

Dielectric Relaxation Study Of 1,2 Dichloroethane With n,n-Dimethylformamide

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ARTICLE INFO

Kevwords:

Complex permittivity, static dielectric properties, excess properties, Kirkwood parameters, thermodynamic properties

ABSTRACT

The time domain reflectometry (TDR) technique has been used for the study of dielectric relaxation spectra of 1,2 dichloroethane (DE) and n,n-dimethylformamide (DMF) binary mixtures. The frequency range used for the study is 10 MHz to 30 GHz. The study is carried out at 10°C, 15°C, 20°C and 25°C temperature for 11 different concentrations. The dielectric parameters such as static permittivity (ϵ 0), dielectric constant at high frequency (ϵ ∞) and relaxation time (τ) have been obtained. The Excess parameters, Kirkwood parameters and thermodynamic parameters are determined.

1. Introduction

The time domain reflectometry is very efficient tool to identify inter and intra molecular interaction between polar molecules. The 1,2-dichloroethane (DE) is an oily, colorless and heavy non-associative liquid which is moderately soluble in water. n,n-dimethylformamide (DMF) is a clear liquid. This colorless liquid is miscible with water and majority of organic liquids. DMF is a polar and a protic solvent with high boiling point. DMF is used as an industrial solvent and in the production of fibers, films and surface coating.

The main objective of this work is to study the dielectric parameters, excess properties, Kirkwood correlation parameters and thermodynamic parameters in the DE-DMF system using time domain reflectometry (TDR) for various concentrations at different temperatures. DE and DMF have high polarity, strong solvating power, and a large liquid range, which makes them industrial and technological important solvents. The DE and DMF linkages (-CH2-Cl) and (NH-

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C=0) respectively, are an important functional groups in chemistry, biochemistry, pharmaceutical and material science.

2. Experimental and data analysis

2.1 Material

1, 2 dicholroethane (AR grade, Qualigens fine chemicals Pvt. Ltd., Mumbai, India) and n,n-dimethylformamide (Merck Specialties, Pvt. Ltd., Mumbai, India) were used without further purification. The solutions were prepared at 11 different volume percentage of DMF in DE from 0% to 100% just before the measurements. Using these volume percent, the mole fraction is calculated as

$$x = (v_1 \rho_1 / m_1) / [(v_1 \rho_1 / m_1) + (v_2 \rho_2 / m_2)]$$
 (1)

where m_i , v_i and ρ_i represent the molecular weight, volume percent and density of the i^{th} (i=1, 2) liquids respectively.

2.2 Apparatus

The complex permittivity spectra were obtained by the time domain reflectrometry (TDR) technique. Tektronix model number DSA8200 Digital serial analyzer sampling mainframe along with the sampling module 80E08 has been used for the TDR technique. A repetitive fast rising voltage pulse with 18ps incident rise time was fed through the coaxial line system of impedance 50 ohm. All measurements are carried out in open load condition. Sampling oscilloscope monitors changes in the step pulse after reflection from the end of the line. Reflected pulse without sample $R_1(t)$ and with sample $R_X(t)$ were recorded in a time window of 5ns and digitized in 2000 points. The nature of the reflected pulse with and without sample for DE and DMF are observed as shown in Fig. 1. After receiving time referenced reflected pulses $R_1(t)$ and $R_X(t)$, these pulses are subtracted and added in the oscilloscope memory. This time dependent subtracted pulse is represented by p(t) and added pulse by q(t) as shown in Figs. 2 and 3.

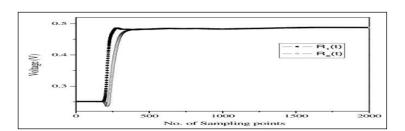


Figure 1: Reflected pulse with sample $R_x(t)$ and without sample $R_1(t)$

2.3 Data analysis

The time dependent data were analyzed to obtain complex reflection coefficient spectra $\rho^*(\omega)$ over the frequency range from 10 MHz to 30 GHz using Fourier transformation as

$$\rho^*(\omega) = (c/j\omega d)[p(\omega)/q(\omega)]$$
 (2)

where $p(\omega)$ and $q(\omega)$ are Fourier transforms of [$R_1(t)$ - $R_x(t)$] and [$R_1(t)$ + $R_x(t)$] respectively, c is the velocity of light, ω is angular frequency, d is the effective pin length and $j = \sqrt{-1}$.

