# Dielectric Measurements on Methyl Acetate + Alcohol Mixtures at (288, 298, 308, and 318) K Using the Time Domain Technique

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Using picosecond time domain reflectometry (TDR), dielectric relaxation studies have been carried out for methyl acetate + methanol, methyl acetate + ethanol, and methyl acetate + propan-1-ol mixtures over the frequency range 10 MHz to 20 GHz at (288, 298, 308, and 318) K. The relaxation in this system can be described by a single relaxation time using the Debye model. The bilinear calibration method and least-squares fit method have been used to obtain dielectric parameters viz. static dielectric constant ($\epsilon_0$), dielectric constant at microwave frequency ($\epsilon_\infty$), and relaxation time ($\tau$). The Kirkwood correlation factors have also been obtained for the systems. The values of the static dielectric constant and the relaxation time increase with an increase in the percentage of alcohol in methyl acetate.

## 1. Introduction

In the study of dielectric relaxation parameters, the Kirkwood correlation factor of polar−polar liquid mixtures has a considerable significance in providing valuable information about solute−solvent interactions. The importance of measuring the dielectric constant of a liquid lies in the fact that it provides valuable information about ordering of the molecules in the liquid-state; that is, it quantifies the extent of polarization of the medium.

The dielectric constants and dielectric losses of some formates are reported by Rajyam and Murty (1973) at radio, ultrahigh, and microwave frequency, at 288, 303, and 323 K. Dielectric relaxation studies in chloral and ethyl trichloroacetate have been carried out by Srivastava and Srivastava (1975). Dielectric relaxation studies of aromatic esters in the microwave region for different temperatures are reported by Saxena et al. (1981) using the Higasi method. Dielectric properties of some organic esters have been investigated by Vasanthakumari and Sobhanadri (1988).

The aim of this paper is to report the dielectric parameters for methyl acetate + methanol, methyl acetate + ethanol, and methyl acetate + propan-1-ol mixtures for different concentrations and at the temperatures (288, 298, 308, and 318) K, over the frequency range 10 MHz to 20 GHz, using time domain reflectometry (TDR) (Puranik et al., 1991).

## 2. Experimental Section

Methyl acetate (AR grade, Sd-Fine chemicals Ltd., Bombay), methanol, ethanol (Spectroscopic grade, Fluka Chemicals, Fabrik, CH-9470 Buchs), and propan-1-ol (AR grade, E Merck, India, Ltd., Bombay) were obtained commercially and used without further purification. The solutions were prepared at different volume percentages of methyl acetate ($\phi_1$) in alcohols in steps of 10 vol %, within a 0.01% error limit.

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**Table 1. Data for the Pure Liquids Used at 298 K**

| liquid | $\epsilon_0$ | $\rho$/g·cm$^{-3}$ |
|---|---|---|
| methanol | 33.10 | 0.7868 |
| ethanol | 25.08 | 0.7851 |
| propan-1-ol | 19.37 | 0.8017 |
| methyl acetate | 6.74 | 0.9279 |

The complex permittivity spectra was studied using time domain reflectometry. The Hewlett-Packard HP 54750A sampling oscilloscope with an HP 54754 A TDR plug-in module was used. A fast rising step voltage pulse of about 39 ps rise time generated by a tunnel diode was propagated through a flexible coaxial cable. The sample was placed at the end of the coaxial line in the standard military application (SMA) coaxial cell.

The sample cell holds the liquid under consideration. The physical dimensions of the cell are very important. The impedance of the cell should be matched with that of the coaxial transmission line to which the cell is connected. If there is impedance mismatch, then unwanted reflections may disturb the wave, thereby causing some errors in measurement. The proper design of a cell includes the inner conductor and outer conductor diameters. The length of the inner conductor is called the pin length of the cell and is a very important factor. The sample length must be enough to avoid unwanted reflections. The impedance of the coaxial line is 50 Ω. The SMA cell has a 3.5 mm outer diameter and a 1.35 mm effective pin length. Since these SMA connectors have already been designed for precise 50 Ω impedance, a special design when used with high frequency is not required. The physical length of the inner conductor can be changed.

