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Hydrogen bond interactions of ethyl acetate with methyl Cellosolve: FTIR spectroscopic and dielectric relaxation studies

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ABSTRACT

H-bond interactions between ethyl acetate (EAC) and methyl cellosolve (MCS) have been investigated using FTIR and dielectric studies. FTIR studies show the presence of (*EAC* CH_3*and/or* CH_2) $C-H\cdots O$ (*ether linkage and/or hydroxyl oxygen of MCS*), (*MCS* CH_3 *and/or* CH_2) $C-H\cdots O$ (*ester linkage and/or carbonyl oxygen of EAC*) and the (*MCS*) $O-H\cdots O$ (*ester linkage and/or carbonyl oxygen of EAC*) H-bonds. A doublet of $\nu(C=O)$ appears in MCS rich solutions while singlet is noticed in EAC rich solutions. The relaxation time of pure *MCS* is smaller than the value for solutions with $X_2 = 0.8$, 0.9. Relatively larger deviation of g_f from unity is observed in EAC rich solutions.

1. Introduction

Hydrogen bond, an important interaction force among the non-covalent interactions, plays a crucial role in the shape and structure of molecules. This highly unique H- bond interaction has been broadly classified as red and blue shifting H- bonds [1]. The emergence of the concept of blue shifting H-bonds has motivated many researchers to investigate various molecular systems to learn about the features of H-bonds and to thereby effectively distinguish the blue and red shifting type [2–5] of interactions. In the curiosity to know about H-bonds in various molecular systems, the organic compounds called esters were also widely studied since they have two electronegative sites (ester linkage oxygen and carbonyl oxygen) [6–12]. On the basis of the reports regarding the blue shifting H-bonds in literature, one can believe that there are possibilities for these two electronegative sites to induce blue shifting H- bonds among ester molecules as well as with other molecules. The molecule with the formula $CH_3-CO-O-CH_2-CH_3$ called ethyl acetate that belongs to esters has been studied in combination with nitrobenzene [13] and chlorobenzene [14] by our research group both experimentally and theoretically. The results obtained show that ethyl acetate (EAC) may form less stable dimers and these dimers of ethyl acetate has been studied with methyl cellosolve(MCS), an alcohol, in the present work. Alcohols are highly polar and self associative and therefore, many works have been carried out on their homo and hetrointeracting nature [15–18]. In particular, this MCS is an important alcohol in the capacity that it has two electronegative sites and one hydroxyl hydrogen. These acceptor and donor sites make MCS a potential candidate that is capable of inducing red and blue shifting H-bonds among MCS as well as EAC molecules. A more rigorous study on the H-bond interactions of MCS with EAC needs quantum chemical calculations also. But, the part of the study dealing with the quantum chemical calculations has been devoted to a future research article. Only the results of FTIR and dielectric studies that give information on the possible interaction forces that are operating between various interaction sites of EAC and MCS, and the changes in the homo and heteroassociated networks with respect to the concentration of MCS/EAC have been presented in this work.

2. Materials and methods

Methyl cellosolve (*MCS*) of anhydrous grade was purchased from Sigma Aldrich, Pvt. Ltd., Mumbai, India and Ethyl acetate (*EAC*) of HPLC grade was obtained from Sisco Research Laboratories Pvt. Ltd., Mumbai, India. These chemicals were used as such without any purification. The FTIR spectra of neat *EAC*, *MCS* and their binary solutions (SS1 = 0.8 *EAC* +0.2 *MCS*, SS2 = 0.6 *EAC* + 0.4*MCS*, SS3 = 0.4 *EAC*

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$+0.6$ *MCS* and SS4 $= 0.2$ *EAC* $+0.8$ *MCS*) were recorded using Perkin Elmer FTIR Spectrophotometer (model: RX1) with a resolution of 4 cm^{-1} at room temperature in 4000 to 400 cm^{-1} region. Here, 0.2, 0.4, 0.6 and 0.8 are the mole fractions.

