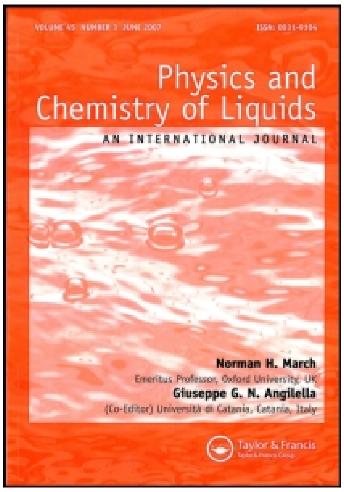

Physics and Chemistry of Liquids
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713646857

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Narasimha H. Ayachit[a]; F. M. Sannaninganavar[b]; D. K. Deshpande[c]
[a] Department of Physics, SDM College of Engg. and Tech. Dharwad, Karnataka, India [b] Department of Physics & BCA, Karnataka Science College, Dharwad, Karnataka, India [c] Department of Physics, Karnataka University, Dharwad, Karnataka, India

To cite this Article Ayachit, Narasimha H. , Sannaninganavar, F. M. and Deshpande, D. K.(2007) 'Viscoelastic studies of some phenols from dielectric measurements', Physics and Chemistry of Liquids, 45: 3, 359 — 368
To link to this Article: DOI: 10.1080/00319100600847622
URL: http://dx.doi.org/10.1080/00319100600847622

Physics and Chemistry of Liquids
Vol. 45, No. 3, June 2007, 359–368

Viscoelastic studies of some phenols from dielectric measurements

NARASIMHA H. AYACHIT*†, F. M. SANNANINGANAVAR‡ and D. K. DESHPANDE§

†Department of Physics, SDM College of Engg. and Tech. Dharwad, Karnataka, India
‡Department of Physics & BCA, Karnataka Science College, Dharwad, Karnataka, India
§Department of Physics, Karnataka University, Dharwad, Karnataka, India

(*Received 18 March 2006; in final form 8 June 2006*)

The variation of dielectric relaxation time with the viscosity of the medium is being exploited in drawing certain quantitative conclusions regarding molecular motion and intermolecular forces in liquids, liquid mixtures, dilute solutions and multi-component polar solutes in dilute solution. With no perfect empirical or theoretical equation in place for the variation of dielectric relaxation time with viscosity, the experimental investigations on different systems only can give an insight. In the present study the results of dielectric measurements carried out on pure samples of *o*-ethyl phenol, 2-*n*-butylphenol, 4-*n*-butylphenol, and 2,6-butylphenol in dilute solutions in different mixed solvents (benzene + paraffin) and on binary mixtures (1 : 1) of *p*-2-*n*-butylphenol + 4-*n*-butylphenol and *p*-bromonitrobenzene + 2,6-butylphenol, are reported. For comparison, the results on *p*-bromonitrobenzene + *m*-bromonitrobenzene as an example of mixture of non-associative liquids was also carried out and the results are presented. Different parameters determined using these dielectric measurements are also presented using different models and these studies indicate that the dielectric behavior at microwave frequencies favor the concept of dynamic viscosity and a single viscoelastic relaxation time for the system under study.

PACS CODE: Dielectric relaxation; Viscosity; Viscoelastic models; Phenols; Dynamic viscosity; High frequency rigidity modulus; Associative type liquids; Non-associative type liquids

1. Introduction

The determination of dielectric relaxation time and its dependence on the temperature and viscosity of the medium are studied as they help in obtaining certain quantitative conclusions regarding molecular motion and intermolecular forces in liquids, liquid mixtures, dilute solutions and multi-component polar solutes in dilute solution [1–45].