All measurements were done 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 this experiment, a time window of 5 ns was used. The reflected pulses without sample $R_1(t)$ and with sample $R_x(t)$ were digitized in 1024 points.

A temperature controller system with a water bath and a thermostat has been used to maintain the constant temperature within the accuracy limit of ±273 K. The sample cell is surrounded by a heat-insulating container through

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**Table 2. Temperature Dependent Dielectric Relaxation Parameters for Methyl Acetate (1)+Methanol(2), Methyl Acetate (1)+Ethanol(2) and Methyl Acetate (1)+Propan-1-ol (2) Binary System**

| $x_2$ | $\epsilon_0$ | $\epsilon_\infty$ | $\tau$ (ps) | $\epsilon_0$ | $\epsilon_\infty$ | $\tau$ (ps) | $x_2$ | $\epsilon_0$ | $\epsilon_\infty$ | $\tau$ (ps) | $\epsilon_0$ | $\epsilon_\infty$ | $\tau$ (ps) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Methyl Acetate + Methanol | | | | | | | | | | | | | |
| | $T = 288$ K | | | $T = 298$ K | | | | $T = 308$ K | | | $T = 318$ K | | |
| 0.0000 | 6.91(1) | 2.00 | 10.51(20) | 6.74(1) | 1.98 | 9.41(20) | 0.0000 | 6.49(1) | 1.94 | 8.87(19) | 6.23(1) | 1.86 | 7.26(25) |
| 0.1789 | 11.82(2) | 2.02 | 13.90(23) | 10.66(2) | 1.96 | 12.36(24) | 0.1789 | 9.98(1) | 1.95 | 11.98(22) | 9.47(2) | 1.84 | 9.87(28) |
| 0.3290 | 15.49(3) | 2.10 | 18.81(30) | 15.01(3) | 2.00 | 17.03(30) | 0.3290 | 13.58(3) | 1.96 | 14.66(28) | 12.91(3) | 1.87 | 13.17(34) |
| 0.4567 | 17.82(5) | 2.12 | 24.64(41) | 17.50(5) | 2.00 | 21.89(36) | 0.4567 | 15.46(4) | 1.97 | 19.05(35) | 14.47(4) | 1.99 | 16.90(38) |
| 0.5666 | 20.67(7) | 2.23 | 28.41(47) | 20.12(6) | 2.08 | 26.03(44) | 0.5666 | 18.09(6) | 1.93 | 22.72(42) | 16.82(6) | 2.11 | 19.41(47) |
| 0.6623 | 23.31(9) | 2.23 | 33.12(56) | 23.02(8) | 2.19 | 30.14(49) | 0.6623 | 21.19(8) | 1.95 | 26.32(48) | 19.19(8) | 2.13 | 22.88(53) |
| 0.7463 | 26.53(10) | 2.48 | 36.84(55) | 25.64(1) | 2.21 | 33.34(53) | 0.7463 | 22.70(8) | 2.00 | 29.46(50) | 22.08(9) | 2.21 | 25.46(54) |
| 0.8207 | 29.86(13) | 2.82 | 41.83(63) | 29.17(12) | 2.49 | 38.80(60) | 0.8207 | 25.38(12) | 2.43 | 33.24(53) | 24.41(11) | 2.24 | 28.70(61) |
| 0.8869 | 32.81(17) | 2.86 | 50.18(76) | 30.24(16) | 2.67 | 46.16(70) | 0.8869 | 28.00(14) | 2.44 | 38.78(60) | 26.05(15) | 2.33 | 32.74(68) |
| 0.9464 | 33.10(17) | 2.96 | 54.37(73) | 31.98(16) | 2.80 | 48.38(70) | 0.9464 | 30.67(14) | 2.52 | 39.32(62) | 27.57(15) | 2.37 | 33.81(68) |
| 1.0000 | 34.00(18) | 3.02 | 60.00(76) | 33.10(17) | 2.83 | 52.86(71) | 1.0000 | 32.12(15) | 2.57 | 41.87(64) | 28.44(16) | 2.40 | 35.42(70) |
| Methyl Acetate + Ethanol | | | | | | | | | | | | | |
| | $T = 288$ K | | | $T = 298$ K | | | | $T = 308$ K | | | $T = 318$ K | | |
| 0.0000 | 6.91(1) | 2.00 | 10.51(20) | 6.74(1) | 1.98 | 9.41(20) | 0.0000 | 6.49(1) | 1.94 | 8.87(19) | 6.23(1) | 1.86 | 7.26(25) |
| 0.1313 | 9.74(1) | 2.03 | 16.84(24) | 9.04(1) | 1.99 | 15.38(21) | 0.1313 | 8.58(1) | 1.98 | 14.30(19) | 8.04(1) | 1.94 | 12.40(23) |
