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# RESEARCH ARTICLE

## DIELECTRIC PROPERTIES AND FLUID STRUCTURE OF BINARY MIXTURES OF DIMETHYL PHTHALATE AND TRIETHYLAMINE AT DIFFERENT TEMPERATURES

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## ABSTRACT

Interactions in the binary mixture of dimethyl phthalate with triethylamine have been studied through the orientation of dipoles using dielectric polarization process. This vital information could be obtained from dielectric measurements by virtue of the influence of local order and the overall dipole moment. The information regarding orientation of electric dipoles in polar liquids was obtained from Kirkwood correlation factor, effective correlation factor, and corrective correlation factor. The Bruggeman parameter determination confirms the molecular interaction between the constituents of the binary mixture and thermo dynamical excess parameters were also measured for analysis for three different temperatures (303K, 313K and 323K).

***Key words:*** Dielectric constant, refractive index, Kirkwood correlation factor, Bruggeman factor, thermo dynamical properties.

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## INTRODUCTION

Thermo dynamical physical properties of liquid systems such as dielectric constant, optical permittivity, Kirkwood correlation factor, effective correlation factor are related to molecular interactions present in binary liquid mixtures. Dimethyl phthalate (DBP) is a commonly used plasticizer. It is used as an additive to adhesives. It is soluble in various organic solvents, e.g. in alcohol, ether and benzene. The variation of these physical properties with composition provides important data about intermolecular interactions and the structure. Triethylamine is used in mosquito control labs to anesthetize mosquitoes. By studying the properties of these mixtures, we can get valuable information that may be helpful in many ways. Moreover, these compounds are strongly associated in solution because of dipole – dipole interaction and hydrogen bonding. The main aim of this study is to find out the intermolecular interaction between Dimethyl phthalate and triethylamine molecules through dielectric study at different temperatures 303K, 313K and 323K. Several factors such as molecular structure, temperature, solvent and solute are the key factors that influence the molecular association. Dielectric studies and hydrogen bonding are the key aspects of binary mixture for the determination of structure analysis (Thenappan and Sankar, 2006). The analysis can be studied only through dielectric behaviour. The study of Kirkwood correlation factor has a considerable significance in providing valuable information about the interaction between the like molecules.

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Many workers had tried to find molecular structure and interaction of alcohols (Dharmalingam and Ramachandran, 2006; Parthipana *et al.,* 2008) and few tried using amines (Thenappan and Subramainian, 2001; Narwade *et al.,* 2011; Liakath Ali Khan *et al.,* 2007) as one of the constituent components of the binary mixture. Dielectric constants, refractive indices and polarizations of binary mixture of polar molecules were estimated by several researchers (Vishwar *et al.,* 2008; Indira and Thenappan, 2010). Many researchers (Parthipan *et al.,* 2008; Partiban *et al.,* 2008; Sengwa *et al.,* 2008) used modified expressions for the study of dipolar excess thermodynamic properties of associated liquids in an inert solvent in the study. This work mainly concentrates to calculate the dipolar excess free energy using the values of the linear correction factor 'g ' for the system of and dimethyl phthalate and triethylamine at 303K, 313K and 323K.

## MATERIALS AND METHODS

The dielectric constants were measured at 1kHz using VLCR-7 meter supplied by Vasavi Electronics, India. Using a water circulating thermostat the temperature was maintained at 303K, 313K and 323K. The refractive indices were measured using an Abbe's refract meter. Densities were determined by using a 10ml specific gravity bottle and a K-Roy microbalance. The chemicals were obtained from SD Fine-Chem Limited, Mumbai (India). The liquids were purified by standard methods.

**Theory:** The Kirkwood correlation (Kirkwood, 1939) factor (g) is a parameter affording information regarding orientation

of electric dipoles in polar liquids. The modified Kirkwood-Frohlich [13] correlation factor (g) for the pure liquid is given by the expression.