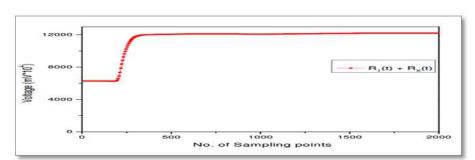
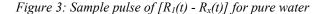
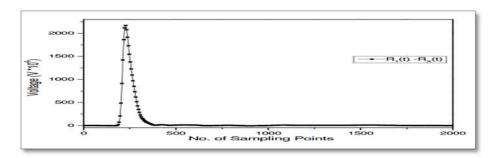


Figure 2: Sample pulse of $[R_1(t) + R_x(t)]$ for pure water





The complex permittivity spectra $\epsilon^*(\omega)$ were obtained from reflection coefficient spectra $\rho^*(\omega)$ by implementing a bilinear calibration method. The general form of the relaxation model is given by the Havriliak-Negami expression .

$$\varepsilon^* (\omega) = \varepsilon_{\infty} + \{ (\varepsilon_0 - \varepsilon_{\infty}) / [1 + (j\omega\tau)^{(1-\alpha)}]^{\beta} \}$$
 (3)

where $\epsilon^*(\omega)$ is the complex permittivity at an angular frequency ω , ϵ_0 is the static permittivity, ϵ_∞ is the permittivity at high frequency, τ is the relaxation time of the system, α is the shape parameter representing symmetrical distribution of relaxation time and β is the shape parameter of an asymmetric relaxation curve.

These α and β values represents the type of model followed by the system under consideration as shown in Tab. 1.

Table 1: Different type of models

S.No	α and β values	Model name	Significance				
1.	α=0, β=1	Debye Model	It gives the complex nature of the dielectric permittivity wi				
			loss. The real part complex permittivity indicates orientation				
			effect of dielectric field with polarization and follows the				
			applied electric field, whereas imaginary part indicates				
			chaotic motion leading to thermal dissipation with opposing				
			the applied field.				
2.	$0 \le \alpha \le 1, \beta=1$	Cole-Cole Model	A material with multiple relaxation frequencies will be				
		(CC)	indicated by a semicircle (symmetric distribution) or an ar				
			(non- symmetric distribution) with its centre lying between				
			the horizontal $\varepsilon''=0$ axis				
3.	$\alpha=0, 0 \le \beta \le 1$	Cole-Davidson	It describes the asymmetric distribution model. It is				
		Model (CD)	corresponding to relaxation time and gives a skewed arc ϵ'				
			(ϵ'') . The molecule become less rigid with increase in chain				
			length and may be relax at many ways.				

The relaxation behavior of DE-DMF system agrees with the Debye model. Therefore the experimental values of ε^* (ω) were fitted with the Debye equation by using least square fit method.

$$\varepsilon^*(\omega) = \varepsilon_{\infty} + \left[\left(\varepsilon_0 - \varepsilon_{\infty} \right) / \left(1 + j\omega \tau \right) \right] \tag{4}$$

where ε_0 , ε_∞ and τ are the adjustable parameters.

The frequency dependent complex permittivity spectrum at 25°C temperature is shown in Fig. 4. The dielectric constant values are high in the lower frequency region and decreases with increase in frequency. The dielectric loss has also studied as a function of frequency for 11 different concentrations at 25°C temperature. At high frequency the dielectrics loss is less indicating very high purity of the DE-DMF liquid mixture. These curves represent the dielectric loss is mainly dependents on the frequency of the applied field. Only one relaxation peak is observed for DE, DMF and DE-DMF mixtures.

The complex plane plots for DE, DMF and DE-DMF mixture at 10, 15, 20 and 25°C temperatures are shown in Fig. 5. The dielectric loss values for pure liquids are very less and increases with the increase in the concentration of liquid mixture.

