Casteel−Amis Equation: Its Extension from Univariate to Multivariate and Its Use as a Two-Parameter Function

Michael S. Ding*

Army Research Laboratory, 2800 Powder Mill Road, Adelphi, Maryland 20783

The report describes the background of the empirical Casteel−Amis equation and its application to fitting experimental data of conductivity as a function of salt content in electrolytes. It further summarizes the attempts since its publication to extend the equation from a univariate to a multivariate function to include additional variables such as temperature and solvent composition, focusing on that to extend it to a bivariate function of salt content and solvent composition with examples from the solutions of $LiPF_6$, $LiBF_4$, LiBOB, Et_4NPF_6, and Et_4NBF_4 in propylene carbonate (PC) + diethyl carbonate and in PC + ethylene carbonate and $LiClO_4$ in PC + acetonitrile. In addition, I discuss, along with experimental data and functional analysis, the validity of using this equation as a two-parameter function in relation to the range of salt content.

Introduction

Casteel and Amis published in 1972 a paper[1] containing a four-parameter equation to describe the electrolytic conductivity of an electrolyte as a function of its salt concentration, which has since been call the Casteel−Amis equation and has been used extensively and successfully to fit measured conductivity data over wide ranges of salt concentration for a great variety of electrolytes.[2−18]

The equation was originally expressed in two different but equivalent forms.[1] The first form contains four general parameters *a*, *b*, *c*, and *d*:

$$\kappa = m^a \exp(-bm^2 + cm + d) \tag{1}$$

where κ is the electrolytic conductivity and *m* is the salt molality. (Although Casteel and Amis used molarity *C* for the unit of salt concentration in their original paper, the use of *m* in place of *C* in the equation has been shown to be equally valid[2,4,5,7,9,11−13,15,16,18] and will therefore be adopted throughout this report.) The second form, a mathematical transformation of the first, contains the parameters *a* and *b* and two new parameters κ_p and m_p in place of *c* and *d*:

$$\kappa_r = m_r^{\,a} \exp[-bm_p^{\,2}(m_r - 1)^2 - a(m_r - 1)] \tag{2}$$

where the relative conductivity $\kappa_r = \kappa/\kappa_p$ and the relative molality $m_r = m/m_p$, with κ_p as the peak conductivity and m_p as the particular *m* at which $\kappa = \kappa_p$. Of the two forms, the second has been the more often used because of the clear physical meanings of parameters κ_p and m_p. It has sometimes even been used in a three-parameter form by setting *b* equal to zero.[19] Casteel and Amis fit their measured $\kappa(m)$ data with eq 1 and demonstrated the high suitability and flexibility of the equation for describing κ of an electrolyte as a function of its *m* in a range substantially extended from its m_p on both sides. Furthermore, upon reducing their measured $\kappa(m)$ data to the form $\kappa_r(m_r)$ for water−ethanol solutions of $MgSO_4$ and $MgCl_2$ and plotting these reduced data together, Casteel and Amis found these data to fall closely together on a common curve, regardless of the nature of the salt, composition of the solvent, and temperature of the electrolyte. They thus proposed that terms *a* and $bm_p^{\,2}$ of eq 2 be viewed as constants, of which an evaluation with a particular electrolyte would turn eq 2 into a two-parameter equation that should be applicable to any electrolyte. This line of thought has sometimes been followed in other studies, and parameters *a* and *b* have been evaluated and discussed in this regard.[15,18]

Because of the success of the Casteel−Amis equation in describing the conductivity of electrolytes over wide ranges of salt concentration, the equation has been extended from a univariate function $\kappa(m)$ to a bivariate function $\kappa(m, v)$ to fit $\kappa(m)$ data measured at different experimental conditions signified by the variable *v*. This has so far been achieved by setting the four parameters of the Casteel−Amis equation equal to functions of *v* in order for the new function to retain the property of the original in the *m* dimension. The first such extension was made by de Diego et al. by setting the four parameters of eq 2 equal to linear functions of temperature *T* in order to fit their measured data of $\kappa(m, T)$.[20,21] They succeeded in using a bivariate function $\kappa(m, T)$ to describe faithfully their experimental data of conductivity for aqueous solutions of HBF_4, H_2SiF_6, and H_2TiF_6 at around (15, 25, 35, and 45) °C. The second such extension of the Casteel−Amis equation was made by Ding to include in this equation the solvent mass fraction *w* in order to fit and describe the measured data of $\kappa(m, w)$.[22−27] He did this by setting the four parameters of eq 1 equal to second- to fourth-degree polynomials of *w* and fitting the resulting $\kappa(m, w)$ equation to the conductivity data measured at particular temperatures. By using an appropriate degree of the polynomial, with the help of commercial mathematical software (Mathematica), he and co-workers obtained bivariate functions $\kappa(m, w)$ that fit the experimental data closely for carbonate solutions of $LiPF_6$, $LiBF_4$, LiBOB, Et_4NPF_6, and Et_4NBF_4,[22−27] where BOB stands for bis(oxalato)borate.[28] These functions, when plotted in the coordinates of *m* and *w*, clearly revealed the dependency of κ on *m* and *w* for these electrolyte systems.