For dielectric relaxation studies, the binary solutions of *EAC* with *MCS* were prepared over the entire composition range. The measurements were carried out using Tektronix DSA8200 Digital Serial Analyzer sampling main-frame along with the sampling module 80E08 over the frequency range: 10 MHz to 30 GHz. The dielectric parameters were extracted by the methods mentioned in the earlier articles [19,20].

3. Results and discussion

3.1. FTIR spectroscopic studies

FTIR spectra of neat *EAC*, *MCS* and their binary solutions at different mole fractions are given in Figs.1 and 2. Since there is a severe overlap of the $C-H$ stretching bands of EAC and MCS, this band has been deconvoluted into four Gaussian peaks for pure components and eight (Fig. S1, the supplementary file) in the case of binary solutions. The fundamental stretching bands $v_{as}(CH_3)$, $v_{as}(CH_2)$, $v_s(CH_3)$, $v_s(CH_2)$, $\nu(C=O)$, $\nu(C-C-O)$ and $\nu(O-CH_2-C)$ of pure *EAC* appear at 2990.2, 2956.1, 2903.9, 2844.8, 1765.3, 1242.9 and 1056.3 cm^{-1}, respectively (Table 1). The $v(O-H)$, $v_{as}(CH_3)$, $v_{as}(CH_2)$, $v_s(CH_3)$, $v_s(CH_2)$, $v(C-O-C)$ and $v(C-OH)$ bands of *MCS* are noticed at 3405.8, 2988.3, 2941.0, 2886.4, 2827.1, 1123.7 and 1067.1 cm^{-1}, respectively (Table 1). Alcohols are always highly self-associating [21] in nature which leads to the formation of multimers, through the strong classical $O-H\cdots O$ hydrogen bonds, of various orders giving rise to a very broad absorption in the 3200–3500 cm^{-1} range [22]. So, the $v(O-H)$ band of pure *MCS* at 3405.8 cm^{-1} is taken as the stretching absorption by the pure *MCS* multimers. Costa et al. [23] have analyzed the molecular aggregation in MCS (2-methoxyethanol) and its solution with acrylamide using Raman Spectroscopy and ab initio calculations and reported that neat MCS exists as dimers. According to Costa et al., the basis for arriving at this conclusion is the large red shifts of the calculated Raman wavenumbers of the $\nu(OH)$, $\nu(CH)$ and $\nu(CO)$ stretching vibrations of the most stable *tgg* conformer of MCS when compared with the experimental values. Since this is completely wrong, $v(O-H)$ band of pure *MCS* at 3405.8 cm^{-1} is taken as the resultant of the $v(O-H)$ of more than one type of multimers including dimers. Using matrix-isolation infrared spectra and conformational analysis based on ab initio MO calculations, Yoshida et al. [24] have reported the coexistence of monomer with dimer and higher order aggregates in neat MCS.

In SS1, $v_{as}(CH_3)$ and $v_{as}(CH_2)$ bands of *EAC* got blue shifted to 3008.3, and 2984.9 cm^{-1}, respectively, while the $v_s(CH_3)$ and $v_s(CH_2)$ has been red shifted to 2885.8 and 2826.1 cm^{-1}(Table 1), respectively. A concomitant red shift of $v(O-H)$, $v(C-O-C)$ and $v(C-OH)$ bands of *MCS* to 3403.8, 1120.6 and 1061.8 cm^{-1}, respectively, can be noticed from the FTIR spectrum of the solution SS1 (Figs. 1 and 2, and Table 1). These shifts may be due to the combined effect of all or any of the four (*EAC* CH_3*and/or* CH_2) $C-H\cdots O$ (*ether linkage and/or hydroxyl oxygen of MCS*) H-bonds formed between EAC and MCS.

