308.15 K
1	0.12000	74.42	96.22	121.67	147.81
2	0.50998	73.78	95.58	121.03	147.17
3	1.09991	72.98	94.78	120.23	146.37
4	2.19964	72.12	93.92	119.37	145.51
5	4.29862	70.97	92.77	118.22	144.36
6	6.09723	70.08	91.88	117.33	143.47
7	7.29603	69.74	91.54	116.88	143.02
8	10.4918	68.69	90.49	115.91	142.05
9	11.4901	68.26	90.06	115.51	141.65
10	13.0872	67.68	89.48	114.93	141.07
11	14.1850	67.39	89.19	114.64	140.78
12	15.0830	67.04	88.84	114.29	140.43

Table 2b. Molar conductances (Λ) and corresponding molalities (m) for KCl in w_{EG} = 0.20 mass fraction of EG in the mixture (EG + W) at different temperatures at pressure p = 0.1 MPa

$w_{EG} = 0.20$					
nr.	$\frac{m \cdot 10^3}{mol \cdot kg^{-1}}$	$\frac{\Lambda}{S \cdot cm^2 \cdot mol^{-1}}$ 278.15 K	$\frac{\Lambda}{S \cdot cm^2 \cdot mol^{-1}}$ 288.15 K	$\frac{\Lambda}{S \cdot cm^2 \cdot mol^{-1}}$ 298.15 K	$\frac{\Lambda}{S \cdot cm^2 \cdot mol^{-1}}$ 308.15 K
1	0.10000	61.92	80.65	104.56	127.90
2	0.49998	61.28	80.01	103.92	127.26
3	0.99993	60.48	79.21	103.12	126.46
4	1.99970	59.62	78.35	102.26	125.60
5	3.99881	58.47	77.20	101.11	124.45
6	5.99732	57.58	76.31	100.22	123.56
7	6.99635	57.24	75.97	99.88	123.22
8	9.99255	56.19	74.92	98.83	122.17
9	10.9910	55.76	74.49	98.40	121.74
10	12.9874	55.18	73.91	97.82	121.16
11	13.9854	54.89	73.62	97.53	120.87
12	14.9832	54.54	73.27	97.18	120.52

The analytical form of the parameters S, E, J, and $J_{3/2}$ was presented previously [11]. The values of Λ^o for all the investigated systems in all measured temperatures were obtained using the well-known procedure given by Fuoss [12]. The calculations were carried out assuming that $R = q$ (q - Bjerrum distance [13]).

The values of Λ^o, K_A, and R for investigated systems were obtained using the well-know three-parametric fitting procedure given by Fuoss [12] and are collected in Table 3.

Figure 1 shows the temperature dependence of the limiting molar conductivity Λ^o of the particular systems. The limiting molar conductivity decreases as the mass fraction of EG increases at all temperatures. The increase of Λ^o with an increase of temperature is due to the decrease of solvent viscosity.

The dependence of the Walden product ($\Lambda_o\eta$) as a function of the mixed solvent composition can be provide information on ion-solvent interactions.

The data included in Table 3 shows that the changes of Walden product as a function of changes of the temperature in studied binary (EG + W) mixtures are very small. Barthel *et al.* [14] measured the conductivity of LiBr in mixtures of propylene carbonate and acetonitrile ($0 \leq w_{PC} \leq 1$) in the temperature range from -35 to 75 °C. The results of their investigations also show that Λ^o increases with an increase in temperature, which is the result of the decrease of solvent viscosity, leading to a Walden product almost independent of temperature [15]. At constant temperature, the Walden product generally little depends on the composition of the solvent. From the value of Walden's product, we can guess about the reduction of the effective size of the ions at the concentration changes. This must be the result of changes in their solvation shells. However, it is difficult to notice that the exchange of water and glycol molecules can lead to a reduction in the size of the solvation shells. These apparent contradictions are explained by the hypothesis of selective ion hydration. The addition of glycol to water does not change the hydration shells of ions, and thus their immediate surroundings do not change their composition while the viscosity increases. In this way ions gain mobility and the values of Walden product increase.