$$\frac{4\pi N_A \mu^2 \rho g}{9KTM} = \frac{(\varepsilon_0-\varepsilon_\infty)(2\varepsilon+\varepsilon_\infty)}{\varepsilon_0(\varepsilon_\infty+2)^2} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (1)$$

where μ the dipole moment in the gas phase, ρ is the density at a temperature T, M is the molecular weight, $\varepsilon_0$ is the dielectric constant of pure liquid, $\varepsilon_\infty$ is the dielectric constant of pure liquid at optical frequency(square of the refractive index), K is the Boltzmann constant and $N_A$ is the Avogadro's number. For a mixture of two polar liquids say A and B the equation is modified by Kumbharkhane *et al.* [14] using some assumptions. Assume that 'g' for the mixture is expressed by an effective average correlation factor ($g^{eff}$) such that Kirkwood equation for the mixture can be expressed as

$$\frac{4\pi N_A}{9KT}\left(\frac{\mu_A^2\rho_A}{M_A}X_A + \frac{\mu_B^2\rho_B}{M_B}X_B\right)g^{eff} = \frac{(\varepsilon_{0m}-\varepsilon_{\infty m})(2\varepsilon_{0m}+\varepsilon_{\infty m})}{\varepsilon_{0m}(\varepsilon_{\infty m}+2)^2} \quad \ldots\ldots (2)$$

Where $X_A$ and $X_B$ are the volume fractions and $\mu_A$ and $\mu_B$ are the dipole moment of the liquids A and B respectively and $g^{eff}$ is the effective Kirkwood correlation factor for a binary mixture. The value of $g^{eff}$ in equation (2) will change from $g_A$ to $g_B$ as the fraction of component B increases from zero to unity. Another way to visualize the variation in Kirkwood correlation factor is to assume that the correlation factor for molecules A and B in a mixture contribute to effective 'g' in proportion to their pure liquid values $g_A$ and $g_B$. Under this assumption Kirkwood equation for the mixture can be written as

$$\frac{4\pi N_A}{9KT}\left(\frac{\mu_A^2\rho_A g_A}{M_A}X_A + \frac{\mu_B^2\rho_B g_B}{M_B}X_B\right)g_f = \frac{(\varepsilon_{0m}-\varepsilon_{\infty m})(2\varepsilon_{0m}+\varepsilon_{\infty m})}{\varepsilon_{0m}(\varepsilon_{\infty m}+2)^2} \quad \ldots (3)$$

Where $g_f$ is the correlation factor for the mixture. In equation (3), $g_f$ is unity for pure liquids and will remain close to unity if there is no interaction between A and B.

The Bruggeman equation for a binary mixture [15] is given by the expression

$$f_B = \left(\frac{\varepsilon_{0m}-\varepsilon_{0B}}{\varepsilon_{0A}-\varepsilon_{0B}}\right)\left(\frac{\varepsilon_{0A}}{\varepsilon_{0m}}\right)^{\frac{1}{3}} = \left(1-\phi_B\right) \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots (4)$$

Where $\varepsilon_{0A}$, $\varepsilon_{0B}$ and $\varepsilon_{0m}$ are the dielectric constants of the liquids A, B and mixture respectively. $f_B$ is the Bruggeman dielectric factor, $\phi_B$ is the volume fraction of the alcohol. It is seen that if there is no interaction, then variation of $f_B$ and $\phi_B$ will be linear. For a non linear variation of $f_B$ with $\phi_B$, the Bruggeman factor is modified as

$$f_B = 1 - \left[a-(a-1)\phi_B\right]\phi_B \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots (5)$$

Where 'a' is the Bruggeman parameter and it is assumed that, the volume fraction of the solvent ($\phi_B$) in the mixture is modified by a factor of [a-(a-1) $\phi_B$]$\phi_B$. This modification of volume may be due to the structural rearrangement of solvent molecules in the mixture. The value of 'a' contains information regarding the change in the orientation of the solvent molecules in the mixture. The original Bruggeman equation assume that the orientation of the solvent molecules does not change (i.e.) there is no interaction between solute and solvent molecules. This is not true for non-polar solvents. Studying these excess dielectric properties, it can be used to access the useful information regarding the structural changes in binary mixture. Let A and B be two molecular systems with measurable macroscopic properties $P_A$ and $P_B$. If we prepare the mixture of A and B having mole fraction $X_A$ and $X_B$ = (1-$X_A$), the excess macroscopic property ($P^E$) corresponding to the mixture is defined as

$$P^E = P_{AB} - (P_A X_A + P_B X_B) \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots (6)$$

