

Dielectric Relaxation Study of 2-Pentenenitrile and 1, 2 Dichloroethane at 450c Temperature Using TDR

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ABSTRACT

The dielectric relaxation study of 2-pentenenitrile with 1, 2 dichloroethane mixture has been carried out at temperature 45°C. The dielectric relaxation study of solute-solvent mixture at microwave frequencies gives information about the formation of monomers and multimers as well as interaction between the molecules of the mixture. The dielectric parameters viz. static dielectric constant (ϵ_0) and relaxation time (τ) have been obtained by the least squares fit method. The static dielectric constant increases and relaxation time decreases with increase in concentration of 2-pentenenitrile in 1, 2-dichloroethane in the system. The time domain reflectometry (TDR) has been used for the study of dielectric relaxation spectra of 2-pentenenitrile (PN) and 1,2 dichloroethane (DCE) binary mixtures. The frequency range has been used as 10 MHz to 20 GHz. The relaxation in this system can be described by a single relaxation time using the Debye model. The Excess parameters such as excess permittivity (ϵ^E) and excess inverse relaxation time $(1/\tau)^E$ of the mixtures have been determined. In the mixtures excess permittivity (ϵ^E) found positive and the excess inverse relaxation time $(1/\tau)^E$ found negative.. The investigation shows that the effective dipoles of the system increases. It also shows that due to the hindering field between the constituent molecules the dipole rotates slowly.

Keywords: TDR, Nitrile, Chlorine, Excess permittivity, Excess inverse relaxation time.

I. INTRODUCTION

The dielectric relaxation parameters of binary mixture give information about solute-solvent interaction. Time Domain Reflectometer (TDR) is used to obtain the dielectric parameters of the system [1-3]. It also provides the information about the charge distribution in a molecular system. The liquid PN is of $C\equiv N$ group and DCE of chlorine group. It is interesting to see the interaction of nitrile group with chlorine-group. The frequency dependent complex permittivity measurements using TDR is more powerful technique because a single measurement covers a wide frequency range in a very short time. Several workers have studied the

temperature dependent dielectric relaxation parameters.

II. MATERIAL AND APPARATUS

A spectrograde 2-pentenenitrile(PN) (Fluka cheme GmbH-9471 Buchs, Steinheim, Switzerland) and AR grade 1, 2 dichloroethane(DCE) (E-Merck) were used without further purification. The solutions were prepared at 11 different volume percentages of PN in DCE from 0 % to 100 % just before the measurements. Using these volume percents the mole fraction is calculated as

$$x_1 = (v_1\rho_1/m_1) / [(v_1\rho_1/m_1) + (v_2\rho_2/m_2)]$$

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. The density and molecular weight of the liquids are as follows:

2-pentenitrile(PN)- density:0.821gmcm⁻³; mol.wt.-81.12

1, 2 Dichloroethane(DCE)-density:1.256gmcm⁻³;mol.wt.-98.96

The complex permittivity spectra were studied using the time domain reflectometry [4-5] method. The Hewlett Packard HP 54750 sampling oscilloscope with HP 54754A TDR plug in module has been used. A fast rising step voltage pulse of about 39 ps rise time generated by a pulse generator was propagated through a coaxial line system of characteristic impedance 50 Ohm. Transmission line system under test was placed at the end of coaxial line in the standard military applications (SMA) coaxial connector with 3.5 mm outer diameter and 1.35 mm effective pin length. All measurements were carried out under open load conditions. The change in the pulse after reflection from the sample placed in the cell was monitored by the sampling oscilloscope. In the experiment, time window of 5 ns was used. The reflected pulse without sample $R_1(t)$ and with sample $R_x(t)$ were digitized in 1024 points in the memory of the oscilloscope and transferred to a PC through 1.44 MB floppy diskette drive.

III. DATA ANALYSIS

The time dependent data were processed to obtain complex reflection coefficient spectra $\rho^*(\omega)$ over the frequency range from 10 MHz to 20 GHz using Fourier transformation [6, 7] as

$$\rho^*(\omega) = (c/j\omega d)[p(\omega)/q(\omega)] \quad (1)$$

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 $j = \sqrt{-1}$.

The complex permittivity spectra $\epsilon^*(\omega)$ were obtained from reflection coefficient spectra $\rho^*(\omega)$ by applying bilinear calibration method [5].

The experimental values of ϵ^* are fitted with the Debye equation [8]

$$\epsilon^*(\omega) = \epsilon_\infty + \frac{\epsilon_0 - \epsilon_\infty}{1 + j\omega\tau} \quad (2)$$

with ϵ_0 , ϵ_∞ and τ as fitting parameters. A nonlinear least-squares fit method [9] was used to determine the values of dielectric parameters. In Eq.(2), ϵ_0 is the static dielectric constant, ϵ_∞ is the limiting high-frequency dielectric constant and τ is the relaxation time.