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*Corresponding author. Email: narasimha1957@yahoo.co.in

Physics and Chemistry of Liquids
ISSN 0031-9104 print/ISSN 1029-0451 online © 2007 Taylor & Francis
DOI: 10.1080/00319100600847622

The work on aromatic molecules in general, and liquid crystal samples in particular is being carried out by the authors and others from this laboratory [1–8]. This paper aims to build upon these studies. The studies pertaining to the effect of viscosity on dielectric relaxation time of polar molecules was first initiated by Debye [32] and these studies were later extended by others and few of such work is cited here from the available literature [1–43]. These studies are mainly based on Debye theory. A closer look reveals that in literature no single expression, either based on theoretical or empirical considerations exists that can predict satisfactorily the dependence of relaxation time on viscosity. The proposed relationship between the relaxation time (τ) and the viscosity (η) can be broadly classified into (i) theoretical and (ii) empirical relationship, the former includes the relationships based on Debye [32] and other viscoelastic models by others [31,33,34], while the other notably includes the relationships proposed by Fisher [35], Chitoku and Higasi [36], and Krishnaji *et al.* [37,38]. Judging from the results of the experimental investigations from different workers it may be concluded that the experimental facts could well be explained in the case of certain simple molecules in the light of Debye theory, but shows some serious anomalies in some other molecules which led to certain modifications to the Debye equation. There are methods available both based on empirical considerations and theoretical considerations in this respect; however, no single method is in a position to explain behavior of all type molecules and their mixtures. This is the reason why in the recent past dielectric studies on different type of molecules and mixtures have gained importance.

Although a body of work has been done on the dependence of relaxation time on viscosity, more experimental investigations seems to be warranted for a better understanding of the variation of the relaxation time with viscosity of the solvent. Hence in the present study dielectric measurements are carried out on pure samples *o*-ethyl phenol, 2-*n*-butylphenol, 4-*n*-butylphenol, and 2,6-butylphenol in dilute solutions in different mixed solvents (benzene + paraffin) at room temperature. Similar measurements on binary mixtures (1 : 1) of *p*-2-*n*-butylphenol + 4-*n*-butylphenol and *p*-bromonitrobenzene + 2,6-butylphenol were also carried at the room temperature. The results are presented and discussed. For comparison, the results on *p*-bromonitrobenzene + *m*-bromonitrobenzene system as an example of mixture of non-associative liquids was also carried out and the results are presented with reference to the results of individual constituents available in literature [2].

2. Method

The quantities relating to the effect of viscosity and dielectric relation determined through the techniques described in section ‘experiment’ later in this article can be used to understand the viscoelastic behavior using the equations presented below.

For dilute solution of a polar solute in a non-polar solvent the loss tangent as a fair approximation is given by

$$\tan\delta = \left[\frac{\varepsilon' + 2}{\varepsilon'}\right]^2 \left[\frac{4\pi C N \mu^2}{27kT}\right]\left[\frac{\omega\tau}{1+(\omega\tau)^2}\right] = P\left[\frac{\omega\tau}{1+(\omega\tau)^2}\right]$$

where, $P=[(\varepsilon'+2)/\varepsilon']^2\ [(4\pi CN\mu^2)/(27kT)]$. P can be treated as constant at a given temperature. When $\omega\tau=1$, tan δ attains a maximum only if the relaxation time of the solute molecule is lower than that corresponding to $\omega\tau=1$ unlike in case of temperature variation [9]. Where it is greater than that corresponding to $\omega\tau=1$, thus restricting to make an appropriate choice for the value of τ from the two possible values (roots) obtained from $(\tan\delta)_{\max}=P/2$ permits one to determine μ. Whereas the value of τ for any value of μ may be determined accordingly, by noting that it is increasing with η as given by Debye relation for $\tau=4\pi\eta a^3/kT$.

Higasi [36] has given an empirical relation on the basis of non-linear relation between τ and η as $\tau=A\eta^{X}$, where A is a constant and is similar to one given by Eyring treating τ as a rate process. This equation indicates that a plot between $\log\tau$ and $\log\eta$ yields a straight line. Barlow *et al.* [34] have proposed an equation by treating the dielectric system as viscoelastic i.e., a material system in which stress is dependent on the strain and may be represented in the form $\eta_D=\eta_S/(1+(\omega\tau)^2)$. According to them a highly viscous liquid exhibits viscoelastic relaxation (τ_{ve}) at microwave frequencies and this can be related to the dynamic viscosity (η_D) and study flow viscosity η_S as given above. Also τ_{ve} is related to high frequency rigidity modulus G_∞ by the equation $\tau_{ve}=\eta_S/G_{\infty}$.