* E-mail: mding@arl.army.mil.

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Table 1. Degree of Polynomial *n* of Equation 3 and Average Fitting Error e_f as a Percentage of the Range of the Measured Data to Which the Polynomial Has Been Used in Fitting Equation 1 in the Temperature Range of (−80 to 60) °C[a]

salt	solvent	n	e_f	solvent	n	e_f
$LiPF_6$	PC + DEC	3	0.54	PC + EC	2	0.63
$LiBF_4$		3	0.46		2	0.37
LiBOB		4	0.64		2	0.25
Et_4NPF_6		3	0.38		2	0.99
Et_4NBF_4		3	0.46		2	0.14
$LiClO_4$	PC + AN	2	0.52			

[a] For $LiClO_4$ in PC + AN, mole fraction x has been used in eq 3 in place of w, and the temperature range is (−35 to 35) °C.

Ding also attempted to devise a trivariate function $\kappa(m, w, T)$ by setting the parameters of eq 1 equal to polynomial functions of both w and T in order to fit the measured $\kappa(m, w, T)$ data. This last attempt was met with only limited success: a good fit was possible only over a limited range of m.[22,23]

The first aim of this report is to provide a brief account of the creation, application, and extension of the Casteel−Amis equation, as has just been done in the Introduction. The second aim is to give a summary of the extension of this equation to include solvent composition to fit the conductivity data for carbonate solutions of $LiPF_6$, $LiBF_4$, LiBOB, Et_4NPF_6, and Et_4NBF_4 and a carbonate−acetonitrile (AN) solution of $LiClO_4$ and to give a brief description of the main features of the resulting $\kappa(m, w)$ surfaces of these fitting functions at different temperatures. The third aim is to discuss the validity of the reduction of eq 2 to the two-parameter function $\kappa(m, \kappa_p, m_p)$ by applying it to all of the above solution systems to observe the constancy of its parameters a and bm_p^2 across the different salts, solvents, and temperatures. A lack of constancy in these parameters, particularly a systematic change in a or bm_p^2 with w or T, would signify a lack of physical foundation for treating eq 2 as a two-parameter function and thus negate the universal validity of using it as such.

Extending the Casteel−Amis Equation to Include Solvent Composition

The extension of the Casteel−Amis equation to include solvent composition as an additional variable was accomplished using eq 1 as the starting form because this form was found to be more conducive to a successful nonlinear regression in its fit to experimental data, particularly after it was extended to a bivariate or multivariate function.[22−25] The inclusion of the mass fraction w of a binary solvent in eq 1 was done by setting its parameters to polynomial functions of w:

$$p = \sum_{i=0}^{n} p_i w^i \tag{3}$$

where p stands for a, b, c, or d of eq 1 and n is the degree of the polynomial. The value of n was chosen for a specific electrolyte system so that its use in eq 3 with eq 1 would result in satisfactory fits to the measured $\kappa(m, w)$ data at temperatures θ from (−80 to 60) °C, but the use of a value higher than n would not improve these fits significantly. (θ represents the temperature in °C, and T represents the temperature in K.[29]) Table 1 lists the values of n actually used in the fit for the solutions of the five salts in propylene carbonate (PC) + diethyl carbonate (DEC) and PC + ethylene carbonate (EC), along with the average fitting errors relative to the ranges of the measured data associated with n. In addition, I have applied the same procedure to fit the $\kappa(m, x)$ data of a PC-AN solution of $LiClO_4$ published by Barthel et al.,[15] where I used mole fraction x in eq 3 in place of w in accordance with the original data and listed the fitting results in the same Table. The small fitting error of 0.52% indicates that the procedure is applicable not only to carbonate solutions but also to other systems as long as the change in κ with w or x is not too abrupt.

