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# Some Transport Properties of Potassium Ferri/Ferro-Cyanide Solutions in a Wide Range of Schmidt Numbers

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**ABSTRACT:** Present work reports the electrochemical diffusion coefficient (EDC) values of ferri-cyanide ions in an electrolytic system frequently used in mass transfer measurements. The measurement methods were the combination of the electrochemical limiting current technique and the rotating disk system. The EDCs of ferri-cyanide ions in the aqueous electrolytic solutions supported with $K_2CO_3$ were measured in a temperature range of (288.15 to 308.15) K. Then, the electrolytic solutions in a wide range of Schmidt numbers (1060−145 000) were prepared by adding glycerol into the electrolytic solution supported with $K_2CO_3$. The EDCs of ferri-cyanide in these electrolytic solutions were measured. Furthermore, the density and the viscosity of all of the prepared solutions were also determined.

## INTRODUCTION

In mass transfer studies, either by molecular diffusion or convection, having the value of the diffusion coefficient is of vital importance for the prediction of concentration gradient, the calculation of mass transfer rates, and the development of mass transfer correlation equations. This coefficient is mainly a function of temperature and composition for a liquid mixture and of temperature and pressure for a gas mixture.

The electrochemical limiting diffusion current technique (ELDCT) has been widely used in convective, both natural and forced, mass transfer studies. With this technique, not only local and average mass transfer measurements can be performed but also measurements of fluid velocity, shear stress, and turbulence fluctuation can be carried out in a rather easy and fast manner.[1−3] The results obtained from mass transfer measurements can be converted to heat transfer data by analogy provided that the necessary conditions are supplied. To be able to develop the dimensionless mass transfer equations from the measurements using ELDCT in different geometries and for different flow conditions, the EDC values must be available for use and have been measured beforehand. The theory and some examples for application of ELDCT to different geometries and different flow conditions such as laminar, turbulent, forced, or natural flow for the mass transfer and shear stress measurements were reported in detail by Selman and Tobias[4] and Newman.[5]

For some liquid mixtures, diffusion coefficients can be estimated by some empirical formulas. However, since the kinetic theory of liquids is not as developed as for gases, no theoretical equation for liquid mixtures has been developed to calculate the diffusion coefficient by using the physical and chemical properties of the mixture, although a number of empirical equations have been derived for this purpose.[6−8] Therefore it is important to produce diffusion coefficient data for liquid mixtures.

The measurements of the electrochemical diffusion coefficients (EDC) have been performed for the ferri-cyanide ions in the electrolyte consisting of potassium ferri-cyanide as cathodic reactant, potassium ferro-cyanide as anodic reactant, and an excess supporting electrolyte, which is the most commonly used electrolytic system. Sodium hydroxide and potassium hydroxide are the most commonly used as supporting electrolytes. Therefore the physical properties of the ferri-ferrocyanide solutions with these supporting electrolytes have been reported by several researchers for different temperature and concentration.[9−14]

The EDC measurements have also been performed for the same electrolytic solutions but containing potassium chloride solution as the supporting electrolyte.[15,16] However, since these supporting electrolytes are corrosive and are pollutants, some other supporting electrolytes such as potassium carbonate,[17] potassium sulfate,[18−21] and sodium carbonate[22] have been used recently.

As there are no data on the diffusion of ferri-cyanide ion as a function of Schmidt number and as most of the other data reported in the literature utilize corrosive supporting electrolytes such as NaOH or KOH, this work aims to provide EDC data over a wide range of Schmidt numbers with with a benign supporting electrolyte of $K_2CO_3$ in a temperature range of (288.15 to 308.15) K.