| 0.2538 | 11.32(2) | 2.18 | 24.27(35) | 11.09(2) | 2.09 | 21.92(29) | 0.2538 | 10.81(1) | 2.01 | 19.89(22) | 9.74(1) | 1.99 | 17.40(23) |
| 0.3683 | 12.90(4) | 2.43 | 35.86(58) | 12.62(3) | 2.24 | 31.57(42) | 0.3683 | 12.34(2) | 2.13 | 28.15(33) | 11.33(2) | 2.01 | 23.80(27) |
| 0.4756 | 14.41(6) | 2.63 | 46.81(75) | 14.06(5) | 2.43 | 40.79(56) | 0.4756 | 13.68(4) | 2.34 | 37.09(50) | 12.18(3) | 2.07 | 30.25(36) |
| 0.5763 | 15.90(4) | 2.43 | 57.86(58) | 15.04(6) | 2.65 | 54.71(73) | 0.5763 | 14.65(5) | 2.47 | 48.01(57) | 14.04(4) | 2.16 | 38.17(46) |
| 0.6711 | 17.41(6) | 2.63 | 63.81(75) | 17.15(7) | 2.75 | 60.70(81) | 0.6711 | 17.03(6) | 2.61 | 58.41(68) | 15.47(5) | 2.31 | 46.56(55) |
| 0.7604 | 18.34(7) | 2.82 | 81.72(91) | 18.03(7) | 2.82 | 78.92(88) | 0.7604 | 17.44(6) | 2.64 | 68.41(71) | 16.12(5) | 2.31 | 54.17(63) |
| 0.8448 | 21.03(8) | 2.95 | 96.04(10) | 20.67(7) | 2.88 | 92.21(92) | 0.8448 | 20.03(6) | 2.70 | 83.41(77) | 18.05(6) | 2.41 | 65.33(68) |
| 0.9145 | 23.97(8) | 3.03 | 123.93(10) | 22.58(7) | 2.87 | 114.89(93) | 0.9145 | 22.09(6) | 2.77 | 103.39(81) | 20.22(7) | 2.53 | 79.41(77) |
| 1.0000 | 26.31(7) | 3.02 | 159.86(10) | 25.08(6) | 2.83 | 130.37(94) | 1.0000 | 23.65(6) | 2.69 | 110.78(83) | 21.31(7) | 2.50 | 87.79(77) |
| Methyl Acetate + Propan-1-ol | | | | | | | | | | | | | |
| | $T = 288$ K | | | $T = 298$ K | | | | $T = 308$ K | | | $T = 318$ K | | |
| 0.0000 | 6.91(1) | 2.00 | 10.51(20) | 6.74(1) | 1.98 | 9.41(20) | 0.0000 | 6.49(1) | 1.94 | 8.87(19) | 6.23(1) | 1.86 | 7.26(24) |
| 0.1055 | 9.99(2) | 2.00 | 14.74(39) | 9.78(1) | 2.00 | 12.96(20) | 0.1055 | 8.89(1) | 2.00 | 11.86(21) | 8.57(1) | 1.96 | 10.00(20) |
| 0.2097 | 10.65(4) | 2.20 | 22.06(61) | 10.48(3) | 2.00 | 19.15(37) | 0.2097 | 10.13(2) | 2.00 | 16.68(32) | 10.00(1) | 2.00 | 14.21(22) |
| 0.3127 | 11.69(6) | 2.52 | 34.88(82) | 11.46(5) | 2.07 | 29.87(58) | 0.3127 | 11.03(3) | 2.07 | 24.86(44) | 10.32(3) | 2.00 | 20.89(35) |
| 0.4144 | 12.71(8) | 2.83 | 48.85(11) | 12.34(7) | 2.17 | 42.27(88) | 0.4144 | 12.80(5) | 2.26 | 33.26(60) | 12.21(3) | 2.01 | 26.88(43) |
| 0.5149 | 13.32(8) | 2.96 | 62.17(13) | 13.07(8) | 2.19 | 51.85(10) | 0.5149 | 12.95(6) | 2.28 | 40.31(75) | 12.23(4) | 2.16 | 33.06(54) |
| 0.6142 | 15.87(9) | 2.97 | 88.31(17) | 15.01(9) | 2.20 | 75.54(14) | 0.6142 | 14.95(8) | 2.30 | 57.05(99) | 13.86(6) | 2.22 | 45.47(73) |
| 0.7124 | 16.10(9) | 2.98 | 115.02(18) | 16.98(10) | 2.24 | 94.63(15) | 0.7124 | 15.10(8) | 2.39 | 72.39(11) | 14.88(7) | 2.35 | 56.35(88) |
| 0.8094 | 17.84(7) | 3.00 | 161.77(17) | 17.93(6) | 2.69 | 141.76(14) | 0.8094 | 16.94(8) | 2.45 | 103.86(13) | 15.42(8) | 2.42 | 79.09(10) |
| 0.9052 | 19.90(6) | 3.00 | 237.45(18) | 18.50(7) | 2.81 | 214.26(17) | 0.9052 | 17.27(8) | 2.56 | 142.58(14) | 15.95(8) | 2.43 | 100.41(12) |
| 1.0000 | 20.82(8) | 3.02 | 358.08(23) | 19.37(7) | 2.83 | 280.68(19) | 1.0000 | 17.91(8) | 2.69 | 195.20(16) | 16.09(8) | 2.50 | 130.70(13) |