Where $P_{AB}$ is the measured value of property P for the mixture. The value of $P^E$ provides information regarding interaction between A and B. $P^E > 0$ indicates that interaction between A and B leads to increase in the value of property P. Similarly, $P^E < 0$ indicates decrease in macroscopic property P. In the present work, excess dielectric properties are determined corresponding to static permittivity and molar volume. The excess permittivity ( $\varepsilon^E$ ) is defined as [16]

$$\varepsilon^E = (\varepsilon_0-\varepsilon_\infty)_m - [(\varepsilon_0-\varepsilon_\infty)_A X_A + (\varepsilon_0-\varepsilon_\infty)_B X_B] \quad \ldots\ldots (7)$$

Where X is the mole fraction and the subscripts m, A and B represent mixture, solvent and solute respectively. The excess permittivity $\varepsilon^E$may provide qualitative information about the multimer formation in mixtures as follows.

1. $\varepsilon^E = 0$ indicates that the liquids A and B do not interact at all.
2. $\varepsilon^E < 0$ indicates that the liquids A and B interact in such a way that the total effective dipole moment gets reduced. The liquids A and B may form multimers leading to less effective dipoles. In general, the negative excess permittivity indicates the formation of multimers in the binary mixtures.
3. $\varepsilon^E > 0$ indicates that the liquids A and B interact in such a way that the total effective dipole moment increases. There is a tendency to form multimers, dipoles aligned in parallel direction.

The excess molar volume may be defined as

$$V^E = V_m - V_A X_A + V_B X_B \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots (8)$$

If $V^E$ is positive then it refers to the breaking of H-bonds and hence demolition of multimers. Negative values of $V^E$ refer to the association due to dipole–dipole interaction. The excess Helmholtz free energy ΔF which is almost equal to the Gibb's free energy ΔG for a condensed matter [16] is given as

$$\Delta F^E = \frac{N_A}{2}\left[\sum_{r=A,B} X_r\mu_r^2(R_{fr}-R_{fr0})\right] + \frac{N_A}{2}\left[\sum_{r=A,B} X_r^2\mu_r^2(g_{rr}-1)(R_{fr}-R_{fr0})\right]$$
$$+\frac{N_A}{2}\left[X_A X_B \mu_A \mu_B (g_{AB}-1)(R_{fA}+R_{fB}-R_{fA0}-R_{fB0})\right]$$
$$= \Delta F_0^E + \Delta F_{rr}^E + \Delta F_{AB}^E \quad (5) \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots (9)$$

Where

$$R_{fr} = \frac{8\pi N_A}{9V_r}\left[\frac{(\varepsilon_{0r}-1)(\varepsilon_{\infty r}+2)}{(2\varepsilon_{0r}+\varepsilon_{\infty r})}\right] \quad \ldots\ldots\ldots\ldots\ldots\ldots (10)$$

$$R_{fr0} = \frac{8\pi N_A}{9V_r}\left[\frac{(\varepsilon_{0m}-1)(\varepsilon_{\infty r}+2)}{(2\varepsilon_{0m}+\varepsilon_{\infty r})}\right] \quad \ldots\ldots\ldots\ldots\ldots\ldots (11)$$

where V is the molar volume of the components and $\varepsilon_{0r}$ is the dielectric constant of the pure liquids.

**Table 1. Dielectric constant, Dielectric constant at optical frequency, effective correlation factor, Kirkwood correlation factor, Excess permittivity and Bruggeman factor for the system of dimethyl phthalate and triethylamine at three different temperatures**