## EXPERIMENTAL DETAILS

The limiting diffusion current technique is based on diffusion-controlled reaction at electrode (nickel, platinum, or stainless steel) surface. In general, the electrolyte used in the mass transfer measurement consists of potassium ferri-cyanide as cathodic reactant, potassium ferro-cyanide as anodic reactant, and an excess

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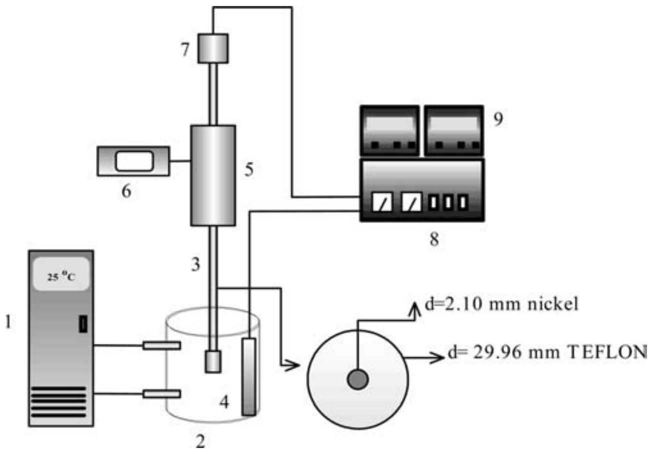

**Figure 1.** Experimental apparatus: 1. circulator for constant temperature; 2. jacketed glass vessel for electrolytic cell; 3. rotating disk electrode (cathode); 4. Ni anode; 5. digitally controlled rotary agitator; 6. rotation speed controller; 7. mercury reservoir for electric contact to rotating cathode; 8. DC power supply; 9. multimeters for measuring potential and current.

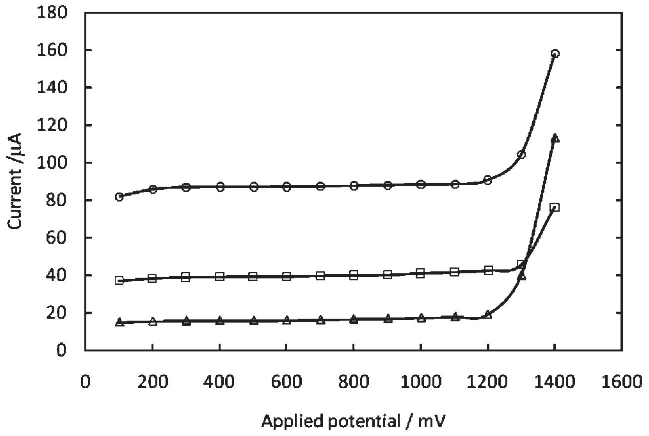

**Figure 2.** Current versus potential curves for solutions at 1000 rpm; ○, solution without glycerol; □, solution with 30% glycerol; Δ, solution with 50% glycerol.

supporting electrolyte. The following reactions take place at cathode and anode, respectively.

$$\mathrm{Fe(CN)_6}^{3-} + \mathrm{e} \rightarrow \mathrm{Fe(CN)_6}^{4-}$$

$$\mathrm{Fe(CN)_6}^{4-} \rightarrow \mathrm{Fe(CN)_6}^{3-} + \mathrm{e} \qquad (1)$$

In limiting current studies, generally the cathodic reduction of ferri-cyanide ion is employed due to better current—voltage behavior. Therefore, to ensure a cathodic controlled reaction, the concentration of ferro-cyanide is taken to be higher than that of ferri-cyanide and the anode larger than the cathode. Thus, the concentration ratio of ferro-cyanide to ferri-cyanide was chosen as 4:1, the area of the anode more than 40 times larger than that of the cathode. The solutions were prepared using A grade chemicals (Merck quality).

To determine the specific gravity of the prepared solutions, a set of hydrometers was employed, and their viscosities were determined using a Brookfield viscometer. The viscosity measurement device automatically gives the viscosity on a digital screen. Before measurement of the viscosity, the accuracy of the reading of the device was checked by viscosity measurements of water.