Examples of $\kappa(m, w)$ surfaces according to these fitting functions are given in Figure 1 for the solutions of $LiPF_6$ in PC + DEC and in PC + EC.[22,24] As shown in Figure 1a, the surfaces of the PC + DEC solution are all dome-shaped as a result of κ peaking in both m and w. Those of the PC + EC solution, however, are all arch-shaped because κ in this solution peaks only in m, as shown in Figure 1b. Corresponding surfaces for the other salts in the same solvents behave similarly.[23,25−27] Note that the speed with which the surfaces of both solutions slope downward with m increases as the temperature is lowered, making the ridges of the $\kappa(m, w)$ surfaces appear narrower.

Another set of $\kappa(m, x)$ surfaces is given in Figure 2 for the solution of $LiClO_4$ in PC + AN on account of its $\kappa(m, x)$ data by Barthel et al.[15] never before being presented this way and the new features in its $\kappa(m, x)$ surfaces due to the presence of the non-carbonate AN. Among these new features, the most striking is that the continuous addition of AN in PC takes the κ of the solution continuously higher without reaching a peak, in sharp contrast to the addition of DEC in PC, as a comparison of Figure 2 with Figure 1a would show. This is likely the result of AN having a low viscosity comparable to that of DEC but a much higher dielectric constant, which prevents the eventual drop in κ that occurs when too much DEC is added to PC because of a strengthening ion association. For the same reasons, κ increases with the addition of the salt without reaching a peak in the AN-rich solvents, again contrasting sharply with the carbonate solvents. This contrast is most vividly demonstrated by a $\kappa(m, x)$ surface of Figure 2, where as x rises from zero and the solvent becomes more PC-rich a peak first appears in the $\kappa(m)$ curve and then shifts to lower and lower m values. These favorable properties of AN have made it the most widely used solvent for double-layer capacitors, though its use for lithium-ion batteries has been limited by its relatively low oxidative potential.[30]

Using Casteel−Amis Equation as a Two-Parameter Function

As suggested by Casteel and Amis, eq 2 can be used as a two-parameter function of $\kappa(m, \kappa_p, m_p)$ for all electrolytes once the terms a and bm_p^2 have been evaluated with a particular electrolyte. We now check the validity of this suggestion by observing the constancy of a and bm_p^2 across the salts, solvent compositions, and temperatures. To this end, the measured $\kappa(m)$ data were first fit with eq 1 for a determination of a, b, c, and d, from which m_p and κ_p were calculated with these relations:

$$m_p = \frac{c + \sqrt{8ab + c^2}}{4b} \tag{4}$$

$$\kappa_p = m_p^{\,a} \exp(-bm_p^{\,2} + cm_p + d) \tag{5}$$

Values of a and bm_p^2 thus determined for the solution $LiPF_6$ in PC + DEC are plotted in Figure 3 as an example, to which corresponding plots for solutions of other salts