Magee [31] has derived the expression $\tau=[((1/2)kT)\ \xi_M\xi_R/(\xi_M+\xi_R)]$, where, ξ_M is frictional constant of the molecule and ξ_R is that of structural region. The later is related to the macroscopic viscosity η by the Stoke's relation $\xi_R=8\pi a_R^3\eta$. A microscopic viscosity η_M between the rotating molecules and its nearest neighbors is associated by the relation $\xi_M=8\pi a_M^3\eta_M$, where a_M is the radius of the polar solute molecule. Thus, the expression for τ can be given $\tau=[8\pi a_R^3\eta'/kT]\ [\eta/(\eta+\eta')]$, where $\eta=a_M^3\eta_M a_R^3$ is a limiting viscosity. It is noted that this equation is similar to Debye equation but incorporates a non-linear viscosity. This is again written as $\tau=\tau'[\eta/(\eta+\eta')]$ where $\tau=[8\pi a_R^3\eta'/kT]$ and τ' is called as limiting relaxation time. This shows that for large viscosity solvents i.e., $\eta\gg\eta'$, τ approaches τ' independent of η but depends upon the size of the solvent. The validity of this equation may be tested by plotting $(1/\tau)$ *versus* $(1/\eta)$ because $(1/\tau)=((\eta'/\tau'\eta)+(1/\tau'))$ and comparing the resulting values of τ' with those available in literature.

3. Experimental

The X band microwave setup was used to measure the dielectric constant and dielectric loss at 9.98 GHz as explained in our earlier work [1]. Microwave bench with necessary accessories supplied by M/S Scanner Ltd, England was used to measure $\varepsilon', \varepsilon''$ – the real and imagined part of the dielectric constant. For the generation of microwave a gun oscillator supplied by M/S Vidut Yantra Ltd, India operating in H_{01} mode was used instead of Klystron (R-302). The frequency 9.98 was fixed such that the intensity is maximum at the output. The technique adopted for these measurements was due to Whiffin and Thompson [24] modified later by Roberts and Von Hippel [25].

The viscosity of the different solutions was measured using Ostwald viscometer. The measured values of viscosity were estimated to be accurate to a second decimal. For a solute in a solvent three-time viscosity were determined three-time.

Different viscous solutions were prepared by mixing liquid paraffin with benzene as given below.

Solvent 1: Pure benzene
Solvent 2: 0.62CP (benzene + paraffin)
Solvent 3: 0.72CP (benzene + paraffin)
Solvent 4: 1.03CP (benzene + paraffin)
Solvent 5: 1.64CP (benzene + paraffin)
Solvent 6: 3.05CP (benzene + paraffin)
Solvent 7: 8.48CP (benzene + paraffin)

4. Results and discussion

The measured values of η_s, ε', ε'' and the derived values of $\tau_s \eta_d \tau_{ve}$ along with the limiting values of τ' and η' (Magee's model) are presented in tables 1 and 2. As it is assumed in the present work that the molecules studied, even in mixtures confirm closely to that predicted by Debye theory, the relaxation time τ determined can be used to calculate the size (radius) of the molecule under study. This physical constant, is determined using the relaxation times τ_s, τ_{ve} and τ' and are compared with values determined by the molar volume assuming the shape of the molecule to be spherical for ease in calculation. These values, namely, a_D, a_{ve}, a_{SR} and a_M respectively are given in table 1 for each molecule along with the high frequency limiting rigidity modulus G_∞. The values for *p*-bromonitrobenzene and *m*-bromonitrobenzene are presented from the earlier works of the author [5].