3. Results and discussions

3.1 Static dielectric parameters

The dielectric parameters of material are a measure of the extent to which the electric charge distributed in the material, when an external electric field is applied to the material.

The variation of static dielectric constant (ϵ_0) and relaxation time (τ) with mole fraction of DMF is shown in Tab. 2. The values of static dielectric constant and relaxation time decrease with increase in temperature as expected it is due to increase of density with decrease in temperature. The value of dielectric parameters increases with increase in the concentration of DMF in DE. The increase in static dielectric constant may be correlated to disturbance in antiparallel arrangement of dipoles which leads to increase effective dipole moment.

Table 2: Dielectric relaxation parameters for DE-DMF mixtures

x ₂	T=10°C	T=15°C	T=20°C	T=25°C	T=10°C	T=15°C	T=20°C	T=25°C
	ϵ_0				τ(ps)			
0.0000	12.57(1)	10.94(1)	10.33(0)	9.98(1)	9.75(3)	9.61(3)	9.42(3)	9.35(3)
0.0980	15.96(2)	14.89(1)	13.96(0)	13.59(0)	13.26(3)	13.17(2)	12.81(2)	12.25(2)
0.1964	19.33(1)	17.97(1)	17.14(1)	16.53(0)	13.63(4)	13.54(2)	13.29(2)	12.93(1)
0.2953	22.47(2)	21.17(1)	20.11(1)	19.15(1)	14.61(3)	14.17(2)	13.57(1)	12.86(1)
0.3946	26.22(1)	24.52(1)	23.39(1)	22.9(1)	15.07(3)	14.51(2)	13.81(1)	13.56(1)
0.4943	26.06(1)	24.62(1)	23.91(1)	23.48(1)	14.99(2)	14.59(2)	14.17(2)	14(2)
0.5945	31.42(1)	29.73(1)	28.6(2)	27.82(2)	14.47(1)	13.8(2)	13.2(2)	12.69(2)
0.6952	36.5(3)	33.81(1)	32.63(1)	30.75(2)	14.43(2)	13.59(1)	13.12(1)	12.09(2)
0.7963	37.22(1)	34.86(3)	33.38(3)	32.16(3)	13.51(0)	12.71(2)	12.1(3)	11.48(3)
0.8979	40.61(2)	37.66(3)	6.05(4)	34.87(3)	12.47(1)	11.58(2)	11.01(3)	10.55(3)
1.0000	41.2(4)	39.08(0)	37.69(2)	36.41(2)	12.18(4)	11.34(0)	10.64(1)	10.18(2)

where x_2 represents the mole fraction of DMF in DE. Number in the bracket represents error in the corresponding value, e.g., 12.57 (1) means 12.57 ± 0.01 .

3.2 Excess parameters

The information related to the structural changes of liquid 1 and 2 may be obtained by excess properties of dielectric constant and relaxation time

The excess permittivity ϵ^{E} is defined as

$$\varepsilon^{E} = (\varepsilon_{0} - \varepsilon_{\infty})_{m} - [(\varepsilon_{0} - \varepsilon_{\infty})_{1} x_{1} + (\varepsilon_{0} - \varepsilon_{\infty})_{2} x_{2}]$$
(5)

where x- mole fraction and suffices m, 1, 2 represents mixture, liquid 1 and liquid 2 respectively.

The excess permittivity (ϵ^E) may provide qualitative information about multimers formation in the mixture as shown in Fig. 6 (a). This figure shows that the excess dielectric constant values are negative in DE rich region and positive in DMF rich region at four different temperatures; this indicates that the effective dipoles of the liquid system get reduced in DE rich region and the effective dipoles of the liquid system get increased in DMF rich region.

Similarly, the excess inverse relaxation time is defined as

$$(1/\tau)^{E} = (1/\tau)_{m} - [(1/\tau)_{1} x_{1} + (1/\tau)_{2} x_{2}]$$
(6)

where x is mole fraction and suffices m, 1, 2 represents mixture, liquid 1 and liquid 2 respectively.