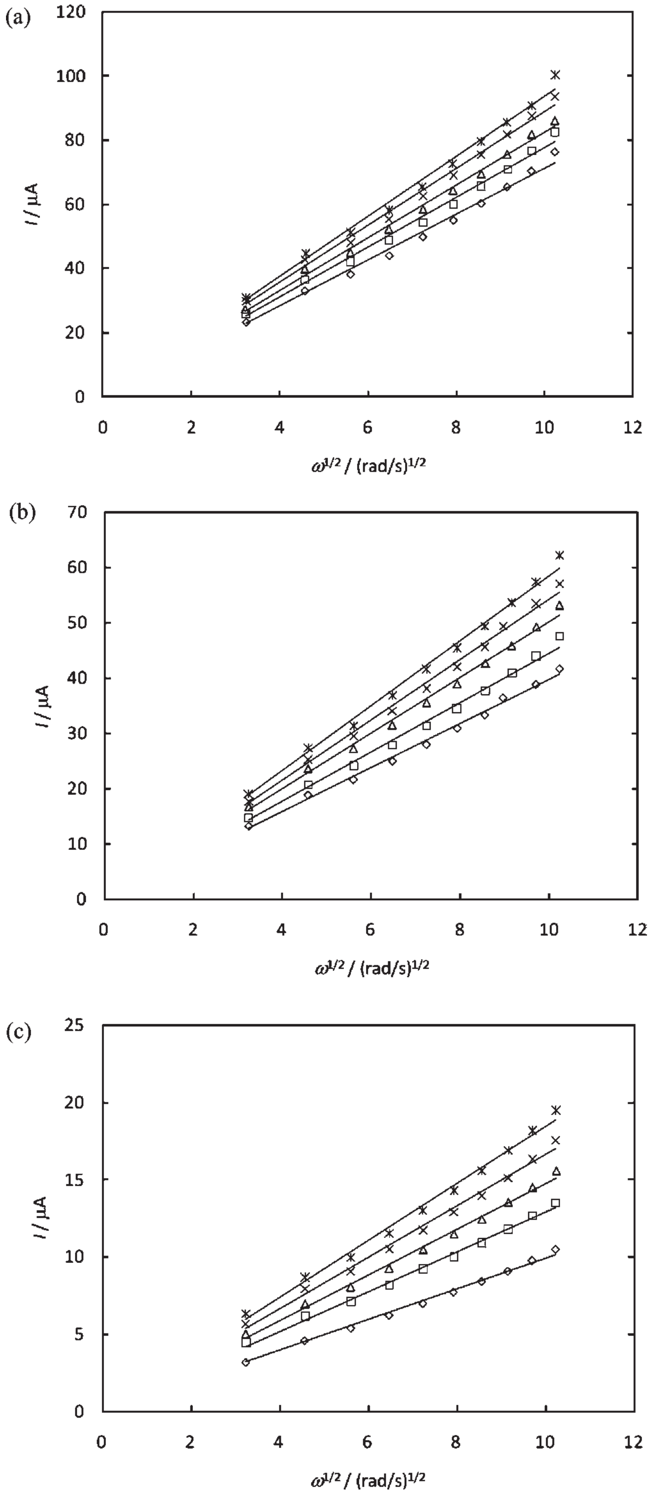

**Figure 3.** Plots of limiting current versus square root of rotation speed: (a) solution without glycerol; (b) solution with 20% glycerol; (c) solution with 50% glycerol. ◇, 15 °C; □, 20 °C; Δ, 25 °C; ×, 30 °C; *, 35 °C.

The EDC measurements of the ferri-cyanide ion were performed using rotating disk method. Experimental set up used in the diffusivity measurements consists of a mechanical mixer with speed controller, a rotating disk, a jacketed solution cell, a DC power supply, and two digital multimeters. A disk with a nominal diameter of 2.0 mm was used as rotating cathode and a stationary plate placed in the cell as anode, both made of 99.98% nickel. The actual diameter of the rotating disk was measured with NIKON MM 400 L video measuring microscope. The diameter of Teflon into which the disk electrode was set was about 3.0 cm. The simple schematic drawing of the experimental set up is shown in Figure 1. A variable speed mechanical mixer with a digital speed controller was employed to rotate the cathode disk. The rotating speed was changed in the range of 100 to 1000 rpm. The contact between rotating disk and electrical circuit was provided by a small mercury reservoir. The electrical circuit consisted of two digital multimeters for the measurement of current and voltage, and a DC for power supply. The temperature of the electrolyte was kept constant at a certain value within ± 0.10 K using a circulating water bath. All precautions and the pretreatments necessary for a reliable reading of the limiting current were performed as suggested by Szanto et al.[23] and Berger and Ziai.[24]