A look at the literature reveals that the experimental facts may be well understood in the case of certain simple molecules according to Debye theory, but reveals some serious anomalies in some other molecules. This has lead to certain modifications to the Debye equation. The assumption of linear dependence on viscosity for the relaxation time irrespective of the size and the shape of the surrounding molecules was found to be valid in the case of some simple molecules and surrounded by medium of low viscosity. Hill [33] proposed a concept of mutual viscosity, on the basis of local viscosity effect between species at the molecular level, which could successfully explain the observations in the case of host of molecules studied. The concept of viscoelastic behavior of the medium under study, namely, the viscoelastic relaxation in the medium caused by shear elastic waves produced by rotating dipoles has helped effectively in certain dielectric systems. Yet another concept that deals with treating the dielectric relaxation to be a co-operative phenomenon, such that the relaxation process involves regions of the liquids [9–15] and not just the rotation of single molecule has also helped to certain extent to explain the observed results. As regards the empirical relationships proposed it may be said that the assumption of non-linear dependence of $\tau = A\eta^X$, where A is constant and x is a parameter which can be co-related with molecular shape and solute–solvent interaction [36] or with ratio of enthalpies [38]. This simple relation has been found to be applicable, no satisfactory physical explanation for viscosity dependence in a quantitative manner can be presented. This relation fails to give a

Table 1. Data for different molecules (η, ε', ε'' and other data).

Molecule	η (CP)	ε'	$\varepsilon'' \times 10^3$	τ_s (ps)	η_d (CP)	τ_{ve} (ps)	$G_\infty = \text{dyne cm}^{-2}$
o-Ethylphenol	0.62	2.285	6.54	3.1	–	–	–
	0.72	2.320	18.15	16.6	–	–	–
	1.03	2.307	13.23	37.8	0.78	9.3	0.111
	1.64	2.293	11.06	48.2	0.80	16.9	0.097
	3.05	2.277	10.64	50.1	0.81	27.6	0.111
	8.48	2.227	8.6	63.0	0.82	50.7	0.167
	$a_M = 3.04$ Å, $a_D = 1.95$ Å, $a_{ve} = 2.73$ Å, $a_{SR} = 2.86$ Å, $\mu = 1.70D$, $G_{\infty\text{mean}} = 0.1215 \text{ dyne cm}^{-2}$, $\tau' = 40.4$ ps, $\eta' = 0.057$CP						
2-*n*-Butylphenol	0.62	2.274	9.01	15.5	–	–	–
	0.72	2.315	9.19	16.6	–	–	–
	1.03	2.292	8.88	20.7	–	–	–
	1.64	2.264	8.57	22.7	1.15	−10.8	0.151
	3.05	2.256	8.43	23.6	1.22	20.4	0.149
	8.48	2.220	8.22	24.3	1.27	39.5	0.214
	$a_M = 2.97$ Å, $a_D = 1.39$ Å, $a_{ve} = 1.86$ Å, $a_{SR} = 2.87$ Å, $\mu = 1.65D$, $G_{\infty\text{mean}} = 0.172 \text{ dyne cm}^{-2}$, $\tau' = 26.3$ ps, $\eta' = 0.37$CP						
4-*n*-Butylphenol	0.62	2.271	15.56	7.4	–	–	–
	0.72	2.315	20.98	16.1	–	–	–
	1.03	2.276	18.82	24.9	0.76	10.0	0.103
	1.64	2.269	17.28	29.6	0.78	17.5	0.094
	3.05	2.255	14.67	38.1	0.79	28.2	0.108
	8.48	2.234	12.31	47.6	0.86	87.3	0.282
	$a_M = 2.97$ Å, $a_D = 1.67$ Å, $a_{ve} = 2.42$ Å, $a_{SR} = 2.44$ Å, $\mu = 1.38D$, $G_{\infty\text{mean}} = 0.147 \text{ dyne cm}^{-2}$, $\tau' = 51.3$ ps, $\eta' = 1.18$CP						
2,6-Butylphenol	0.62	2.392	9.49	2.5	–	–	–
	0.72	2.303	29.93	16.1	–	–	–
	1.03	2.309	28.03	21.9	0.74	10.4	0.099
	1.64	2.270	25.54	28.0	0.76	17.9	0.092
	3.05	2.254	23.43	32.3	0.77	28.7	0.106
	8.48	2.215	20.13	34.9	0.78	52.4	0.162
	$a_M = 3.39$ Å, $a_D = 1.64$ Å, $a_{ve} = 2.33$ Å, $a_{SR} = 2.68$ Å, $\mu = 2.73D$, $G_{\infty\text{mean}} = 0.115 \text{ dyne cm}^{-2}$, $\tau' = 38.5$ ps, $\eta' = 0.67$CP						
p-Bromo nitrobenzene	0.62	2.267	15.88	7.3	–	–	–
	0.72	2.321	21.51	16.1	–	–	–
	1.03	2.300	20.08	22.9	0.78	9.5	0.108
	1.64	2.248	18.38	26.9	0.79	17.1	0.096
	3.05	2.238	17.27	30.1	0.81	27.6	0.111
	8.48	2.194	16.37	31.9	0.83	50.4	0.168
	$a_M = 3.12$ Å, $a_D = 1.63$ Å, $a_{ve} = 2.25$ Å, $a_{SR} = 2.58$ Å, $\mu = 2.10D$, $G_{\infty\text{mean}} = 0.121 \text{ dyne cm}^{-2}$, $\tau' = 33.3$ ps, $\eta' = 0.65$CP						
m-Bromo nitrobenzene	0.62	2.352	22.31	5.1	–	–	–
	0.72	2.367	38.74	16.1	–	–	–
	1.03	2.352	36.47	22.4	0.76	10.0	0.104
	1.64	2.315	33.81	26.2	0.78	17.5	0.094
	3.05	2.313	31.68	29.8	0.79	28.0	0.109
	24.59	2.203	23.10	44.4	0.83	88.8	0.277
	$a_M = 3.12$ Å, $a_D = 1.60$ Å, $a_{ve} = 2.33$ Å, $a_{SR} = 2.38$ Å, $\mu = 3.15D$, $G_{\infty\text{mean}} = 0.146 \text{ dyne cm}^{-2}$, $\tau' = 42.6$ ps, $\eta' = 1.06$CP						