From Fig. 6 (b), the excess inverse relaxation time values are negative in DE-DMF system for various concentrations at all temperatures. The negative value of excess inverse relaxation time indicates slower rotation of the dipole of the system.

Figure 4: Complex permittivity spectra $\varepsilon^*(\omega)$ of DE-DMF mixture for 11 different concentrations at 25°C

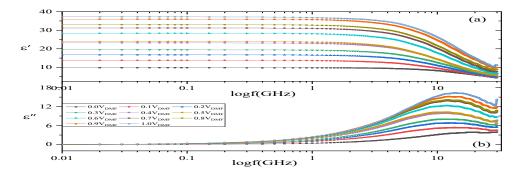
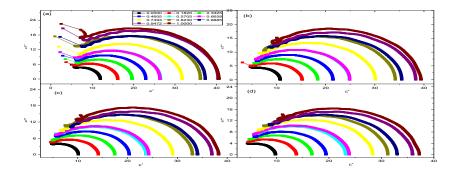


Figure 5: Complex plane plot of dielectric constant (ϵ ') vs. dielectric loss (ϵ '') for various concentrations of FA in DE at (a) $T = 10^{\circ}$ C, (b) $T = 15^{\circ}$ C, (c) $T = 20^{\circ}$ C, and (d) $T = 25^{\circ}$ C

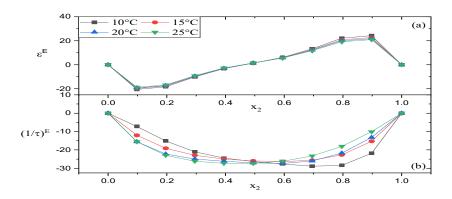


The experimental values of both the excess parameters were fitted to the Redlich-Kister equation.

$$A^{E}=(x_{1} x_{2}) \sum_{n} B_{n} (x_{1} x_{2})^{n}$$
 (7)

where A is either ε^E or $(1/\tau)^E$, x_1 and x_2 are molar fraction of two liquids, the coefficient B_n is a fitting coefficient to be determined by the least square fit method, for n=0 to 3 are listed in Tab. 3. The negative sign represents formation more favorable, whereas the positive sign represents breaking more favorable.

Figure 6: Excess parameters of DMF in DE (a) ε^E versus mole fraction (x_2) of DMF in DE (b) $(1/\tau)^E$ versus mole fraction (x_2) of DMF in DE



T/°C	Excess permittivity (ϵ^{E})				Exce	Excess inverse relaxation time $(1/\tau)^E$			
	B_0	\mathbf{B}_1	B_2	B ₃	B_0	B_1	B_2	B ₃	
10	6.46	83.1	20.29	355.28	-105.37	-28	-85.96	-109.23	
15	6.5	78.9	14.14	336	-103.99	-13.41	-73.78	-9.9	
20	6.71	77.99	9.67	323.03	-108.21	-13.48	-80.1	51.05	
25	6.85	72.63	6.43	316.55	-109.01	11.43	-51.87	45.7	

Table 3: Bn quoficient of the Redlich-Kister equation

3.3 Kirkwood parameters

In binary polar liquid 1 & 2, molecules of liquid 1 interact with the molecules of liquid 2, resulting in the formation of multimer-type of structures, resulting dipolar reorientation. The value of g for pure liquid may be obtained by the expression

$$\frac{4\Pi N\mu^2 \rho}{9kTM} g = \frac{(\varepsilon_0 - \varepsilon_\infty)(2\varepsilon_0 + \varepsilon_\infty)}{\varepsilon_0(\varepsilon_\infty + 2)^2}$$
 (8)

where μ is dipole moment in gas phase, ρ is density at temperature T, M is molecular weight, k is Boltzman constant, N is Avogadro's number.

Fig. 7 (a) represents the variation of Kirkwood effective factor (g^{eff}) with the volume percent of DMF. From Fig. 7 (a) the g^{eff} values for pure DMF are higher than unity and for pure DE are less than unity at all the temperatures. The Kirkwood parameter for pure DMF liquid indicates the dipole gets aligned such that the effective dipoles become large whereas for pure DE indicates that the molecules prefer an ordering with antiparallel dipoles. The values of g^{eff} seems that the parallel alignment in the DMF rich region and anti-parallel alignment in the DE rich region of electrical dipoles with the applied field of the system.