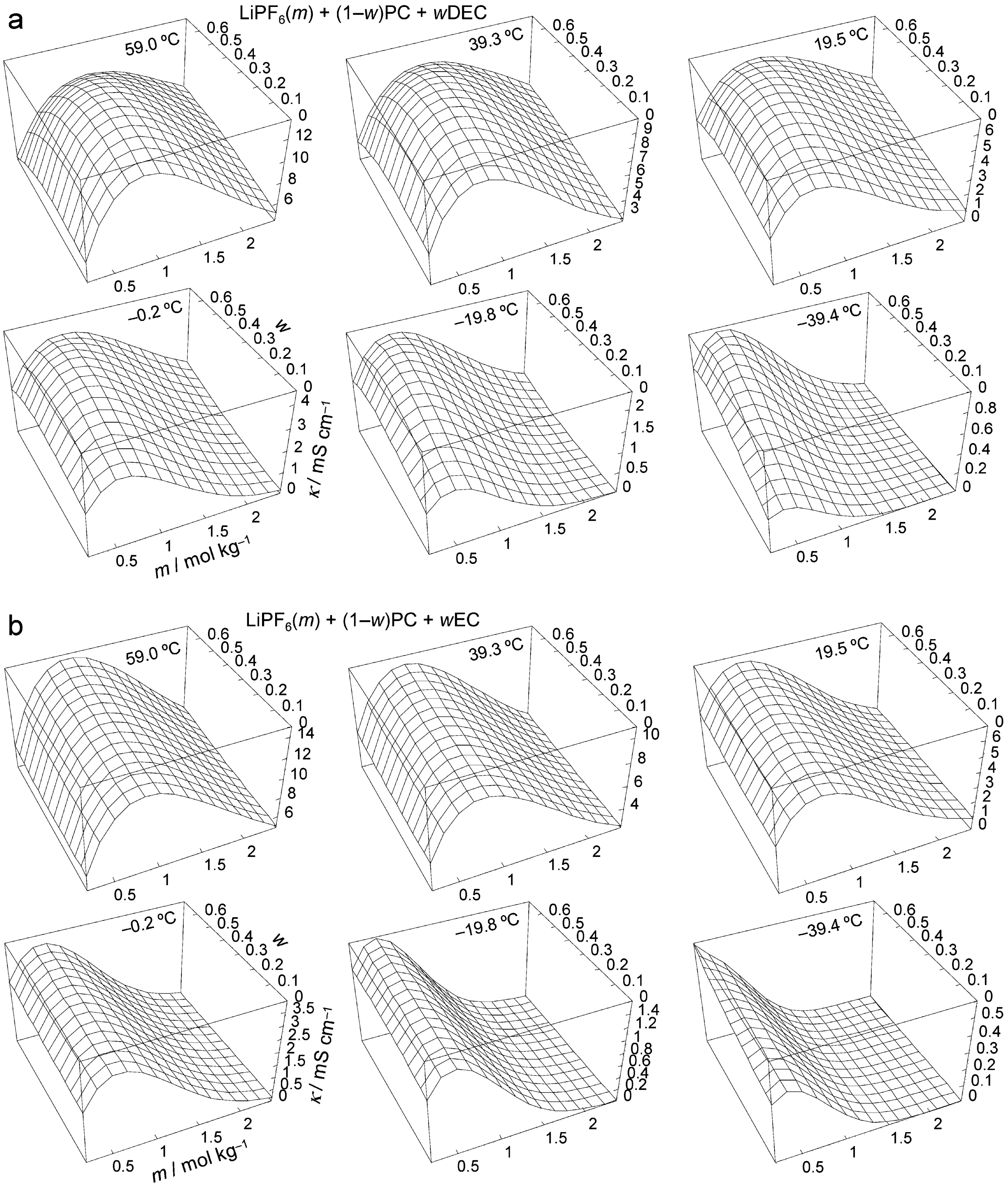

Figure 1. Change in conductivity κ with simultaneous changes in salt molality m and solvent mass fraction w for the solutions $LiPF_6(m)$ + $(1 - w)$PC + wDEC (a) and $LiPF_6(m)$ + $(1 - w)$PC + wEC (b) at temperatures of (59.0, 39.3, 19.5, −0.2, −19.8, and −39.4) °C.

would look similar. It is apparent from the Figure that the changes in a and bm_p^2 with either w or θ are systematic and generally in opposite directions. In particular, a decreases and bm_p^2 increases in value as θ is lowered.

Corresponding to these changes in value of a and bm_p^2, the associated experimental data when plotted in their reduced form of $\kappa_r(m_r)$ would deviate from a common curve of $\kappa_r(m_r, a, bm_p^2)$, where a and bm_p^2 are assumed to be constant. Such deviation is demonstrated with the measured data for $LiPF_6$ in PC at temperatures from (−80 to 60) °C in Figure 4a and for $LiPF_6$ in PC + DEC solvents at different compositions at −10 °C in Figure 4b. In both cases, as would be the case for other solutions, the deviations become particularly apparent at values of m higher than m_p, growing larger with m. Is this observation a mere coincidence for the electrolytes of this report, or does it manifest something deeper?