[a] Numbers in parentheses indicate the error in the values. For example, 6.91(1) means 6.91 ± 0.01.

which the water of constant temperature using a temperature controller system is circulated. The temperature at the cell is checked using the electronic thermometer.

## 3. 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 (Samulon, 1951; Shannon, 1949) 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, $\omega$ is angular frequency, and $d$ is the effective pin length.

The complex permittivity spectra $\epsilon^*(\omega)$ were obtained from reflection coefficient spectra $\rho^*(\omega)$ by using the bilinear calibration method (Cole et al., 1989). Methyl acetate and the respective alcohol were used as calibrating liquids.

The experimental values of $\epsilon^*$ are fitted with the Debye expression (Havriliak and Negami, 1966; Cole and Cole, 1941; Davidson and Cole, 1950)

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

with $\epsilon_0$, $\epsilon_\infty$, and $\tau$ as fitting parameters. A nonlinear least-squares fit method (Bevington, 1969) was used to determine the values of dielectric parameters.

## 4. Results and Discussion

The density and $\epsilon_0$ values of the pure liquids used are given in Table 1. The static dielectric constant and relaxation time obtained by the least-squares fit method are listed in Table 2. The values of the static dielectric constant and the relaxation time increase as the percent of methyl acetate in alcohol increases, for all temperatures, for all the systems.

The Kirkwood correlation factor $g$ (Frolhich, 1949) is also a parameter for getting information regarding the orientation of the electric dipoles in polar liquids. The $g$ for a pure liquid may be obtained by the expression

$$\frac{4\Pi N\mu^2\rho}{9kTM}g = \frac{(\epsilon_0 - \epsilon_\infty)(2\epsilon_0 + \epsilon_\infty)}{\epsilon_0(\epsilon_\infty + 2)^2} \quad (3)$$

where $\mu$ is the dipole moment in the gas phase, $\rho$ is the density at temperature $T$, $M$ is the molecular weight, $k$ is the Boltzmann constant, and $N$ is Avogadro's number.