**Table 1. Statistical Values for Data in Figure 3 ($y = ax$)**

| Figure 3 | $T$/°C | $a$ | $R^2$ | $SE^*$ | $SEE^*$ |
|---|---|---|---|---|---|
| (a) | 15 | 7.1296 | 0.9894 | 0.0738 | 1.7704 |
| | 20 | 7.7668 | 0.9921 | 0.0678 | 1.6265 |
| | 25 | 8.2621 | 0.9945 | 0.0591 | 1.4177 |
| | 30 | 8.8983 | 0.9929 | 0.0724 | 1.7388 |
| | 35 | 9.3619 | 0.9911 | 0.0862 | 2.0685 |
| (b) | 15 | 3.9852 | 0.9938 | 0.0300 | 0.7189 |
| | 20 | 4.4600 | 0.9924 | 0.0383 | 0.9206 |
| | 25 | 5.0195 | 0.9940 | 0.0379 | 0.9105 |
| | 30 | 5.4244 | 0.9940 | 0.0412 | 0.9849 |
| | 35 | 5.8568 | 0.9936 | 0.0460 | 1.1049 |
| (c) | 15 | 0.9952 | 0.9947 | 0.0071 | 0.1710 |
| | 20 | 1.2926 | 0.9949 | 0.0087 | 0.2095 |
| | 25 | 1.4775 | 0.9943 | 0.0107 | 0.2576 |
| | 30 | 1.6629 | 0.9932 | 0.0132 | 0.3159 |
| | 35 | 1.8447 | 0.9927 | 0.0153 | 0.3670 |

The essential transport equation for the electrochemical systems can be expressed as[25]

$$N_i = -z_i u_i F c_i \nabla \Phi - D_i \nabla c_i + V c_i \qquad (2)$$

where the three terms on the right side represent the contributions of ionic migration due to electrical potential, molecular diffusion due to concentration difference, and bulk movement due to fluid movement, respectively. For the limiting current conditions having supporting electrolyte, the first and third terms are omitted. The simultaneous solution of the well-defined hydrodynamic equation and the mass transfer equation for the rotating disk electrode at the limiting current conditions is given by the Levich equation as follows:[25,26]

$$I = 0.6205 F n A \nu^{-1/6} D^{2/3} w^{1/2} C \qquad (3)$$

where $I$ is the current, $F$ Faraday's constant, $A$ the area of the cathode, $n$ the number of transferred electron, $\nu$ the kinematic viscosity of the electrolyte, $C$ concentration of the ferri-cyanide, $D$ the EDC of the ferri-cyanide, and $w$ the rotating speed. According to eq 3, when the current is plotted versus $w^{1/2}$ the slope of the curve is to be equal to $0.6205 F n A \nu^{-1/6} D^{2/3} C$, and then the EDC can be calculated from this expression.

The composition of the solutions is 5 mol·m$^{-3}$ ferri-cyanide, 20 mol·m$^{-3}$ ferro-cyanide, and 500 mol·m$^{-3}$ $K_2CO_3$. For obtaining Schmidt numbers in a wide range, glycerol was added into the electrolytic solution. For this purpose, (10 to 50) % in weight glycerol containing solutions were prepared. To observe the effect of temperature, the measurements were taken in the temperature range of (288.15 to 308.15) K. The rotating speed of RDE was between (100 to 1000) rpm.