Costa et al. [23] have reported that both the hydroxyl and ether linkage oxygens of *MCS* involve in the H-bond interactions during the formation of MCS-acrylamide aggregates. The possibility for the presence of the (*MCS* CH_3 *and/or* CH_2) $C-H\cdots O$ (*ester linkage and/or carbonyl oxygen of EAC*) H-bond interactions can be ascertained if the red and blue shifts of $\nu(C=O)$ and $\nu(O-CH_2-C)$, respectively, of *EAC* are correlated with the shifts suffered by the $v(O-H)$, $\nu(CH_3)$ and $\nu(CH_2)$ of MCS (Figs. 1 and 2, and Table 1). The $v(O-H)$ is found to be red shifted to 3403.83 cm^{-1} in the solution (SS1) in which the concentration of MCS is one fourth of the EAC concentration. Under this condition, normally, the $v(O-H)$ should be shifted to higher wavenumber due to the dissociation of MCS multimers. Instead, a red shift has been observed which confirms the participation of hydroxyl hydrogen in H-bond interaction, which is stronger than the H-bonds existing among self-associated MCS/EAC networks, with EAC ester linkage and/or carbonyl oxygen.

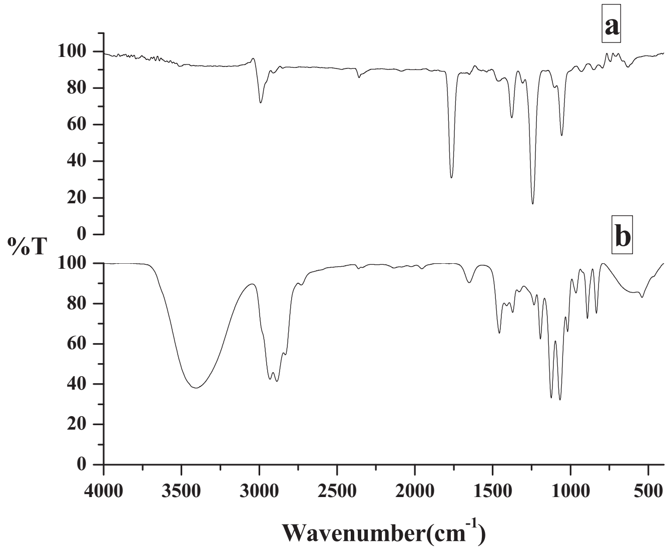

Fig. 1. Experimental FTIR spectrum of (a) Neat ethylacetate (EAC) and (b) Neat methyl cellosolve (MCS).

In the solution SS2 also, the same trend of frequency changes as observed in SS1 occurs with the exception of the shift in $\nu(O-H)$ which has been shifted to the high wave number side. This shows that all the interactions existed in SS1 continue to exist in SS2. At the same time, now the number of $O-H$ hydrogens that are free from H-bond interactions may be more than that involved in H-bond interactions with either another MCS molecule or with EAC molecule or with both of them. In the remaining solutions SS3 and SS4, all the frequency changes noticed in SS1 continue with the exception that a doublet of $\nu(C=O)$ appears in SS3, one at 1759.9 and the other at 1739.8 cm^{-1}, and this doublet, which appears as a somewhat broad absorption, is very weak in SS4 because of the smaller number of EAC molecules when compared with the number of MCS molecules. In our earlier works, it has been reported that the dimerization of EAC with the absence of interaction at the