Figure 2. shows the change of K_A with the increase of temperature. Namely, K_A increases with the increasing of the temperature. The increase of ion association with the increase of EG content can be explained in terms of the mixture permittivity decrease. The values of K_A for KCl in (EG + W) mixtures indicate no significant ion-association. Lebed et al. [16] are reported conductivity data for LiCl, NaCl, KCl, RbCl, CsCl, Me_4NCl, Et_4NCl, Bu_4NCl, NaF, NaBr and $NaBPH_4$ in pure EG over the temperature range 5-175 °C. They got $K_A = 1.06$ dm^3 mol^{-1} at 298.15 K (relative permittivity is 40.29 and viscosity of the solvent is 16.91 mPa s). The analysis of the changes in the value of the association constant (K_A) for KCl is presented in our work. It should be remembered that the association increases with the increase in the content of the organic component in the mixed water-organic solvent. This is due to the fact that the relative permittivity (ε_r) of such mixture decreases as the organic component increases. The effect of this is that electrically neutral ionic pairs appear in the solution.

Bjerrum [13] has already made the first attempts to mathematically describe the issues related to the formation of ion pairs. According to him, the necessary condition for the formation of such ionic associates is the appearance of the ions in such distance that guarantees obtaining adequate energy of interaction. An analysis of the work of many researchers indicates that the relationship of the logarithm of the association constant of the studied ions to the inverse of electric permittivity should be linear. This is not always the case because the deriving equations on an association is constant, the environment in which the ions are found is considered to be continuous. The authors here do not take into account the structural properties of the solvent. It should be remembered that the formation of ion pairs is not only associated with the existence of Coulomb forces, but also associated with ion-dipole interactions, the possibility of forming covalent and hydrogen bonds, and the existence of dispersion forces. The association constant (K_A) values for KCl presented in our work are small and decrease with the increase of the organic component content in the tested mixtures.

As we mentioned earlier, it is related to the fact that the value of the relative permittivity of the tested mixed solvent decreases with the increase in the amount of organic component. Such K_A decreases are described by all ion association models. Identical relationships have been observed in previous works [17-20]. This nature of the changes may result from the spatial structure of the solvent and the effects associated with its incorporation into the spaces between the ions forming ionic pairs. It should also be noted that for large ions, much lower conductivity values are most often caused by their size, e.g. for BPh_4^- and Bu_4N^+ ions. Similar conclusions result from the analysis of changes in Walden's product. Values of the Walden product should depend only on the value of the so-called Stokes radius, whose values result from the effective ion rays [21]. Therefore, the analysis of changes in the $\Lambda_0\eta$ value as a function of the mixed solvent composition refers in consequence to the analysis of its changes as a function of the effective ion size.

The values of the Walden product in dependence on the solvent permittivity are shown in Figure 3. Differences in hydrodynamic radius (see Table 3.) are reflected in the Walden product.

Table 2c. Molar conductances (Λ) and corresponding molalities (m) for KCl in w_{EG} = 0.30 mass fraction of EG in the mixture (EG + W) at different temperatures at pressure p = 0.1 MPa

		w_{EG} = 0.30			
nr.	$\frac{m \cdot 10^3}{mol \cdot kg^{-1}}$	$\frac{\Lambda}{S \cdot cm^2 \cdot mol^{-1}}$	$\frac{\Lambda}{S \cdot cm^2 \cdot mol^{-1}}$	$\frac{\Lambda}{S \cdot cm^2 \cdot mol^{-1}}$	$\frac{\Lambda}{S \cdot cm^2 \cdot mol^{-1}}$
		278.15 K	288.15 K	298.15 K	308.15 K
1	0.11000	45.47	59.83	78.82	98.40
2	0.50498	44.83	59.19	78.18	97.76
3	1.10991	44.03	58.39	77.38	96.96
4	2.09967	43.17	57.53	76.52	96.10
5	4.14872	42.02	56.38	75.37	94.95
6	6.11721	41.13	55.49	74.48	94.06
7	7.27605	40.58	54.94	73.93	93.51
8	10.3919	39.61	53.97	72.96	92.54
9	11.3903	39.31	53.67	72.66	92.24
10	13.1870	38.73	53.09	72.08	91.66
11	14.0852	38.44	52.80	71.79	91.37
12	15.1828	38.09	52.45	71.44	91.02

Table 2d. Molar conductances (Λ) and corresponding molalities (m) for KCl in w_{EG} = 0.40 mass fraction of EG in the mixture (EG + W) at different temperatures at pressure p = 0.1 MPa

		w_{EG} = 0.40			
nr.	$\frac{m \cdot 10^3}{mol \cdot kg^{-1}}$	$\frac{\Lambda}{S \cdot cm^2 \cdot mol^{-1}}$	$\frac{\Lambda}{S \cdot cm^2 \cdot mol^{-1}}$	$\frac{\Lambda}{S \cdot cm^2 \cdot mol^{-1}}$	$\frac{\Lambda}{S \cdot cm^2 \cdot mol^{-1}}$