An uncertainty analysis was performed using the method described by Holman.[27] The uncertainty of a calculated parameter

**Table 2. Measured Properties for Electrolytic Solutions with no Glycerol and Comparison with Literature**

| electrolyte | $T$, K | $\rho$, kg·m$^{-3}$ | $\nu \times 10^6$, m$^2$·s$^{-1}$ | $D \times 10^{10}$, m$^2$·s$^{-1}$ | $Sc$ | ref |
|---|---|---|---|---|---|---|
| 0.5 M $K_2CO_3$<br>5 mol·m$^{-3}$ $K_3Fe(CN)_6$<br>20 mol·m$^{-3}$ $K_4Fe(CN)_6$ | 288.15 | 1066.0 | 1.303 | 6.13 | 2130 | present work |
| | 293.15 | 1064.0 | 1.189 | 6.81 | 1745 | |
| | 298.15 | 1062.5 | 1.073 | 7.28 | 1475 | |
| | 303.15 | 1060.5 | 0.981 | 7.98 | 1232 | |
| | 308.15 | 1058.5 | 0.897 | 8.44 | 1069 | |
| 0.3 M $K_2SO_4$<br>5 mol·m$^{-3}$ $K_3Fe(CN)_6$<br>5 mol·m$^{-3}$ $K_4Fe(CN)_6$ | | | | 7.35 | 1360 | Barbeu et al.[19] |
| 0.04 kg·L$^{-1}$ $K_2SO_4$<br>25 mol·m$^{-3}$ $K_3Fe(CN)_6$<br>25 mol·m$^{-3}$ $K_4Fe(CN)_6$ | 295.15 | 1008 | 0.99 | 6.81 (±0.09) | 1454 | Wein et al.[18] |
| 0.5 M NaOH<br>20 mol·m$^{-3}$ $K_3Fe(CN)_6$<br>20 mol·m$^{-3}$ $K_4Fe(CN)_6$ | 298.15 | 1027 | 0.935 | 7.10 | 1317 | Saraç et al.[9] |
| 1 M $Na_2CO_3$<br>1 mol·m$^{-3}$ $K_3Fe(CN)_6$<br>10 mol·m$^{-3}$ $K_4Fe(CN)_6$ | 312.15 | 1098.5 | 0.956 | 6.4 | 1494 | Griffiths and Leon[22] |
| 0.3 M $K_2SO_4$<br>25 mol·m$^{-3}$ $K_3Fe(CN)_6$<br>25 mol·m$^{-3}$ $K_4Fe(CN)_6$ | 293.15 | | 1.00 | 8.33 | 1200 | Dib et al.[21] |

based on the measured variables is given as

$$\omega_{\mathrm{R}} = \sqrt{\sum_{i=1}^{n}\left(\frac{\partial R}{\partial x_i}\omega_{xi}\right)^2} \tag{4}$$

where $R$ is the calculated parameter, $\omega_{\mathrm{R}}$ the uncertainty in the calculated parameter, $x_i$ the independent variables used to calculate the result, and $\omega_{xi}$ the uncertainty in the independent variables. The uncertainties in the measured parameters were calculated to be ± 1.0 % for the density, $\rho$ ($\mathrm{kg \cdot m^{-3}}$), and ± 1.0 % for viscosity, $\mu$ (Pa·s). The EDCs were calculated from the slope of the straight lines obtained by plotting the limiting current versus the square root of the rotation speed, $w^{1/2}$, according to eq 3. The uncertainty arising from slope of curves was taken as (slope ± SE), which changes in the range of 0.7 to 1.0 %. The uncertainty in the EDC and *Sc* number were calculated to be ± 2.10 and 2.53 %, respectively. Every measurement was taken thrice, and the average of these three measurements was used for the calculations. The maximum and minimum deviation from the average was found to be + 5.81 % and − 3.97 %, respectively.