IV. RESULTS AND DISCUSSION

The static dielectric constant (ϵ_0) and relaxation time (τ) obtained by fitting experimental data with the Debye equation are listed in Table 1. The values of static dielectric constant (ϵ_0) increases and relaxation time (τ) values decrease at 10% then increase for 20% and then it shows decreasing trend with the increase of concentration of PN into DCE.

The information related to liquids 1 and 2 interaction may be obtained by excess properties [10] related to the permittivity and relaxation times in the mixture. The excess permittivity ϵ^E is defined as

$$\epsilon^E = (\epsilon_0 - \epsilon_\infty)_m - [(\epsilon_0 - \epsilon_\infty)_1 x_1 + (\epsilon_0 - \epsilon_\infty)_2 x_2] \quad (3)$$

Where x - mole fraction and suffices m , 1, 2 represents mixture, liquid 1 (PN) and liquid 2 (DCE) respectively. The excess permittivity may provide qualitative information about multimers formation in the mixture.

Similarly, the excess inverse relaxation time is defined as

$$(1/\tau)^E = (1/\tau)_m - [(1/\tau)_1 x_1 + (1/\tau)_2 x_2] \quad (4)$$

Where $(1/\tau)^E$ is excess inverse relaxation time which represents the average broadening of dielectric spectra.

The inverse relaxation time analogy is taken from spectral line broadening (which is inverse of the relaxation time) in the resonant spectroscopy [11].

The experimental values of both the excess parameters were fitted to the Redlich-Kister equation [12, 13]

$$A^E = (x_1 x_2) \sum_n B_n (x_1 - x_2)^n$$

Where A is either ϵ^E or $(1/\tau)^E$. By using these B_n values, A^E values were calculated.

Figure 1, shows behavior of excess permittivity and excess inverse relaxation time for the system as a function of volume concentration of PN in DCE at 45°C temperature.

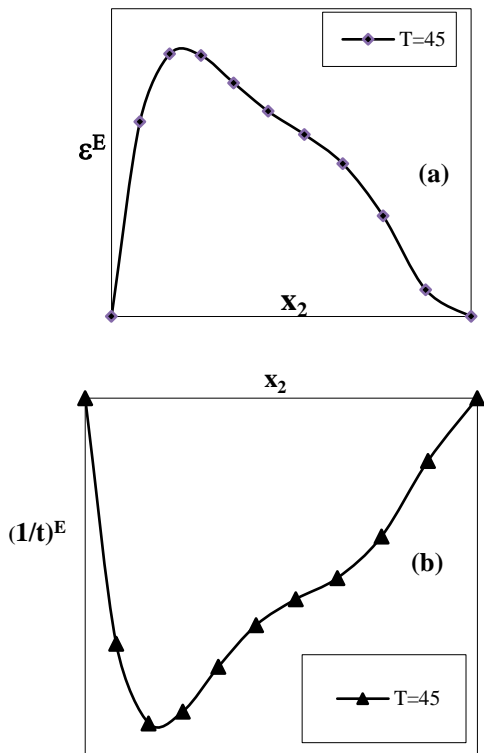


Figure 1. (a) The excess permittivity (ϵ^E) versus volume fraction of PN in DCE (b) The excess inverse relaxation time $(1/\tau)^E$ versus volume fraction of PN in DCE.

Table1: Static dielectric constant (ϵ_0) and relaxation time (τ) for 45°C temperature.

Vol. percentage of PN in DCE	ϵ	τ (ps)
0	10.88	11.46
10	13.11	11.2
20	15.69	11.67
30	18.24	11.37
40	20.83	11.04
50	23.43	10.33
60	25.87	9.36
70	28.98	8.84
80	31.99	6.78
90	35.19	5.2
100	37.48	3.52

In the system of PN-DCE, from the excess permittivity (ϵ^E) curve we can see that the excess permittivity (ϵ^E) values are positive for all concentrations. The positive peak is obtained at 0.1618

concentrations. From positive peak the values are gradually decreases. The positive values indicate that; there is formation of monomeric structures which leads to increase in total number of dipoles in the system. It also shows parallel alignment of the dipoles.

The excess inverse relaxation time $(1/\tau)^E$ values are negatives for all concentrations. The negative peak is obtained at 0.1618 concentrations. From the peak point to pure PN the values are decreases continuously. The negative values of $(1/\tau)^E$ indicates that; the effective dipoles in the system creates hindering field. It also represents that; the effective dipole rotation becomes slowly.

V. CONCLUSION

The dielectric parameters such as dielectric constant (ϵ_0) and relaxation time (τ) values of 2-pentenenitrile (PN)

and 1, 2 dichloroethane (DCE) mixture are obtained by analyzing dielectric relaxation spectra. The excess permittivity and excess inverse relaxation time values are also reported at 45°C temperature for 11 different concentrations in the frequency range of 10 MHz to 20 GHz. This data provides information regarding solute-solvent interaction. From the present study we can conclude that; the total number of dipoles in the system increases and shows the parallel alignment of the dipoles in the system. We also conclude that; the dipoles produces opposing field to each other in the mixture and the dipoles in the system rotates slowly.

VI. REFERENCES

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