		278.15 K	288.15 K	298.15 K	308.15 K
1	0.11000	34.87	46.02	60.92	77.72
2	0.50498	34.23	45.38	60.28	77.08
3	1.11991	33.43	44.58	59.48	76.28
4	2.10967	32.57	43.72	58.62	75.42
5	4.13872	31.42	42.57	57.47	74.27
6	6.10722	30.53	41.68	56.58	73.38
7	7.26606	29.98	41.13	56.03	72.83
8	10.2921	29.01	40.16	55.06	71.86
9	11.2905	28.71	39.86	54.76	71.56
10	13.0872	28.13	39.28	54.18	70.98
11	14.0952	27.84	38.99	53.89	70.69
12	15.0830	27.49	38.64	53.54	70.34

Table 3. Limiting molar conductances (Λ^{o}), association constants (K_A), distance parameters (R), and Walden products ($\Lambda^{o}\eta$) for KCl in (EG + W) mixtures at different temperatures[a]

T	278.15 K	288.15 K	298.15 K	308.15 K
w_{EG} = 0.10				
Λ^{o} [S cm^2 mol^{-1}]	74.469 ±0.02	96.294 ±0.01	121.72 ±0.01	147.88 ±0.02
K_A [dm^3 mol^{-1}]	0.42 ±0.04	0.98 ±0.03	1.61 ±0.02	2.32 ±0.03
$\Lambda^{o}\eta$ [S cm^{2}mol^{-1}P]	0.146	0.143	0.140	0.136
R [10^{-8} cm]	2.1	2.9	3.8	4.5
w_{EG} = 0.20				
Λ^{o} [S cm^2 mol^{-1}]	61.952±0.01	80.693±0.02	104.59±0.02	127.96±0.01
K_A [dm^3 mol^{-1}]	1.50±0.04	2.91±0.02	4.02±0.04	4.60±0.03
$\Lambda^{o}\eta$ [S cm^{2}mol^{-1}P]	0.159	0.158	0.155	0.150
R [10^{-8} cm]	2.6	3.5	4.5	5.3
w_{EG} = 0.30				
Λ^{o} [S cm^2 mol^{-1}]	45.513±0.01	59.904±0.01	78.872±0.02	98.459±0.01
K_A [dm^3 mol^{-1}]	2.81±0.02	4.12±0.04	5.52±0.03	6.23±0.03
$\Lambda^{o}\eta$ [S cm^{2}mol^{-1}P]	0.157	0.155	0.152	0.147
R [10^{-8} cm]	3.3	4.6	5.4	6.3
w_{EG} = 0.40				
Λ^{o} [S cm^2 mol^{-1}]	34.915±0.01	46.043±0.02	60.994±0.01	77.792±0.01
K_A [dm^3 mol^{-1}]	4.22±0.02	5.11±0.04	6.52±0.04	7.21±0.02
$\Lambda^{o}\eta$ [S cm^{2}mol^{-1}P]	0.158	0.155	0.150	0.146
R [10^{-8} cm]	5.9	7.2	8.3	9.2

[a] w_{EG} is the mass fraction of EG in the mixture.

The temperature dependence of the association constant was used to calculate the free energy of Gibbs (ΔG^{o}).

$$\Delta G^{o}(T) = - R\, T \ln K_A(T) \tag{4}$$

$\Delta G^{o}(T)$ can also be expressed by the polynomial equation:

$$\Delta G^{o}(T) = A + B\,T + C\,T^{2} \tag{5}$$

The entropy and enthalpy of ion association are defined as

$$\Delta S^{o}(T) = -\left(\delta \Delta G^{o}/\delta T\right)_{p} = -B - 2\,C\,T\,, \tag{6}$$