**Table 3. Measured Properties for Electrolytic Solutions with Glycerol for Different Glycerol Percentages and Temperatures with $K_2CO_3$ as Supporting Electrolyte**

| glycerol, wt % | $T$, K | $\rho$, $\mathrm{kg \cdot m^{-3}}$ | $\nu \times 10^6$, $\mathrm{m^2 \cdot s^{-1}}$ | $D \times 10^{10}$, $\mathrm{m^2 \cdot s^{-1}}$ | *Sc* |
|---|---|---|---|---|---|
| no glycerol | 288.15 | 1066.0 | 1.304 | 6.128 | 2128 |
| | 293.15 | 1064.0 | 1.189 | 6.809 | 1746 |
| | 298.15 | 1062.5 | 1.073 | 7.281 | 1474 |
| | 303.15 | 1060.5 | 0.981 | 7.977 | 1232 |
| | 308.15 | 1058.5 | 0.897 | 8.439 | 1069 |
| 10 % | 288.15 | 1088.5 | 1.695 | 5.026 | 3372 |
| | 293.15 | 1086.5 | 1.500 | 5.580 | 2688 |
| | 298.15 | 1085.5 | 1.368 | 5.733 | 2386 |
| | 303.15 | 1083.5 | 1.232 | 6.501 | 1895 |
| | 308.15 | 1082.0 | 1.123 | 7.181 | 1564 |
| 20 % | 288.15 | 1110.5 | 2.332 | 3.110 | 7499 |
| | 293.15 | 1108.5 | 2.039 | 3.560 | 5726 |
| | 298.15 | 1108.0 | 1.796 | 4.118 | 4361 |
| | 303.15 | 1105.5 | 1.615 | 4.505 | 3584 |
| | 308.15 | 1103.0 | 1.441 | 4.913 | 2934 |
| 30 % | 288.15 | 1138.0 | 3.335 | 2.348 | 14201 |
| | 293.15 | 1132.5 | 2.924 | 2.792 | 10471 |
| | 298.15 | 1130.5 | 2.566 | 3.173 | 8087 |
| | 303.15 | 1128.0 | 2.234 | 3.513 | 6359 |
| | 308.15 | 1126.0 | 1.998 | 3.545 | 5637 |
| 40 % | 288.15 | 1158.5 | 5.218 | 1.097 | 47569 |
| | 293.15 | 1156.5 | 4.405 | 1.356 | 32493 |
| | 298.15 | 1155.0 | 3.762 | 1.619 | 23239 |
| | 303.15 | 1153.5 | 3.242 | 1.817 | 17843 |
| | 308.15 | 1151.5 | 2.857 | 2.014 | 14188 |
| 50 % | 288.15 | 1186.0 | 9.022 | 0.621 | 145355 |
| | 293.15 | 1184.5 | 7.543 | 0.878 | 85860 |
| | 298.15 | 1181.0 | 6.308 | 1.027 | 61441 |
| | 303.15 | 1179.0 | 5.280 | 1.173 | 45029 |
| | 308.15 | 1176.0 | 4.477 | 1.315 | 34054 |

## ■ RESULTS AND DISCUSSIONS

Before the limiting current measurements, the current−voltage measurements were performed to see whether a good typical plateau is obtained. The current−potential curves for the electrolytic solutions are given in Figure 2. As seen from the figure, the limiting current plateau is very good for the solution with and without glycerol. From this figure, 0.65 V was chosen as working potential difference.

The plots of the limiting current versus the square root of the rotation speed for the solutions are presented in Figure 3 as an example for the solutions without glycerol, with (20 and 50) % (in weight) glycerol. The lines representing the measurements for the solutions especially at higher temperatures seem to deviate slightly from linear behavior, when the rotation speed increased. In the Levich approximation, it is accepted that there is no contribution of bulk movement to the mass transfer in the film layer. The possibility of producing some bulk effect in the boundary layer just next to the rotating solid surface can increase with increasing rotation speed due to some imperfectness on the surface. Table 1 shows statistical values for the best curve fits in Figure3. It is seen that the standard errors (*SE*) are very low and the linear determination coefficients ($R^2$) and consequently, the linear correlations coefficients ($R$) are very high. These values confirm that this deviation from linearity is statistically acceptable. Therefore, the variation of the limiting current with the square root of the rotation speed can be taken as linear.