The answer to the first part of the question is no. Looking at the papers that tabulated values of a, b, and m_p, I found quite large variations in the values of both parameters a and bm_p^2 among different authors and for different electrolytes at different temperatures.[1,11,15,18] When these values were substituted into eq 2 and the equation was plotted over the range of m from 0 to 4, all of the curves

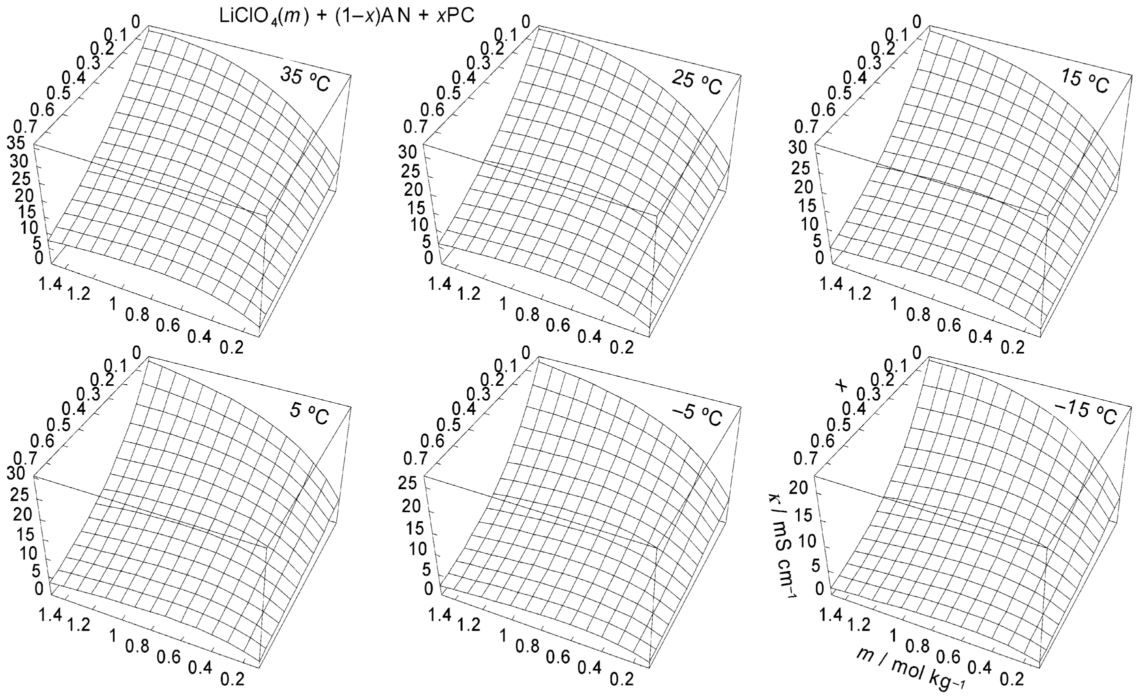

Figure 2. Change in conductivity κ with simultaneous changes in salt molality m and solvent mole fraction x for the solution $LiClO_4(m) + (1 - x)AN + xPC$ at temperatures of (35, 25, 15, 5, −5, and −15) °C.

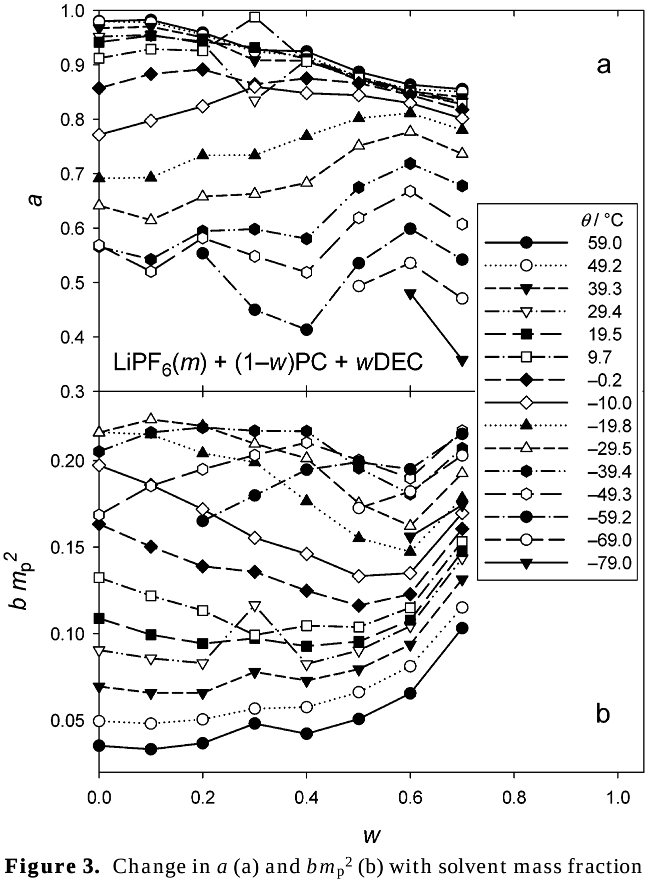

Figure 3. Change in a (a) and bm_p^2 (b) with solvent mass fraction w at temperatures θ from (60 to −80) °C in 10 °C intervals, as indicated, for the solution $LiPF_6(m) + (1 - w)PC + wDEC$.