$$\Delta H^{o} = \Delta G^{o} + T\,\Delta S^{o} \tag{7}$$

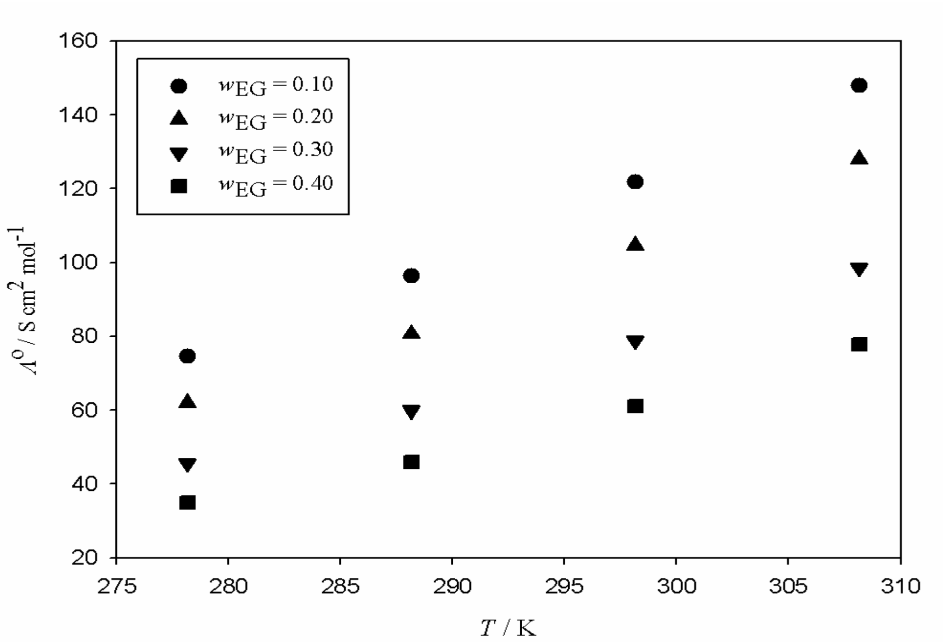

Figure 1. Temperature dependence of Λ^{o} (KCl) in 0.10, 0.20, 0.30, and 0.40 mass fraction of EG in the mixture (w).

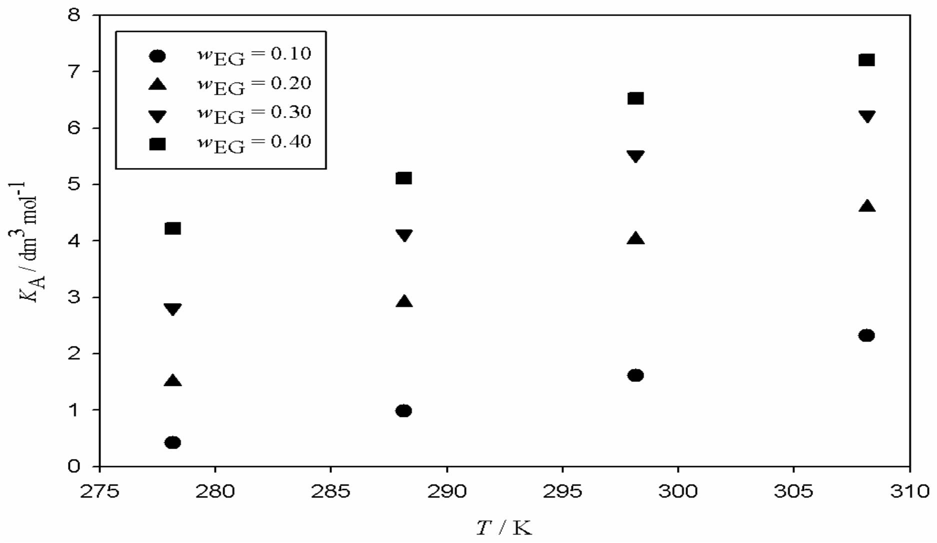

Figure 2. Temperature dependence of the association constant of K^{+} and Cl^{-} ions in different mixtures of (EG + W).