The EDC values calculated from the slope of the lines in these plots. For comparison, some measured property values of the present work and the values from the literature are given in Table 2. This table shows that the measurements of the present work are in good agreement with the values given in the literature. All of the EDC values are given in Table 3 together with the density, kinematic viscosity and Schmidt number values of the solutions. As seen from this table, the EDCs enhance with decreasing glycerol percentage and rising temperature. The viscosity and density values for water-glycerol with electrolytes were always measured higher than those without electrolytes.

Wein et al.[18] worked with the solution having the composition of 25 $\mathrm{mol \cdot m^{-3}}$ ferri-cyanide, 25 $\mathrm{mol \cdot m^{-3}}$ ferro-cyanide, and 0.04 $\mathrm{kg \cdot L^{-1}}$ $K_2SO_4$, containing glycerol in the range of

**Table 4. Electrochemical Diffusion Coefficients for Electrolytes with Glycerol from Literature and Comparison with Present Work**

| | | glycerol, wt % | | | |
|---|---|---|---|---|---|
| | | 0 | 20 | 30 | 40 |
| ECD, $\mathrm{m^2 \cdot s^{-1}}$ | Wein et al.[18] (295.15 K)<br>($K_2SO_4$ as supporting electrolyte) | $6.81 \times 10^{-10}$ | $3.48 \times 10^{-10}$ | $2.28 \times 10^{-10}$ | $1.42 \times 10^{-10}$ |
| | present work (293.15 K)<br>($K_2CO_3$ as supporting electrolyte) | $6.81 \times 10^{-10}$ | $3.56 \times 10^{-10}$ | $2.39 \times 10^{-10}$ | $1.36 \times 10^{-10}$ |

Table 5. Values of $D\mu/T$

| | $D\mu/T \times 10^{15}$, $\mathrm{N \cdot K^{-1}}$ | | | | | |
|---|---|---|---|---|---|---|
| | glycerol, wt % | | | | | |
| temperature, K | 0 | 10 | 20 | 30 | 40 | 50 |
| 288.15 | 2.96 | 3.22 | 2.80 | 3.09 | 2.30 | 2.31 |
| 293.15 | 2.94 | 3.10 | 2.75 | 3.15 | 2.36 | 2.68 |
| 298.15 | 2.79 | 2.86 | 2.75 | 3.09 | 2.36 | 2.57 |
| 303.15 | 2.73 | 2.86 | 2.65 | 2.92 | 2.24 | 2.41 |
| 308.15 | 2.60 | 2.83 | 2.54 | 2.59 | 2.15 | 2.25 |
| Av. value | 2.80 | 2.98 | 2.70 | 2.97 | 2.28 | 2.44 |

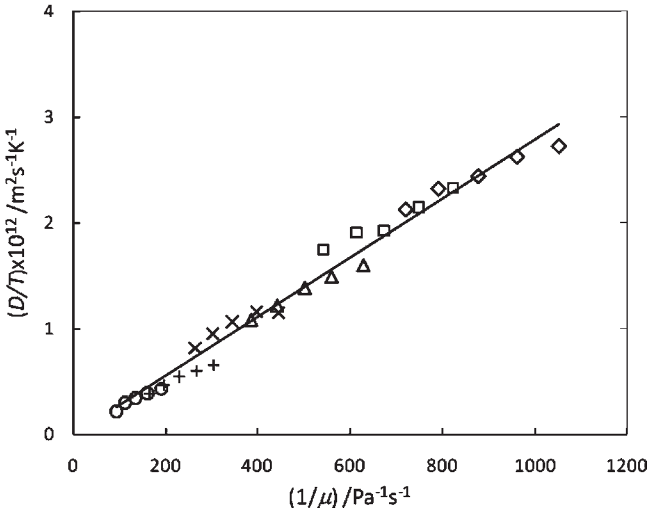

Figure 4. Plot of $D/T$ versus $1/\mu$ for electrolytic solutions with different glycerol content (weight %): ◊, with no glycerol; □, 10 %; Δ, 20 %; ×, 30 %; *, 40 %; ○, 50 %.