The corresponding equation for the mixtures is not available in the literature. However, for the mixture of two polar liquids, say A and B, eq 3 is modified (Kumbharkhane et al., 1993) using different assumptions:

**Table 3. Values of $g^{eff}$ and $g_f$ for the Methyl Acetate (1) + Methanol (2), Methyl Acetate (1) + Ethanol (2), and Methyl Acetate (1) + Propan-1-ol (2) Binary Systems**

| | $g^{eff}$ | | | | $g_f$ | | | |
|---|---|---|---|---|---|---|---|---|
| $x_2$ | 288 K | 298 K | 308 K | 318 K | 288 K | 298 K | 308 K | 318 K |
| Methyl Acetate + Methanol | | | | | | | | |
| 1.0000 | 2.66 | 2.68 | 2.69 | 2.44 | 1.00 | 1.00 | 1.00 | 1.00 |
| 0.9464 | 2.73 | 2.72 | 2.70 | 2.49 | 1.06 | 1.05 | 1.04 | 1.05 |
| 0.8869 | 2.85 | 2.71 | 2.59 | 2.48 | 1.16 | 1.09 | 1.04 | 1.09 |
| 0.8207 | 2.74 | 2.77 | 2.47 | 2.45 | 1.16 | 1.17 | 1.04 | 1.13 |
| 0.7463 | 2.58 | 2.57 | 2.34 | 2.35 | 1.16 | 1.15 | 1.04 | 1.14 |
| 0.6623 | 2.40 | 2.45 | 2.32 | 2.16 | 1.15 | 1.17 | 1.11 | 1.12 |
| 0.5666 | 2.27 | 2.28 | 2.10 | 2.01 | 1.18 | 1.18 | 1.09 | 1.13 |
| 0.4567 | 2.08 | 2.11 | 1.91 | 1.84 | 1.21 | 1.22 | 1.11 | 1.14 |
| 0.3290 | 1.94 | 1.94 | 1.80 | 1.75 | 1.30 | 1.29 | 1.20 | 1.25 |
| 0.1789 | 1.57 | 1.45 | 1.39 | 1.35 | 1.28 | 1.17 | 1.13 | 1.15 |
| 0.0000 | 0.91 | 0.91 | 0.90 | 0.88 | 1.00 | 1.00 | 1.00 | 1.00 |
| Methyl Acetate + Ethanol | | | | | | | | |
| 1.0000 | 2.94 | 3.02 | 2.81 | 2.60 | 1.00 | 1.00 | 1.00 | 1.00 |
| 0.9245 | 2.75 | 2.67 | 2.70 | 2.54 | 0.98 | 0.93 | 1.01 | 1.02 |
| 0.8448 | 2.47 | 2.51 | 2.51 | 2.32 | 0.94 | 0.93 | 0.99 | 0.99 |
| 0.7604 | 2.20 | 2.24 | 2.23 | 2.12 | 0.89 | 0.88 | 0.94 | 0.96 |
| 0.6711 | 2.15 | 2.19 | 2.25 | 2.09 | 0.94 | 0.93 | 1.02 | 1.02 |
| 0.5763 | 2.02 | 1.96 | 1.97 | 1.94 | 0.96 | 0.91 | 0.97 | 1.03 |
| 0.4756 | 1.87 | 1.89 | 1.89 | 1.72 | 0.99 | 0.97 | 1.03 | 1.00 |
| 0.3683 | 1.72 | 1.74 | 1.75 | 1.64 | 1.02 | 1.01 | 1.08 | 1.07 |
| 0.2538 | 1.54 | 1.56 | 1.56 | 1.43 | 1.06 | 1.06 | 1.11 | 1.07 |
| 0.1313 | 1.35 | 1.28 | 1.24 | 1.18 | 1.13 | 1.06 | 1.06 | 1.05 |
| 0.0000 | 0.92 | 0.93 | 0.91 | 0.89 | 1.00 | 1.00 | 1.00 | 1.00 |
| Methyl Acetate + Propan-1-ol | | | | | | | | |
| 1.0000 | 2.95 | 2.83 | 2.69 | 2.47 | 1.00 | 1.00 | 1.00 | 1.00 |
| 0.9052 | 2.81 | 2.69 | 2.58 | 2.44 | 1.03 | 1.02 | 1.03 | 1.06 |
| 0.8094 | 2.50 | 2.60 | 2.52 | 2.35 | 0.99 | 1.07 | 1.09 | 1.10 |
| 0.7124 | 2.23 | 2.45 | 2.23 | 2.26 | 0.97 | 1.10 | 1.05 | 1.15 |
| 0.6142 | 2.20 | 2.14 | 2.20 | 2.09 | 1.05 | 1.06 | 1.14 | 1.16 |
| 0.5149 | 1.81 | 1.83 | 1.88 | 1.82 | 0.96 | 1.01 | 1.07 | 1.11 |
| 0.4144 | 1.72 | 1.72 | 1.85 | 1.81 | 1.03 | 1.06 | 1.18 | 1.24 |
| 0.3127 | 1.56 | 1.58 | 1.56 | 1.50 | 1.07 | 1.11 | 1.14 | 1.15 |
| 0.2097 | 1.40 | 1.43 | 1.42 | 1.44 | 1.12 | 1.16 | 1.19 | 1.27 |
| 0.1055 | 1.30 | 1.32 | 1.22 | 1.20 | 1.25 | 1.28 | 1.21 | 1.24 |
| 0.0000 | 0.83 | 0.83 | 0.82 | 0.80 | 1.00 | 1.00 | 1.00 | 1.00 |