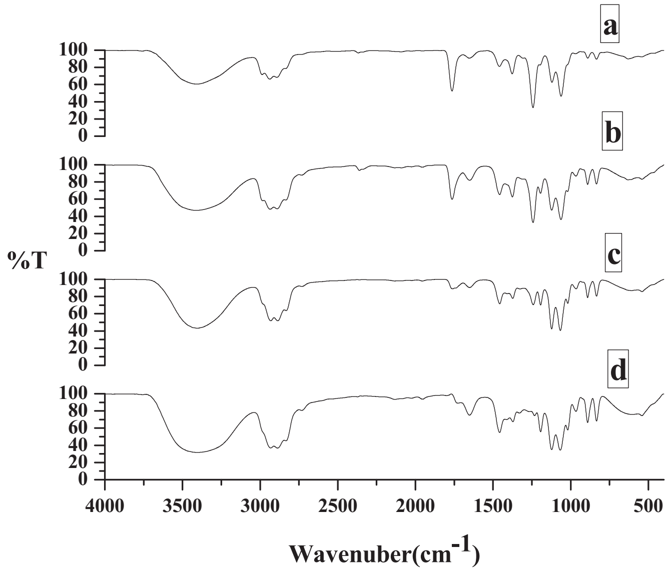

Fig. 2. Experimental FTIR spectra of the binary solutions (a) SS1 (0.8 EAC + 0.2 MCS), (b) SS2 (0.6 EAC + 0.4 MCS), (c) SS3 (0.4 EAC + 0.6 MCS) and (d) SS4 (0.2 EAC + 0.8 MCS).

Table 1
FTIR Spectral band assignment for neat *EAC*, *MCS* and their binary solutions at various mole fractions.

Vibrational bands	Wavenumber (cm^{-1})				
	EAC	SS1 0.8EAC+0.2MCS	SS2 0.6EAC+0.4MCS	SS3 0.4EAC+0.6MCS	SS4 0.2EAC+0.8MCS
CH_3 asy.str	2990.2	3008.3	3004.1	3002.7	3002.1
CH_2 asy.str	2956.1	2984.9	2971.5	2957.8	2968.7
CH_3 sy.str	2903.9	2885.8	2885.7	2885.8	2883.1
CH_2 sy.str	2844.8	2826.1	2823.7	2823.3	2824.7
$C=O$ str	1765.3	1764.2	1763.3	1759.9 1739.8	1726.0
$C-C-O$ str	1242.9	1242.9	1242.6	1241.1	1232.4
$O-CH_2-C$ str	1056.3	1061.8	1062.5	1066.4	1066.9
Vibrational bands	**MCS**				
$O-H$ str	3405.8	3403.8	3410.1	3404.3	3402.4
CH_3 asy.str	2988.3	2996.9	2990.1	2987.3	2989.3
CH_2 asy.str	2941.0	2942.7	2941.9	2932.9	2939.1
CH_3 sy.str	2886.4	2841.6	2838.3	2836.9	2839.9
CH_2 sy.str	2827.1	2813.3	2811.5	2811.3	2812.2
$C-O-C$ str	1123.7	1120.6	1122.5	1122.6	1122.4
$C-OH$ str	1067.1	1061.8	1062.5	1066.4	1066.9

asy.str- Asymmetric stretching vibration, sy.str- Symmetric stretching vibration, str- Stretching vibration.

$C=O$ oxygen gives rise to a $\nu(C=O)$ band at a blue shifted position from the monomer band [13] and that with the presence of interaction at the site of carbonyl oxygen results in a $\nu(C=O)$ band at a red shifted frequency [14]. Therefore, the presence of the doublet of $\nu(C=O)$ at a wave number lower than the carbonyl stretching band of neat EAC is the indication that there are two $C=O$ oxygens, one of which is bonded to MCS multimers and the other to another EAC molecule. In EAC rich solutions (SS1 and SS2), the $\nu(C=O)$ band appears as a single symmetric band and this means that most of the heteroassociated carbonyl oxygens are under the same chemical surrounding. The appearance of the broad absorption of $O-H$ stretching vibrations in all the solutions (Figs. 1 and 2) strengthens the point that *MCS* molecules maintain their multimer structure in all the binary compositions.