| Temperature K | $X_2$ | □□□□□□ | □□□□□□□□ | $g^{eff}$ | $g_f$ | $\varepsilon^E$ | $f_B$ |
|---|---|---|---|---|---|---|---|
| 303 | 0.0 | 8.5230 | 2.2127 | 1.0440 | 1.0000 | 0.0000 | 1.0000 |
| | 0.1 | 7.7640 | 2.1954 | 1.0260 | 0.9860 | -0.1620 | 0.9020 |
| | 0.2 | 7.0320 | 2.1860 | 1.0020 | 0.9670 | -0.3040 | 0.8030 |
| | 0.3 | 6.0770 | 2.1688 | 0.9270 | 0.8980 | -0.6630 | 0.6670 |
| | 0.4 | 5.3750 | 2.1500 | 0.8890 | 0.8670 | -0.7650 | 0.5590 |
| | 0.5 | 4.9140 | 2.1313 | 0.9030 | 0.8870 | -0.6280 | 0.4850 |
| | 0.6 | 4.6860 | 2.1057 | 1.0040 | 0.9970 | -0.2500 | 0.4460 |
| | 0.7 | 4.4590 | 2.0820 | 1.1440 | 1.1550 | 0.1260 | 0.4070 |
| | 0.8 | 4.0090 | 2.0607 | 1.2300 | 1.2720 | 0.2780 | 0.3260 |
| | 0.9 | 3.1240 | 2.0124 | 1.0410 | 1.1260 | 0.0210 | 0.1500 |
| | 1.0 | 2.4740 | 1.9636 | 0.8310 | 1.0000 | 0.0000 | 0.0000 |
| 313 | 0.0 | 8.4379 | 2.1960 | 1.0000 | 1.0000 | 0.0000 | 1.0000 |
| | 0.1 | 7.6787 | 2.1833 | 0.9838 | 0.9930 | -0.1665 | 0.9024 |
| | 0.2 | 6.9468 | 2.1771 | 0.9625 | 0.9830 | -0.3123 | 0.8039 |
| | 0.3 | 5.9915 | 2.1641 | 0.8910 | 0.9680 | -0.7747 | 0.6676 |
| | 0.4 | 5.2899 | 2.1436 | 0.8596 | 0.9510 | -0.7758 | 0.5603 |
| | 0.5 | 4.8291 | 2.1237 | 0.8809 | 0.9370 | -0.6368 | 0.5286 |
| | 0.6 | 4.6007 | 2.0964 | 0.9945 | 0.9390 | -0.2579 | 0.4476 |
| | 0.7 | 4.3737 | 2.0704 | 1.1602 | 0.9560 | 0.1210 | 0.4084 |
| | 0.8 | 3.9237 | 2.0423 | 1.2942 | 0.9640 | 0.2789 | 0.3275 |
| | 0.9 | 3.0392 | 1.9932 | 1.1454 | 0.9540 | 0.0235 | 0.1510 |
| | 1.0 | 2.3891 | 1.9466 | 1.0001 | 1.0000 | 0.0000 | 0.0000 |
| 323 | 0.0 | 8.3528 | 2.1827 | 1.1042 | 1.0000 | 0.0000 | 1.0000 |
| | 0.1 | 7.5936 | 2.1739 | 1.0772 | 0.9815 | -0.1709 | 0.9027 |
| | 0.2 | 6.8617 | 2.1656 | 1.0474 | 0.9614 | -0.3151 | 0.8045 |
| | 0.3 | 5.9064 | 2.1541 | 0.9587 | 0.8881 | -0.6796 | 0.6685 |
| | 0.4 | 5.2048 | 2.1354 | 0.9120 | 0.8547 | -0.7830 | 0.5615 |
| | 0.5 | 4.7440 | 2.1176 | 0.9189 | 0.8746 | -0.6466 | 0.4871 |
| | 0.6 | 4.5156 | 2.0883 | 1.0205 | 0.9921 | -0.3662 | 0.4488 |
| | 0.7 | 4.2886 | 2.0615 | 1.1611 | 1.1644 | 0.1130 | 0.4097 |
| | 0.8 | 3.8386 | 2.0266 | 1.2474 | 1.3153 | 0.2773 | 0.3875 |
| | 0.9 | 2.9541 | 1.9763 | 1.0033 | 1.1629 | 0.0126 | 0.1520 |
| | 1.0 | 2.3040 | 1.9282 | 0.6738 | 0.9999 | 0.0000 | 0.0000 |