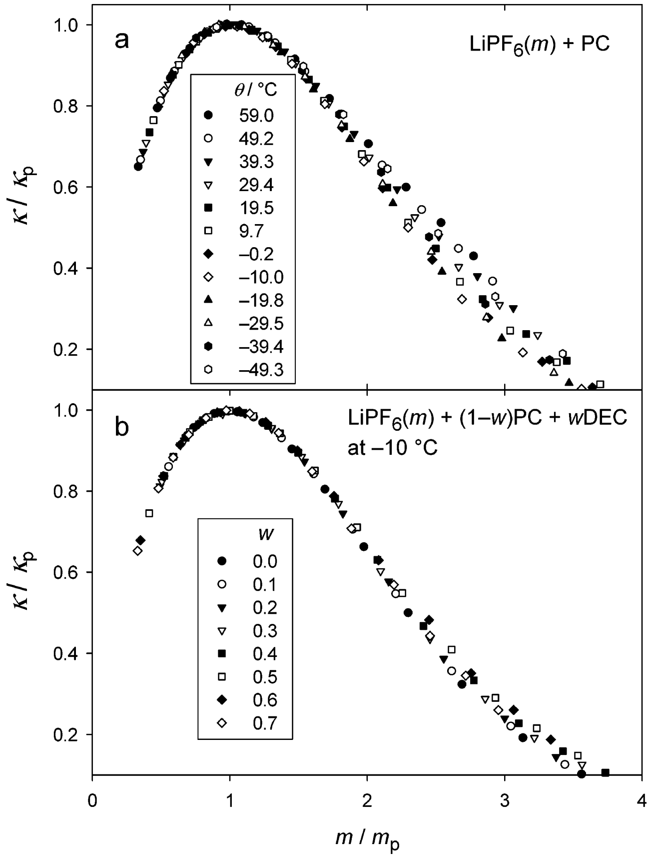

Figure 4. Measured conductivity data plotted in the form of $\kappa_r(m_r)$, where $\kappa_r = \kappa/\kappa_p$ and $m_r = m/m_p$ with κ_p as the peak conductivity and m_p as the molality corresponding to κ_p for the solution $LiPF_6(m) + (1 - w)PC$ at different temperatures θ (a) and for the solution $LiPF_6(m) + (1 - w)PC + wDEC$ for different values of w at −10 °C (b).

showed significant deviation from a common curve, especially at values of m higher than 2, as shown in Figure 4.

The answer to the second part of the question seems to be yes. As Figure 3b shows and as can be found in other publications,[1,11,15,18] parameter bm_p^2 generally rises in value when the temperature is lowered. This correlation of bm_p^2 with temperature is entirely consistent with the observation that the conductivity surfaces of Figure 1 slope downward faster with decreasing temperature because this

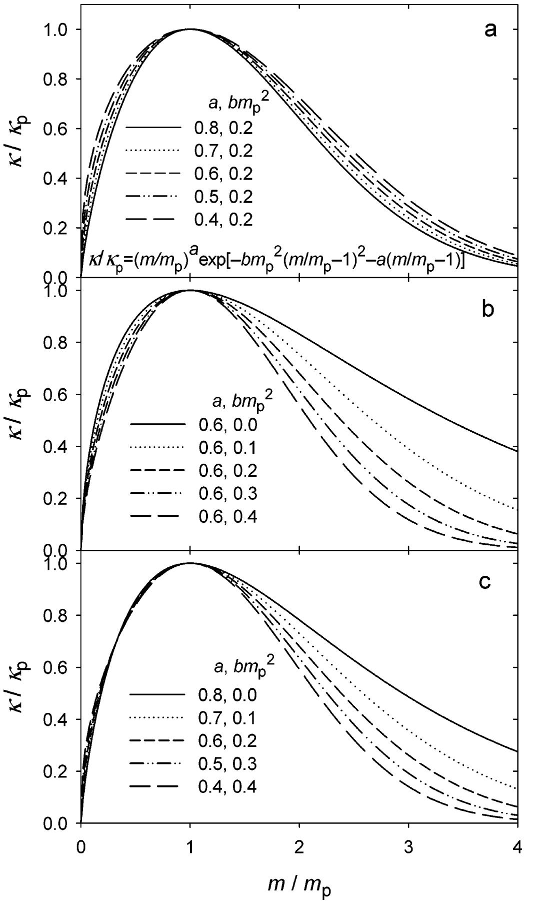

Figure 5. Plots of eq 2, as written in plot a, with variation in *a* (a), in bm_p^2 (b), and in both *a* and bm_p^2 in opposite directions (c) around the base values of (0.6, 0.2) for (a, bm_p^2). The values of (a, bm_p^2) for each situation are indicated.