3.2. Dielectric relaxation studies

The plot of dielectric permittivity and dielectric loss against the logarithmic frequency is given in the Fig. 3. In the present work, we have applied two different relaxation models for fitting complex permittivity spectra. Debye model has been used to fit pure *EAC* permittivity data as in the earlier reports [13,14,25,26] and Cole- Davison model is used for neat *MCS* data. The Debye model was found suitable for all the solutions $(0 \le X_2 \le 0.9)$ except pure *MCS* which was fitted using Cole- Davison model. The static dielectric constant, dielectric constant at high frequency limit and the dielectric relaxation time obtained from complex permittivity spectra are given in Table 2. In the complex permittivity spectra, the dielectric loss peak of pure *MCS* appears in the low frequency range which can be taken as a result of *MCS*···*MCS* self associated multimers of various orders. The dielectric loss peaks of the solutions with the concentration range $0 \le X_2 \le 0.4$ exceed the frequency range of 30 GHz. As the concentration of *MCS* decreases in the *EACMCS* solutions, the peaks shift towards the high frequency range. The dissociation of *MCS* multimers and the formation of new *EAC*···*MCS* hydrogen bonded networks might have shifted the peaks of the solutions to the high frequency side. The relaxation time (τ) of pure *MCS* is 29.07 ps which is greater (Table 2) than that of pure *EAC* ($\tau = 4.59$ ps). This very high difference between relaxation time values occurs because of the *MCS* multimers. In the investigation of methyl acetate with alcohols, Yaseen et al. [27] have reasoned out that the high relaxation time for alcohols are due to multimer structures joined via $O-H\cdots O$ bonds. Similarly in our study, the self associated multimers may take more time to complete their relaxation process than the *EAC* monomers or dimers. The viscosities of the solutions may also play a role in the relaxation process. The viscosities of *EAC* and *MCS* are 0.423 mPa s [28] and 1.543 mPa s [29], respectively. The less viscous *EAC* dipoles can easily reorient than that of *MCS* dipoles and take smaller time to relax when the external field is applied. The tendency of alcohols to form $O-H\cdots O$ H-bonds and their relatively higher viscous nature are the major factors that contribute to their relaxation process.

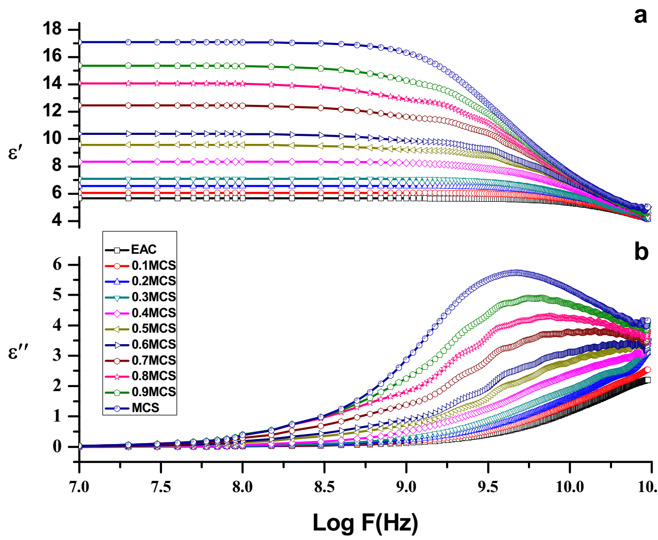

Fig. 3. The plot of a) dielectric permittivity(ε′) and b) dielectric loss (ε″) against log F for EACMCS solutions at 298 K.

Notably the τ value at the concentrations $X_2 = 0.8$ and 0.9 is higher than that of pure *MCS*. The relaxation time values for the solutions have been reported to be greater than that of pure *MeOH* over the concentration range $0.58 \le X_2 \le 0.91$ in the case of the TDR studies on the solutions of dibutylether (*DBE*) with methanol(*MeOH*) [22]. In that case, it has been confirmed that the τ value greater than that of the pure MeOH is due to the formation of DBE-MeOH complexes of size larger than the MeOH multimers. So, the τ value for the solutions with the concentrations $X_2 = 0.8$ and 0.9 is clearly due to the slowdown in the relaxation process as a result of the formation of *EACMCS* complex structures of size larger than pure *MCS* multimers. The MCS multimers which do not take part in the heteroassociation also has contribution to the relaxation process.