Fig. 7 (b) shows the variation of Kirkwood correlation factor (g_f) with the volume percent of DMF. The g_f values give the information about the angular correlation within the molecules of the system. The values of g_f are greater than unity for all concentrations at various temperatures in DE-DMF system. It indicates the effective dipole values in DE-DMF system will be higher than the average value in pure liquids. This also seems that strong inter molecular interaction is observed in DE-DMF system for 11 different concentrations at all temperatures.

1.2 15°C 25°C 25°C 25°C (a) 1.10 0.0 0.2 0.4 \$\phi_2\$ 0.6 0.8 1.0 (b) \(\frac{1}{2} \) (b)

0.4

0.6

0.2

0.0

Figure 7: Kirkwood parameters of DMF in DE at different temperatures (a) Variation of g^{eff} versus volume percent (ϕ_2) of DMF in DE (b) Variation of g_f versus volume percent (ϕ_2) of DMF in DE

3.4 Bruggeman parameters

The modified Bruggeman equation is also a parameter which may be used an indicator of liquid 1 and 2 interactions. The Bruggeman factor f_B is given by

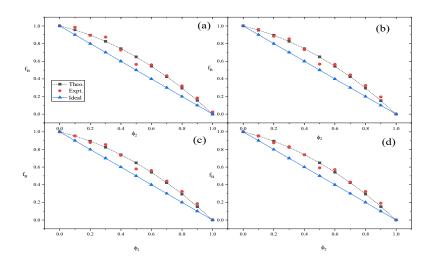
$$f_{\rm B} = \left(\frac{\varepsilon_{\rm 0m} - \varepsilon_{\rm 02}}{\varepsilon_{\rm 01} - \varepsilon_{\rm 02}}\right) \left(\frac{\varepsilon_{\rm 01}}{\varepsilon_{\rm 0m}}\right)^{1/3} = (1 - \phi_2) \tag{9}$$

0.8

1.0

According to Eq. 9, a linear relationship is expected which will gives a straight line when a graph is plotted f_B against ϕ_2 (DMF). However, here the experimental values of f_B were found to deviate from the linear relationship. The Bruggeman dielectric factor f_B versus volume fraction (ϕ_2) of DMF at various temperatures is given in Fig. 8.

Figure 8: Bruggeman factor (f_B) versus volume fraction (ϕ_2) of DMF at (a) 10° C (b) 15° C (c) 20° C (d) 25° C. Dashed line represents theoretical Bruggeman model, solid line represents Bruggeman model obtained from equation and marker \bullet denotes experimental results



3.5 Thermodynamic parameters

The thermodynamic parameters like molar enthalpy of activation (ΔH) and molar entropy of activation (ΔS) are determined from the Eyring rate equation utilizing least square fit method.

$$\tau = \frac{h}{KT} \exp \left(\Delta H - T \Delta S\right) / RT \tag{10}$$

In binary mixture of polar liquids, there is a change in the energy of the system. This change in energy can be interpreted with thermodynamic parameters such as free energy of activation (ΔG), molar enthalpy of activation (ΔH) and molar entropy of activation (ΔS) are shown in Tab. 3.

The ΔG values for pure DMF are greater than DE for all temperatures and these values increases with the increase in the temperature. This indicated that the DMF molecules in the mixture dominate over DE molecules. This dominance of DMF over DE is primarily due to presence of C=O and its large dipole moment values. The molar enthalpy of activation (ΔH) values is negative for all concentrations of the system.

The plot of ln (τ T) versus 1000/T of DE-DMF for various concentrations should be a straight line is as shown in Fig. 9.