parameter is the coefficient of the quadratic term in the exponent of eq 2, which dominates the decline of κ_r as m_r grows considerably larger than unity. It is probable that bm_p^2 is somehow related to the viscosity of the electrolyte. If this is true, then this would preclude any electrolyte from having overlapping $\kappa_r(m_r)$ curves for different temperatures at high salt concentrations. If we choose a pair of base values (0.6, 0.2) for (a, bm_p^2) that are typical of published values and vary bm_p^2 around 0.2, then we can see dramatic changes in the shape of the $\kappa_r(m_r)$ curves that use varying values for the parameters, as plotted in Figure 5b. However, the magnitude of the change is much smaller in the region of $m_r < 1$ than in that of $m_r > 1$, increasing rapidly as m_r becomes significantly greater than 1. However, if we vary a around the base value of 0.6 and keep bm_p^2 at 0.2 and plot, then we get Figure 5a, which shows that the changes in shape of the $\kappa_r(m_r)$ curves this time are more evenly distributed on the m_r axis and significantly smaller in magnitude at high values of m than that caused by the variation of bm_p^2. Most interestingly, if we vary both variables by equal amounts but in opposite directions, as we have done in Figure 5c, then we have $\kappa_r(m_r)$ curves that overlap almost perfectly at low values of m_r but deviate from a common curve quite strongly as m_r becomes significantly greater than unity. This simulated situation is well represented by the real examples of Figure 4. More importantly, correlated changes in opposite directions in a and bm_p^2 can be observed in nearly all published data.[1,11,15,18] It is therefore clear that treating a and bm_p^2 as two constants in eq 2 for all electrolytes is not a universally valid approach to describing their κ−m relationships, particularly for more concentrated electrolytes. The fact that the $\kappa_r(m_r)$ curves in m_r values from zero to slightly above unity for many electrolytes lie on a common curve is probably due to the combination of all of these curves having to pass the two common points of (0, 0) and (1, 1) and the usual opposite changes in a and bm_p^2 offsetting each other in their effects on the $\kappa_r(m_r)$ curves. Therefore, if one has to use eq 2 as a two-parameter function, then the application range needs to be restricted to values of m_r not much beyond unity.

Conclusions

The Casteel−Amis equation can and has been extended from a univariate to a bivariate or multivariate function by setting its four parameters equal to polynomial functions of the variable(s) to be included, such as temperature and solvent composition. In the latter case, the four parameters were set equal to polynomial functions of solvent composition from second to fourth degree, depending on the complexity of the change in conductivity with solvent composition. For the electrolyte solutions of $LiPF_6$, $LiBF_4$, LiBOB, Et_4NPF_6, and Et_4NBF_4 of salt content m in PC + DEC and PC + EC of solvent mass fraction w in the second component, this approach resulted in fitting functions that described the measured $\kappa(m, w)$ data in the m range of (0 to 2.5) mol kg^{-1}, the w range of (0 to 0.7), and the θ range of (−80 to 60) °C, with a typical fitting error of 0.5% of the data range. For a solution of $LiClO_4$ in AN + PC of solvent mole fraction x, the same procedure produced fitting functions $\kappa(m, x)$ in the m range of (0.1 to 1.5) mol kg^{-1}, the x range of (0, 0.8), and the θ range of (−35 to 35) °C, with an average fitting error of 0.5% of the data range. Furthermore, the data treated in this report, plus other published data, were examined, processed, and plotted for the purpose of verifying the validity of using the Casteel−Amis equation as a two-parameter function for all electrolytes, as Casteel and Amis proposed in their original paper. It was concluded that this suggestion would be valid only at salt concentrations not much beyond that where the conductivity peak occurs. Beyond that, such use was expected to result in substantial errors, which would grow with increasing salt content.

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