The plot of static dielectric constant which varies non-linearly against the mole fraction of *MCS* has been given in the Fig. 4. This type of non-linearity is discussed as a consequence of both homo and heterointeractions in various reports [30–32]. In the present investigation, this non-linearity can be attributed to the presence of (*EAC*

Table 2
Static dielectric constant (ε_0), dielectric constant at high frequency limit (ε_∞), relaxation time (τ), effective Kirkwood correlation factor (g^{eff}) and corrective Kirkwood correlation factor (g_f) for the binary solutions of EAC with MCS. Here, (X_2) is the mole fraction of MCS at 298 K.

x_2 (mole/l)	ε_0	ε_∞	τ/ps	g^{eff}	g_f
0.00	5.65	2.062	4.59	0.79	1.00
0.11	6.07	1.883	7.55	0.87	0.85
0.19	6.56	1.895	8.35	0.89	0.82
0.27	7.09	1.905	10.50	0.91	0.79
0.39	8.34	1.908	14.87	1.00	0.82
0.49	9.57	1.910	25.24	1.09	0.85
0.59	10.38	1.920	27.44	1.11	0.84
0.71	12.26	1.930	28.86	1.23	0.90
0.80	13.84	1.955	30.92	1.31	0.93
0.89	15.20	1.985	32.20	1.35	0.95
1.00	17.21	1.987	29.07	1.46	1.00

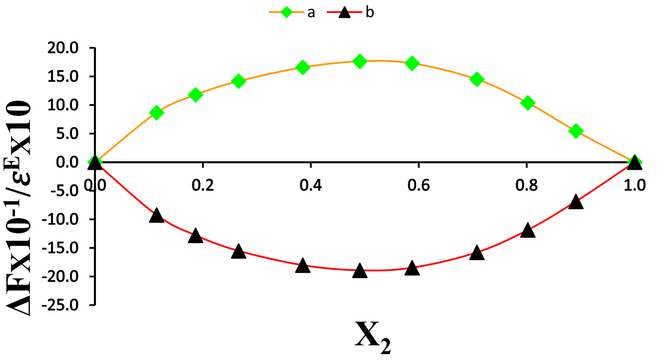

Fig. 5. Plot of a) excess free energy ΔF (J/mol) and b) excess permittivity ε^E against the mole fraction(X_2) of MCS in EACMCS binary solutions.

$CH_3$and/or CH_2) $C - H\cdots O$ (ether linkage and/or hydroxyl oxygen of MCS) and the (*MCS CH_3 and/or CH_2) $C - H\cdots O$ (ester linkage and/or carbonyl oxygen of EAC*) H-bond interactions which have been predicted using FTIR approach. The g^{eff} value for neat *EAC* is 0.79, less than unity, and is the consequence of the antiparallel orientation of *EAC* dipoles, of monomer or cyclic dimeric structures of *EAC* which has been confirmed in our earlier report [13] using FTIR and theoretical studies, in their pure form. For pure *MCS*, the g^{eff} value is 1.46 showing the parallel orientation of *MCS* dipoles that arise out of multimers of various orders. This parallel alignment prevails over the concentration range $0.49 \le X_2 \le 1$ and in the remaining solutions ($0 \le X_2 < 0.49$), the antiparallel orientation of dipoles arising out of the heterointeraction of EAC with lower order *MCS* multimers and pure EAC molecules that have not participated in heterointeraction due to their number exceeding that of MCS networks can be noticed.

The strength of heterointeractions can be studied from g_f values which are found to be slightly deviated from unity for all the binary solutions. The deviation of g_f values from unity has been discussed [33–35] in literature as an index of the strength of the heterointeraction. A small deviation of g_f values from unity has been observed for the binary mixture of caprylic acid with tetrahydrofuran [33] between which feeble heterointeraction existed. In the concentration range of $X_2 = 0.1$ to 0.6 for the present system, the g_f values deviate relatively more from unity compared with the other solutions. This is the consequence of heterointeractions dominating in the *EAC* rich solutions. In the remaining solutions, the *MCS⋯MCS* homo interacting multimers dominate. The negative deviation of excess dielectric constant (Fig. 5) appears over the entire concentration range of the *EACMCS* binary solutions. In general, the negative deviation is due to the following factors: i) decrease in the degrees of the molecular dipoles [36], ii) decrease in the total number of effective dipoles [37], iii) presence of heterointeractions between the solute and solvent [38] and decrease in the dielectric relaxation strength in the binary solutions [13]. In the present work, the decrease in the total number of effective dipoles is not possible because both of the solution constituents are self-associative in nature. Of the four, only the three factors excluding the decrement in the number of dipoles can be held responsible for the negative deviation of excess dielectric constant. The positive deviation of excess free energy values (Fig. 5) for all the *EACMCS* solutions indicates the dominant contribution from rupture in the self associating [39,40] species which are the MCS multimers of various orders and EAC dimers. The asymmetry (Fig. 5) in the curve may be due to the difference in the extent of heteroassociation which depends on the relative number and strength of the self-associated MCS and EAC networks as discussed using FTIR results.