## RESULTS AND DISCUSSION

Table 1 shows the static permittivity ($\varepsilon_0$), the static permittivity at high frequency ($\varepsilon_\infty$) normally calculated from the square of the refractive index for sodium line, effective correlation factor ($g^{eff}$) and Kirkwood correlation factor ($g_f$), excess permittivity ($\varepsilon^E$), Bruggemann factor ($f_b$) for the mixture of dimethyl phthalate with triethylamine for three different temperatures 303K, 313K and 323K. In Table 2, the values of excess dipolar free energy ($\Delta F_0{}^E$) due to long range electrostatic interaction, $\Delta F_{rr}{}^E$ the excess dipolar energy due to short range interaction between identical molecules, $\Delta F_{AB}{}^E$ the excess dipolar energy due to short range interaction between dissimilar molecules and the total excess Helmholtz free energy of mixing $\Delta F^E$ are listed for the binary mixture of dimethyl phthalate and triethylamine for above the three temperatures. For the mixture of dimethyl phthalate with triethylamine, the values of $g^{eff}$ for pure phthalate is slightly greater than unity at all temperature indicating the molecular dipoles have parallel orientation among themselves. The $g^{eff}$ value is greater than unity in amine rich region is the indication of the parallel alignment between the dipoles. On mixing triethylamine with dimethyl phthalate, the intermolecular interaction between phthalate and amine increases as the concentration of amine increases. Similar behavior is observed at all temperatures. Similar observations are carried out for the mixture of ester and acid by Thennappan *et al.* The values of $g_f$ are close to unity for low concentrations of triethylamine indicating weak interaction between the compounds. When the concentration increases i.e., for 70 % to 90% concentration of triethylamine, the value of $g_f$ deviates more from unity indicating strong heterogeneous intermolecular interactions.

As the temperature increases the values of $g_f$ are found to be greater than unity for high temperature 323K indicating that the effective dipoles in the mixture are larger than the average of those in the pure compounds (Pawar *et al.,* 2005). This may be due to the formation of multimers between the components. Figure 2 shows the plot of excess permittivity with mole fraction. The excess permittivity in the mixture of dimethyl phthalate and triethlyamine is such that in phthalate rich region it is negative, and is positive in the amine rich region. On mixing amine with phthalate, a decrease in the number of dipoles takes place and it reaches minimum for $X_2 = 0.4$ mole fraction of amine. This shows that, the hetero interaction between the -NH group of amine and hydrogen molecules of phthalate are at a maximum at about $X_2 = 0.4$. On further increasing the amines molecules, the number of dipoles increases. This similar behavior is observed for all temperatures studied. The plot of Bruggeman factor $f_B$ versus volume fraction of triethylamine is shown in figure 4. The plots for $f_B$ also deviate from linearity, which shows the existence of intermolecular interactions between the phthalate and amine. The different components of thermodynamic parameters $\Delta F^E$ such as $\Delta F_0{}^E$, $\Delta F_{rr}{}^E$ and $\Delta F_{AB}{}^E$ are given in Table 2 for the binary mixture of dimethyl phthalate and triethylamine. It is found that the values of $\Delta F_0{}^E$ for system this system is positive for all the temperatures studied. As the concentration increases, the magnitude of this value increases indicating the existence of long range dipole-dipole interaction. This long range dipole – dipole interaction is maximum in the 1:1 combination region. The values $\Delta F_{rr}{}^E$ predict the information of the short range interaction between similar molecules. The values obtained are very low, which indicates that, the short range interaction through hydrogen bonding is very weak.