3.3

3.2

3.1

Figure 9: Arrhenius behavior of DE-DMF system at different temperatures

Table 4: Molar enthalpy of activation (ΔH), entropy of activation (ΔS) and free energy of activation (ΔG) for DE-DMF system

1000/Temp(k)

3.4

3.5

3.6

	Δ0	ΔH KJ	ΔS KJ		
T=10°C	T=15°C	T=20°C	T=25°C		
9.531456	9.706369	9.881282	10.0562	-0.36863	-0.03498
10.2796	10.43845	10.5973	10.75615	1.28872	-0.03177
10.33409	10.5157	10.69732	10.87893	0.05489	-0.03632
10.49968	10.6225	10.74532	10.86815	3.54789	-0.02457
10.55252	10.69073	10.82895	10.96716	2.7296	-0.02764
10.53937	10.71009	10.88081	11.05153	0.87667	-0.03414
10.45911	10.57792	10.69673	10.81555	3.7343	-0.02376
10.46163	10.54909	10.63655	10.72402	5.51119	-0.01749
10.29569	10.38699	10.47828	10.56958	5.12839	-0.01826
10.09401	10.17798	10.26194	10.34591	5.34163	-0.01679
10.04445	10.11525	10.18605	10.25685	6.03713	-0.01416
	9.531456 10.2796 10.33409 10.49968 10.55252 10.53937 10.45911 10.46163 10.29569 10.09401	T=10°C T=15°C 9.531456 9.706369 10.2796 10.43845 10.33409 10.5157 10.49968 10.6225 10.55252 10.69073 10.53937 10.71009 10.45911 10.57792 10.46163 10.54909 10.29569 10.38699 10.09401 10.17798	9.531456 9.706369 9.881282 10.2796 10.43845 10.5973 10.33409 10.5157 10.69732 10.49968 10.6225 10.74532 10.55252 10.69073 10.82895 10.53937 10.71009 10.88081 10.45911 10.57792 10.69673 10.46163 10.54909 10.63655 10.29569 10.38699 10.47828 10.09401 10.17798 10.26194	T=10°C T=15°C T=20°C T=25°C 9.531456 9.706369 9.881282 10.0562 10.2796 10.43845 10.5973 10.75615 10.33409 10.5157 10.69732 10.87893 10.49968 10.6225 10.74532 10.86815 10.55252 10.69073 10.82895 10.96716 10.53937 10.71009 10.88081 11.05153 10.45911 10.57792 10.69673 10.81555 10.46163 10.54909 10.63655 10.72402 10.29569 10.38699 10.47828 10.56958 10.09401 10.17798 10.26194 10.34591	T=10°C T=15°C T=20°C T=25°C 9.531456 9.706369 9.881282 10.0562 -0.36863 10.2796 10.43845 10.5973 10.75615 1.28872 10.33409 10.5157 10.69732 10.87893 0.05489 10.49968 10.6225 10.74532 10.86815 3.54789 10.55252 10.69073 10.82895 10.96716 2.7296 10.53937 10.71009 10.88081 11.05153 0.87667 10.45911 10.57792 10.69673 10.81555 3.7343 10.46163 10.54909 10.63655 10.72402 5.51119 10.29569 10.38699 10.47828 10.56958 5.12839 10.09401 10.17798 10.26194 10.34591 5.34163

where x2 represents the volume percent of DMF in DE

4. Conclusion

Dielectric parameters, excess parameters, Kirkwood parameters and thermodynamic parameters have been reported for DE-DMF system for various concentrations and temperatures. As the concentration of DMF increases in DE the static permittivity, relaxation time and Kirkwood correlation factor g^{eff} values increases for all temperatures. The values of ϵ^E and g^{eff} seems that the parallel alignment in the DMF rich region and anti-parallel alignment in the DE rich region of electrical dipoles with the applied field of the system.

Acknowledgement

The department of Science and Technology, New Delhi is grateful acknowledged (Project no. SR/S2/LOP-25/2007) for laboratory facility. We are thankful to the A. C. Kumbharkhane for providing TDR facility and Dr. S. C. Mehrotra, Ramanujan Chair Professor, Department of Computer Science and IT, Dr. B.A.M. University, Aurangabad for their valuable discussion and suggestions.

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