constant value of x over a wide range of viscosity for example it gives a large value for low viscosity solutions as compared to that for high viscosity solutions.

The study of mixtures of polar liquids provides an experimental approach to the problem of defining the volume involved in the relaxation process. If the relaxation consists in single molecular re-orientation, an *A–B* mixture should show separate

Table 2. Data for different mixtures (η, ε', ε'' and other data).

Mixture	η (CP)	ε'	$\varepsilon'' \times 10^3$	τ_s (ps)
4-*n*-Butylphenol + 2,6-butylphenol	0.62	2.385	22.26	5.0
	0.72	2.356	39.00	16.1
	1.03	2.308	24.25	45.0
	1.64	2.281	19.96	57.3
	3.05	2.286	17.36	66.4
	8.48	2.230	16.02	70.6
p-Bromo nitrobenzene + 2,6-butylphenol	0.62	2.438	27.06	5.4
	0.72	2.422	44.93	16.1
	1.03	2.418	34.90	33.8
	1.64	2.324	32.85	34.8
	3.05	2.316	28.09	43.3
	8.48	2.297	23.48	53.7
p-Bromo nitrobenzene + *m*-bromo nitrobenzene	0.62	2.635	23.63	6.4
	0.72	2.619	34.37	16.1
	1.03	2.555	23.20	40.1
	1.64	2.532	19.09	51.1
	3.05	2.473	15.92	61.5
	24.59	2.394	14.87	64.1

absorption centers corresponding to the relaxation times of the constituents A and B; provided their τ values are sufficiently different (by a factor of 5–7) to allow of their resolution in practice. If the volume participating in the molecular relaxation is sufficiently large to have a composition indistinguishable from the overall average, then a single absorption will appear for the mixture. Based on these considerations a general trend for the observed dielectric absorption is that a mixture of two associated liquids and two non-associated liquids appear to merge so completely molecularly as to give only a single intermediate relaxation process. In other words, these media behave like microscopically homogeneous ones so that a single relaxation process may be expected. However, a polar mixture having its constituents as one associative and other non-associative type liquid may microscopically inhomogeneous and if τ values of constituent molecules are sufficiently well separated so as to allow of their resolution might exhibit two absorption peaks or, in other words, the constituents may retain their individual character.