(0 to 60) % (in weight). His measurements were only for the temperature 295.15 °C; for this reason his results were compared with the results of the present work taken at 293.15 °C in Table 4. As seen from this table, there is a good agreement between the results.

As a traditional relation, the viscosity and diffusivity of liquid are related to each other in the form of the Stokes–Einstein parameter $(D\mu/T)$.[28] The results of this ratio for the present work are given in Table 5. As seen from the table, even though this ratio changes little for a certain solution with a given glycerol percentage, in general, showing behavior of decreasing with increasing temperature; this value can be accepted as constant and an average value can be taken for a given solution. This situation is confirmed by the graph of $D/T$ versus $1/\mu$ as seen in Figure 4. The figure shows that the values correlate well on a straight line having the equation of

$$D/T = 2.790 \times 10^{-15}/\mu \tag{5}$$

with a linear determination coefficient of $R^2 = 0.9807$ and standard error of estimation of $1.0917 \times 10^{-13}$.

The EDC can be related to the temperature with a relation in the form of the Arrhenius equation as $D = A_0 e^{-(E/RT)}$.[28] Here, $E$ resembles the energy barrier for an ion to diffuse through the solution. This energy can be obtained by drawing a plot of ln $D$ versus $1/T$, as shown in Figure 5. This energy can be calculated from the slope of the obtained line. The values of this energy, which can also be called activation energy, are summarized in Table 6. As seen from this table, the minimum energy amount necessary for an ion to diffuse through the solution increases with increasing glycerol content of the electrolytic solution.

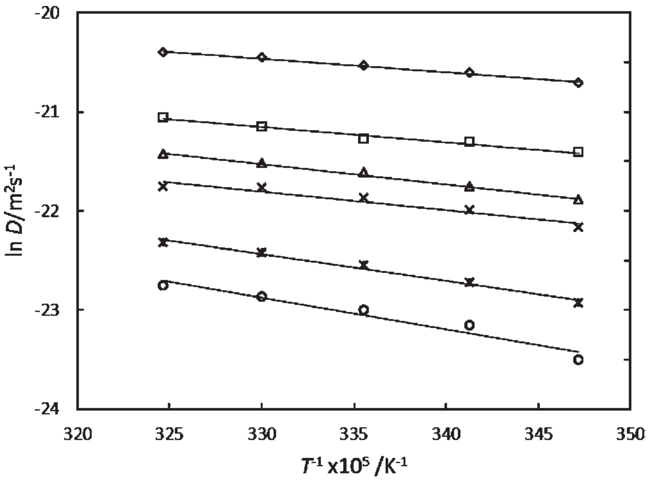

Figure 5. Plot of ln $D$ versus $1/T$ for electrolytic solutions with different glycerol content (weight %): ◊: with no glycerol; □, 10 %; Δ, 20 %; ×, 30 %; *, 40 %; ○, 50 %.

Table 6. Kinetic Constants for Electrochemical Diffusion Coefficients

| glycerol, wt % | $A_0 \times 10^7$, $\mathrm{m^2 \cdot s^{-1}}$ | $E$, $\mathrm{kJ \cdot mol^{-1}}$ |
|---|---|---|
| 0 | 79.180 | 11.614 |
| 10 | 1.033 | 12.572 |
| 20 | 3.757 | 16.993 |
| 30 | 1.450 | 15.365 |
| 40 | 0.122 | 22.204 |
| 50 | 0.399 | 26.513 |

## ■ CONCLUSIONS

In light of the findings of this work, in which the measurements of the EDCs, density, and viscosity were performed for an electrolyte solution with Schmidt numbers in the range of 1060 to 145 000 in a temperature range of (288.15 to 308.15) °C, the following conclusion can be made: An increase in the glycerol percentage resulted in the reduction of the EDC due to increasing viscosity. The values of $D\mu/T$ show a tendency to slightly decrease with increasing temperature for a given solution. However, it can be taken as a constant value. This work provides some useful transport properties for the use of mass transfer studies in a wide range of Schmidt numbers.

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