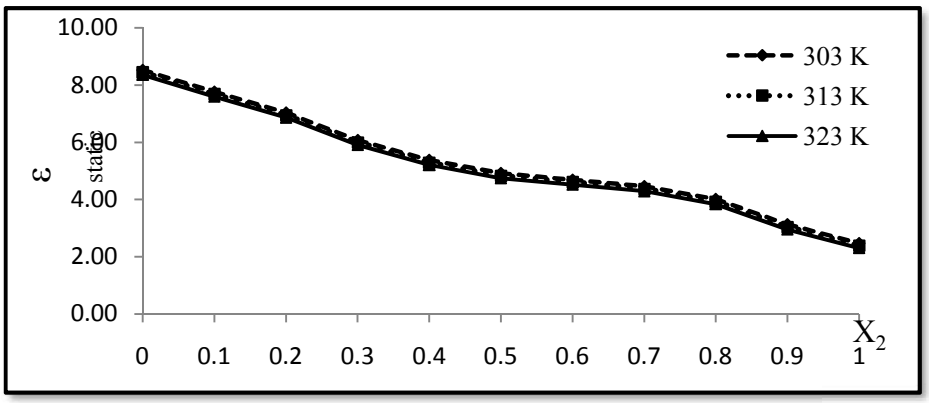


**Figure 1 : Plot of the static permittivity with mole fraction of dimethyl phthalate + triethylamine**

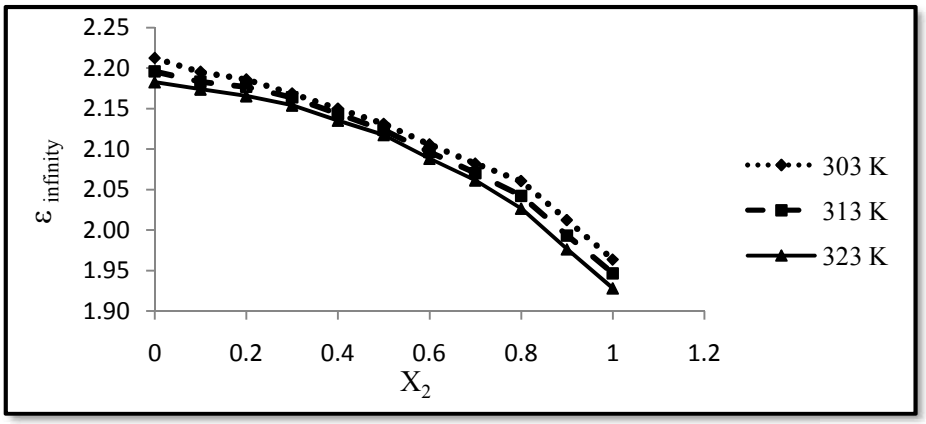


**Figure 2 : Plot of optical permittivity with mole fraction of dimethyl phthalate + triethyl amine**

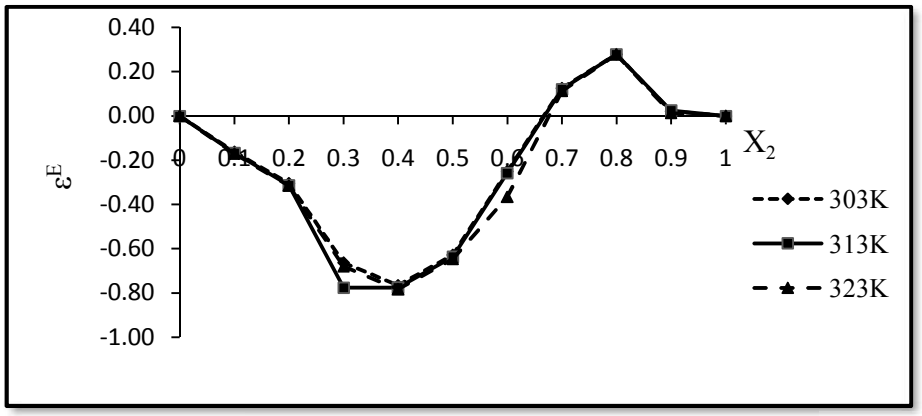


**Figure 3 : Plot of excess permittivity with mole fraction of dimethyl phthalate + triethylamine**

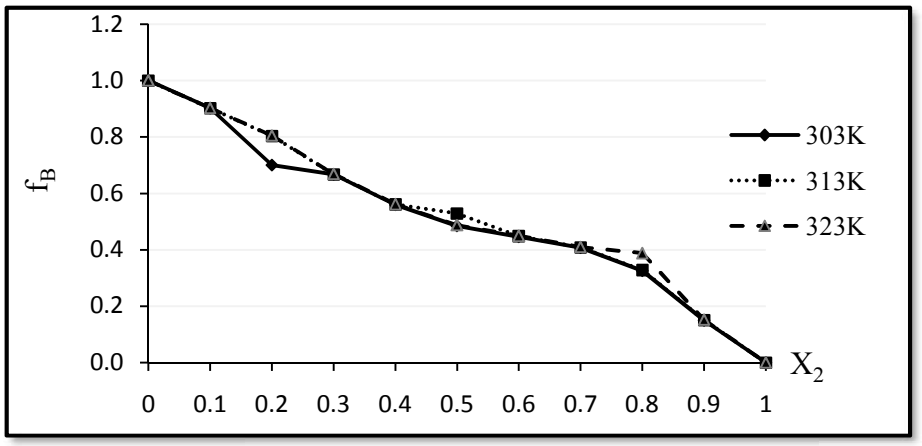


**Figure 4 : Plot of Bruggeman parameter with volume fraction of dimethyl phthalate + triethylamine**

**Table 2. Variation of Plot $\Delta F_0^E$, $\Delta F_{rr}^E$, $\Delta F_{AB}^E$ and $\Delta F^E$ with mole fraction $X_2$ of dimethyl phthalate and triethylamine**