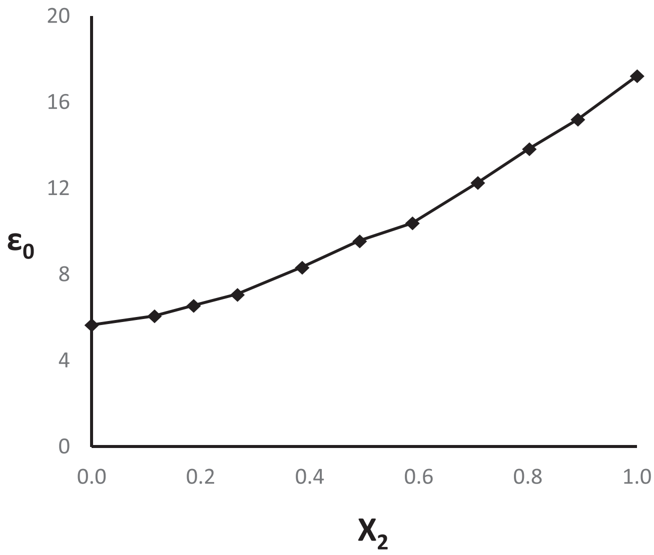

Fig. 4. ε_0 vs X_2 plot for EACMCS binary solutions at 298 K. Here, X_2 is the mole fraction of MCS.

4. Conclusions

From the results of FTIR and dielectric studies on *EACMCS* solutions, the present work can be summarized with the following conclusions.

- FTIR studies have shown the presence of (*EAC $CH_3$and/or CH_2) $C - H\cdots O$ (ether linkage and/or hydroxyl oxygen of MCS*), (*MCS CH_3 and/or CH_2) $C - H\cdots O$ (ester linkage and/or carbonyl oxygen of EAC*) and the (*MCS*) $O - H\cdots O$ (*ester linkage and/or carbonyl oxygen of EAC*) H-bond interactions.
- The presence of the doublet of $\nu(C = O)$ of EAC in MCS rich solutions (SS3 and SS4) at a wave number lower than the carbonyl stretching band of neat EAC shows that there are two $C = O$ oxygens, one of which is bonded to the methyl/methylene/hydroxyl hydrogen of MCS multimers and the other to the methyl/methylene hydrogen of another EAC molecule.
- In EAC rich solutions (SS1 and SS2), a single $C = O$ stretching band with considerable symmetry appears suggesting that EAC monomers are bonded with MCS multimers.
- The relaxation time value of pure *MCS* is lesser than for the solutions with $X_2 = 0.8$, 0.9 which reveals the formation of *EAC - MCS* hetero structures whose size is larger than the *MCS* multimers.
- Relatively larger deviation of g_f from unity is observed in EAC rich solutions.

Supplementary data to this article can be found online at https://doi.org/10.1016/j.molliq.2020.112490.

CRediT authorship contribution statement

A. Mahendraprabu: Writing - original draft, Investigation. **T. Sangeetha:** Validation, Formal analysis. **P.P. Kannan:** Validation, Formal analysis. **N.K. Karthick:** Writing - review & editing. **A.C. Kumbharkhane:** Resources. **G. Arivazhagan:** Conceptualization, Writing - review & editing, Supervision.

Declaration of competing interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests.

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