(1) Assume that, for the mixture, $g^{eff}$ is the effective correlation factor in the mixture. The Kirkwood equation for the mixture may be expressed as

$$\frac{4\Pi N}{9kT}\left(\frac{\mu_1^2\rho_1}{M_1}\phi_1 + \frac{\mu_2^2\rho_2}{M_2}\phi_2\right)g^{eff} = \frac{(\epsilon_{0m} - \epsilon_{\infty m})(2\epsilon_{0m} + \epsilon_{\infty m})}{\epsilon_{0m}(\epsilon_{\infty m} + 2)^2} \quad (4)$$

where $g^{eff}$ is the effective Kirkwood correlation factor for a binary mixture, with $\phi_1$ and $\phi_2$ as the volume fractions of liquids 1 and 2, respectively.

(2) Assume that the correlation factors for molecules 1 and 2 are affected by the same amount $g_f$ in the mixture; under this assumption the Kirkwood equation for the mixture is

$$\frac{4\Pi N}{9kT}\left(\frac{\mu_1^2\rho_1 g_1}{M_1}\phi_1 + \frac{\mu_2^2\rho_2 g_2}{M_1}\phi_2\right)g_f = \frac{(\epsilon_{0m} - \epsilon_{\infty m})(2\epsilon_{0m} + \epsilon_{\infty m})}{\epsilon_{0m}(\epsilon_{\infty m} + 2)^2} \quad (5)$$

where $g_f$ is the corrective Kirkwood correlation factor.

In eq 4, the values of $g^{eff}$ will change from $g_1$ to $g_2$, as the concentration of molecule 2 will increase from 0% to 100%. In eq 5, the values of $g_f$ will remain close to unity if there is no interaction between 1 and 2. The deviation from unity will indicate the interaction between 1 and 2.

The calculated values of $g_f$ and $g^{eff}$ are tabulated in Table 3. The $g^{eff}$ values decrease with an increase in the percentage of methyl acetate in ethanol and propan-1-ol. But for the methyl acetate + methanol system, no systematic change in $g^{eff}$ is observed. The $g_f$ values are much deviated from unity, indicating stronger intermolecular interaction in solute and solvent, for all the systems.

## 5. Conclusion

The dielectric constants, the relaxation times, and the Kirkwood correlation factors for methyl acetate + alcohol mixtures have been reported. The dielectric constant and relaxation time values increase with an increase in the alcohol concentration in methyl acetate and decrease with temperature. The Kirkwood correlation factor deviates from unity, indicating stronger intermolecular interaction in the systems.

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