| Temperature K | $X_2$ | $\Delta F_0^E$ J/mole | $\Delta F_{rr}^E$ J/ mole | $\Delta F_{□□}^E$ J/ mole | $\Delta F^E$ J/ mole |
|---|---|---|---|---|---|
| 303 | 0.1 | 49.1452 | 2.9917 | -1.2122 | 50.9248 |
| | 0.2 | 94.6958 | 5.7507 | -0.1488 | 100.2977 |
| | 0.3 | 164.5122 | 8.9187 | 5.4200 | 178.8509 |
| | 0.4 | 202.7494 | 10.6697 | 6.9310 | 220.3501 |
| | 0.5 | 202.6057 | 11.3145 | 4.5260 | 218.4462 |
| | 0.6 | 163.1577 | 11.5726 | -0.1268 | 174.6035 |
| | 0.7 | 115.5956 | 12.0355 | -3.0691 | 124.5621 |
| | 0.8 | 80.5621 | 11.8097 | -0.0867 | 92.2851 |
| | 0.9 | 69.3457 | 7.2972 | 0.9434 | 77.5863 |
| 313 | 0.1 | 50.1872 | 5.1875 | -2.5334 | 52.8413 |
| | 0.2 | 96.0192 | 9.7690 | -1.8238 | 103.9644 |
| | 0.3 | 166.7274 | 14.9950 | 4.4887 | 186.2111 |
| | 0.4 | 205.5808 | 17.7003 | 6.4730 | 229.7541 |
| | 0.5 | 205.4407 | 18.4222 | 4.2994 | 228.1623 |
| | 0.6 | 165.1616 | 18.3678 | -0.4967 | 183.0326 |
| | 0.7 | 116.6034 | 18.6581 | -3.5407 | 131.7209 |
| | 0.8 | 80.9901 | 18.0362 | -0.2744 | 98.7519 |
| | 0.9 | 70.3070 | 11.0970 | 0.6085 | 82.0126 |
| 323 | 0.1 | 50.5626 | 7.2827 | -3.8228 | 54.0224 |
| | 0.2 | 96.8382 | 13.7007 | -3.6836 | 106.8554 |
| | 0.3 | 168.6441 | 21.0369 | 3.3000 | 192.9810 |
| | 0.4 | 208.2706 | 24.8238 | 5.9760 | 239.0704 |
| | 0.5 | 208.2235 | 25.8010 | 4.1727 | 238.1971 |
| | 0.6 | 167.1257 | 25.6529 | -0.8028 | 191.9759 |
| | 0.7 | 117.5658 | 25.9915 | -3.9492 | 139.6082 |
| | 0.8 | 81.3889 | 25.1086 | -0.4837 | 106.0139 |
| | 0.9 | 71.3161 | 15.4969 | 0.0787 | 86.8917 |

The magnitude of $\Delta F_{AB}^E$ gives the information on the strength of interaction between unlike molecules. For our system it is found that, strength of interaction is maximum between the unlike molecules about 30% to 70% and it is very low in phthalate and amine rich regions. Finally the positive values of $\Delta F^E$ for the present system for all concentration and at all temperatures indicates the formation of β-clusters. Due to the formation of β-clusters the effective dipole moment increases (Gupta *et al.,* 2005; Vijaya Krishna and Sreehari Sastry, 2010).

## Conclusion

The values of dielectric constant, dielectric constant at optical frequency, excess permittivity, Kirkwood correlation factor, Bruggeman parameter, long range and short range interaction between like and unlike molecules are given for the combination of binary mixture of dimethyl phthalate and triethyl amine at different temperatures (303K, 313K and 323K). The energy of dimethyl phthalate increases in a rapid way with the addition of triethylamine. The strength of interaction between the molecules reach a maximum when the concentrations of triethyl amine and phthalate are nearly equal. The above listed data can be used to interpret liquid structure in above mixture.

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