In the light of the above, the experimental values obtained and the different parameters determined as presented in tables 1 and 2 for different systems the general observation made are presented below. Following Higassi, a plot of τ and η in general for any polar molecule is non-linear but a plot of $\log(\tau)$ *versus* $\log(\eta)$ is expected to be linear. In figure 1 such plots are shown. Figure 2 shows the plots of $(1/\tau)$ *versus* $(1/\eta)$ from which the limiting values of η' and τ' are determined.

(a) It is seen from the table that there is a close agreement between μ and τ values determined using Gopal krishna method [27] for these molecules reported in the literature and those reported in table 1. This is quite expected as in both the cases some consideration of Debye single relaxation is implicit. Further, the plots shown in figures 1 and 2 are linear on wider range of values although an indication of non-linearity appears at higher values. In consistency with this, the plots of $(1/\tau)$ *versus* $(1/\eta)$ show practically a linearity over whole range. This linearity of graphs is

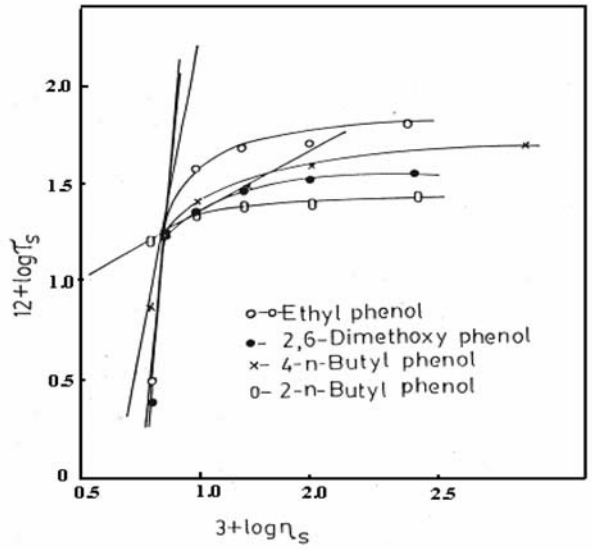

Figure 1. Plot of log τ_s *vs.* log η_s.

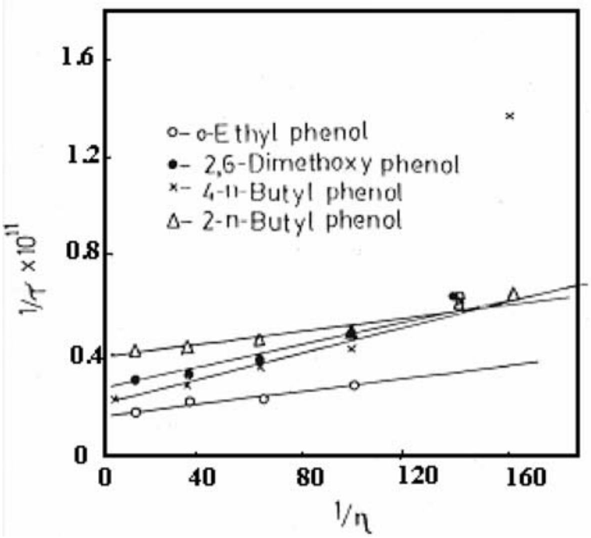

Figure 2. Plot of $1/\tau$ *vs.* $1/\eta$.

indicative towards the justification of the use of Debye relations for the molecules under study with fair approximation that the shape of these molecules do not much depart from their assumed shape being spherical.