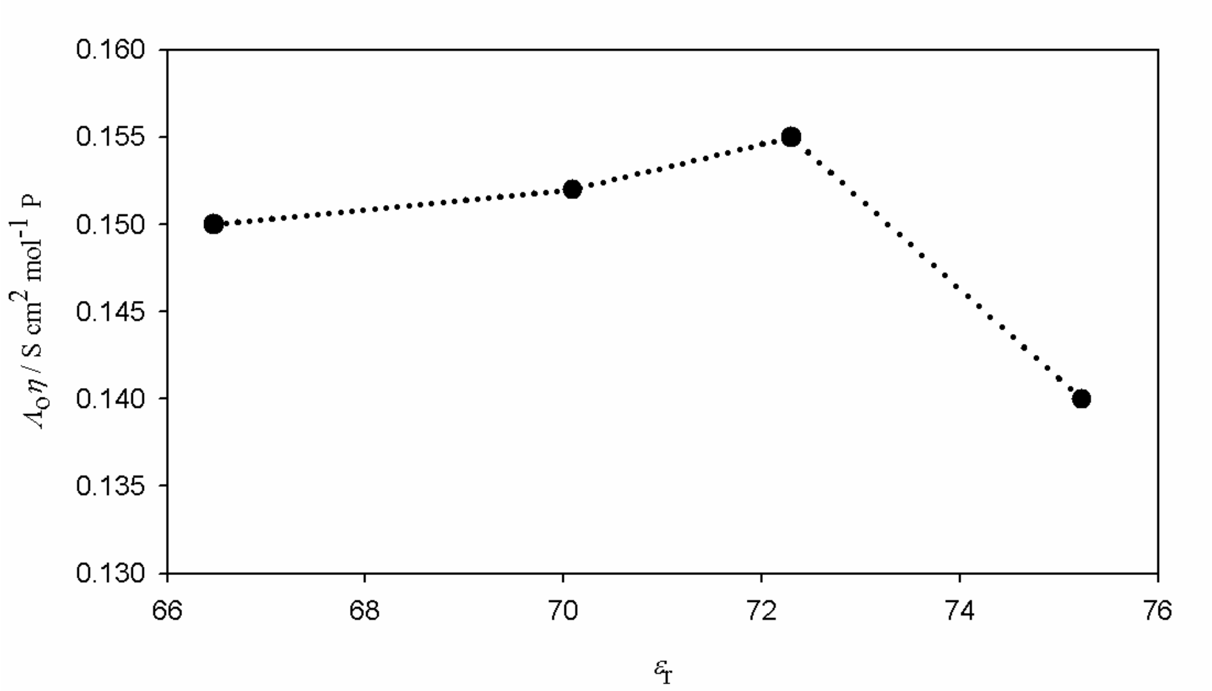

Figure 3. The plot of the Walden product ($\Lambda^o\eta$) as a function of the relative permittivity (ε_r) of (EG + W) mixtures at T = 298.15 K.

The thermodynamic functions described above (ΔG^o, ΔS^o, ΔH^o) were measured at 298.15 K and presented in Table 4.

Table 4. Standard thermodynamic quantities for the ion-association reaction in (EG + W) mixtures at 298.15 K

w_{EG}	ΔH^o / J mol^{-1}	ΔG^o / J mol^{-1}	ΔS^o / J K^{-1} mol^{-1}
0.10	32405	-1181	112.6
0.20	22302	-3449	86.4
0.30	14717	-4235	63.6
0.40	9576	-4647	48.2

Increasing the temperature to increasingly negative ΔG^o values causes a shift in the create ion pair equilibrium, which is the reason for the peripheral reduction of ion solvation by increasing the concentration. The evaluated values of thermodynamic functions af association suggest the spontaneity of the association process. The values of ΔH^o are positive and suggest that the ion-pairing process is endothermic. Because the Gibbs-free energy is negative, enthalpic effects seem to dominate over the entropic effects, in (EG + W) in the tested concentration range.

This suggests that as the concentration increases, the intermolecular interactions weaken and the number of intermolecular associations decreases.

4. CONCLUSIONS

In this work, the conductivity of KCl in binary mixtures of ethylene glycol + water was determined at various temperatures, at atmospheric pressure in the composition range of 0.10, 0.20, 0.30 , and 0.40 mass fraction of EG. Based on the experimental conductivity values, calculated limiting molar conductances (Λ^{o}), association constants (K_A), distance parameters (R), and Walden products ($\Lambda^{o}\eta$) for KCl in (EG + W) mixtures, and calculated standard thermodynamic quantities for the association reaction of K+ and Cl- ions in investigated systems, the folowing conclusions can be drawn:

- The molar conductivity decreases when the amount of ethylene glycol increases, which can be seen at all temperatures. However, the increase in the Λ^{o} value is caused by the decrease in the solvent viscosity.

- At constant temperature, the Walden product ($\Lambda^{o}\eta$) generally little depends on the composition of the solvent.

- Values of K_A increases with increasing temperature. The increase of ion association with the increase of EG content can be explained in terms of the mixture permittivity decrease. The values of K_A for KCl in (EG + W) mixtures indicate no significant ion-association.

- The evaluated values of thermodynamic functions of association suggest the spontaneity of the association process and endothermic character, respectively. Because Gibbs-free energy is negative, enthalpic effect seems to dominate over the entropic effects, and thus, the ion pair formation of KCl in (EG + W) mixtures is spontaneous.

- The research will be extended to same systems in order to get information about limiting tarnsference numbers of KCl and limiting molar conductivity of K^+ and Cl^- ions.

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