(b) Referring to the table 1 it is readily seen that as η_s increases τ_s also increases as expected but the proportion by which these parameters change is different for different molecules. This has been also found true in case of mixtures as can be seen from table 2. In other words, the change in the value of τ from one viscosity value to another, in general is small, compared to the change in viscosity values. Further, it may be noted that $\tan\delta$ in each case attains a maximum value and this value enables one to evaluate the required molecular parameter μ and τ. In the four phenols studied here as η_s increases τ_s increases but not in the same proportion and the $\tan(\delta)$ values attains its maximum for $\eta = 0.72$CP in all the cases except for $\eta = 1.03$CP in case of 2-*n*-butylphenol. As pointed out earlier, it can be seen that although the measured value of the microscopic viscosity varies from 0.62 to 24.59CP, the variation of dynamic or the effective value is very small and is centered around 0.8 or 1.15CP. This trend is seen to a lesser extent in case of *o*-ethylphenol.

(c) It is clear from figures 1 and 2 that the plots of $\log(\tau)$ *versus* $\log(\eta)$ are linear up to low viscosities of the surrounding medium (=2CP) and that with higher viscosity values it deviates from linearity. This probably is indicative of the fact that the failure of Debye model at the higher viscosity values, which uses the expression $\tau = 4\pi\eta a^3/kT$. This view is consistent with certain observations available in literature [33–42]. This non-linear dependence of τ on η may be explained on the basis of viscoelasticity of the solvent medium [34] or Magee's approach [31] incorporating these concepts.

(d) Usually the values of η_d determined by extrapolating the linear region indicate that they are much smaller compared to the corresponding values of macroscopic viscosities. This indicates that effective frictional resistance experienced by the molecule during reorientation is less and this results in shorter viscoelastic relaxation times. It may be seen from this that the linear relation between $\log(\eta_{\min}/\eta_{\max})$ and $\log(\tau_{\min}/\tau_{\max})$ is better when $\tau_{\max}$ either τ_{ve} or τ 'is substituted than τ_s and η_d or η' is substituted for $\eta_{\max}$ instead of η_s. Further, the determined values of molecular radii using τ_{ve} or τ (a_{ve}, a_{SR}) are expected to be nearer to the molar radius a_M than the value a_D determined using τ_s i.e., simple Debye relation for τ. In the present study in the case of 4-*n*-butylphenol with the limiting values of viscosity used and the corresponding relaxation time, namely, $\eta_{\min} = 0.62$CP, $\tau_{\min} = 15.5$ ps, $\eta_{\max} = 8.48$CP, $\tau' = 51.3$ ps, it can be seen that the linear relation $\log(\eta_{\min}/\eta_{\max})$ and $\log(\tau_{\min}/\tau_{\max})$ is better than that for the $\tau_{\max}$ value corresponding to $\tau_s = 24.3$ ps.

It is suggested in the literature that a polar molecule departs in shape from sphere, the rotation occurs without any considerable displacement of the surrounding molecules and the relaxation time is insensitive to the macroscopic viscosity of the medium. On the other hand, if the molecule is unsymmetrical in shape, its rotation at least one axis involves the displacement of neighboring molecules and the corresponding relaxation time depends markedly on the viscosity of the medium. In the present work, the molecules except being associative in nature are spherical in shape; the measurements are taken at a fixed temperature and the same kind of solute solvent interactions are involved, the departure from linear relation between $\log(\tau)$ and $\log(\eta)$ may be attributed to the dynamical nature of the viscosity. In other words, the measured values of macroscopic viscosity of the medium are frequency dependent. These observations seem to be true when we consider the results obtained in the case of mixtures in the present work. The proportions by which τ and η change are different in each case of the molecule.

Thus it may be said that for the study of dielectric behavior microwave frequencies the concept of dynamic viscosity and a single viscoelastic relaxation time and also the model proposed by Magee which incorporates the above concepts in addition to accounting for the shape and size of the molecules might be better suited.

Acknowledgment

The authors (NHA, FMS) acknowledge the help and encouragement by their respective principals and managements in carrying out this work.

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