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Dielectric Relaxation Phenomenon between Aniline and 2-Alkoxyethanols in Different Non Polar Solvents by Frequency Domain Technique

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Abstract: The dielectric relaxation studies have been carried out on the ternary mixture of aniline with 2-alkoxyethanols (2-methoxyethanol, 2-ethoxyethanol and 2-butoxyethanol) in the dilute solution of non polar solvents benzene, CCl_4, 1,4–dioxane and toluene. The solutions were prepared for various molar ratios (1:3, 1:2, 1:1, 2:1 and 3:1) over the frequency at 9.43GHz in room temperature by using X-Band microwave technique were studied. The dielectric parameters like ε_0, ε', ε'' and $\square_\square$ are calculated. In addition, the dielectric relaxation times $\square_1 \square\square\square_2$ and $\square_0$ have been calculated by employing Higasi's method and activation energies $\square f_\square$ and $\square f_\square$ have been determined using dielectric data. The most likely association between hydroxyl groups of 2-alkoxyethanols with the amino group of aniline was studied. The results showed that the interaction of the order: 2-butoxyethanol >2-ethoxyethanol >2-methoxyethanol and play an important role in determination of the strength of hydrogen bond.
Key words: Dielectric Relaxation, ternary mixtures, 2-Alkoxyethanols, Aniline, X-Band Microwave technique

I. INTRODUCTION

Studying intermolecular interactions between liquid mixtures (both binary and ternary) playing important roles in various fields of chemical engineering systems, electrochemical cells, heterogeneous catalysis [1-2] and biological systems [3-5]. Generally liquid mixtures are mainly between two protic liquids, aprotic and protic liquid, and two aprotic liquids. And the mixing of two liquids such as solute–solute and solute–solvent have been reported to result in specific interactions such as hydrogen bonding, dipole–dipole, and charge transfer reactions[6].

2-alkoxyethanol and aniline are known for their self-association through intermolecular hydrogen bonding. Aniline has a phenyl group attached to an amino group having various applications. For example, as additives to rubber processing chemicals, herbicides [7],rocket fuel [8], synthetic dye industry [9] and emerged as an analgesic drug[10]. In the other hand, 2-alkoxyethanols is bifunctional organic compound and it contains both hydroxyl and ethergroups as well as an alkyl group. 2-alkoxyethanol is commercially known as cellosolves, in their pure state which is self-associated and form cyclic dimers in which the hydroxy proton of one cellosolve molecule is bound to the ether oxygen atom of the other [11].

The present paper reports dielectric constants, dielectric loss, dielectric relaxation time and activation free energy for ternary mixtures containing aniline with2-alkoxyethanols (2-methoxyethanol, 2-ethoxyethanol and 2-butoxyethanol) in different non polar solvents (benzene, CCl_4, 1,4 dioxane and toluene) at room temperatures. Studying intermolecularHbonding between hydroxyl group of 2-alkoxyethanol and N-H group of aniline and interactions between the mixtures containing 2-alkoxyethanol are very important not only because of their selfassociation, but also due to the strong intramolecular effects produced by the presence of –O– and –OH groups in the same molecule [12].

II. EXPERIMENTAL

A. Materials
Chemical used for the present investigation are analytic reagents with minimum assay, Merck variety of aniline, benzene, CCl4, 1,4-dioxane and toluene. 2-alkoxyethanols (2-methoxyethanol, 2-ethoxyethanol and 2-butoxyethanol) were purchased from sigma Aldrich. The chemicals were used without purification.

B. Method
The static dielectric constants were measured by heterodyne beat method at the room temperature using a Dipole meter supplied by Mittal Enterprises, New Delhi. The refractive index was measured by using Abbe's refractometer. The measurement of dielectric

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constant (ε') at an angular frequency and dielectric loss (ε'') were carried in the X-band microwave frequency of 9.43GHz. Viscosities have been measured by an Oswald's Viscometer of with 10ml capacity at room temperature. The flow time of water and liquid mixtures are measured by a digital stop watch. The density of the pure and liquid mixtures has been measured by relative measurement using specific gravity bottle of 10 ml capacity. The proton donors (2-alkoxyethanols) and the acceptor (aniline) under study were separately dissolved at the same molar concentration (0.3mol/L) in the different solvents like benzene, CCl_4, 1,4-dioxane and toluene. Their dielectric constants were measured separately. Then the two solutions were mixed in different proportions at constant measurements. As the maximum deviation of dielectric constant for all the systems occurs at equimolar ratio of the solutes, it is presumed that the deviation is due to the formation of 1:1 complexes.

C. Estimation of Dielectric Parameters
According to Higasi's method [13], the average relaxation time τ_1 is described by

$$\tau_1 = \frac{a''}{(a' - a_\infty)}$$

While the overall dielectric relaxation τ_2 and the mean relaxation time τ_0 is given by,

$$\tau_2 = \frac{(a_0 - a')}{\omega a''}$$

$$\tau_0 = \sqrt{\tau_1 \times \tau_2}$$

Where ω is the angular frequency, a_0, a', a'' and a_∞ are defined by the following equations

$$\varepsilon_0 = \varepsilon_{01} a_0 w_2$$
$$\varepsilon' = \varepsilon_1' + a' w_2$$
$$\varepsilon_\infty = \varepsilon_{1\infty} + a_\infty w_2$$

in which subscript 1 refers to the pure solvent, subscript 2 refers t the solute, subscript 0 refer to the static frequency measurements and w_2 is the mass fraction of the solute.
The molar free energies have been calculated using the Eyring's equation [14],

$$\tau = \left(\frac{h}{kT}\right) \exp\left(\frac{\Delta F_\tau}{RT}\right)$$

$$\tau = \left(\frac{Nh}{V}\right) \exp\left(\frac{\Delta F_\eta}{RT}\right)$$

Where, h is the Plank's constant, k the Boltzmann constant, N is the Avogadro number and V is the molar volume and ΔF_τ & ΔF_η are the molar free energies for the dielectric relaxation process and the viscous flow process respectively.

III.   RESULT AND DISCUSSION

The ternary systems selected were anilinewith 2-Alkoxyethanols(2-methoxyethanol, 2-ethoxy ethanol and 2-butoxyethanol) in different non polar solvents (benzene, ccl₄, 1,4-dioxane and toluene) at room temperatures. The value of relaxation time τ_1, τ_2 and τ_0 for all the systems were calculated by Higasi's et al. method [13]. It is observed that the value of relaxation time τ_1, τ_2 and τ_0 increases with increasing chain length of 2-Alkoxyethanols and the observed values are reported in the Table 1- 3. The increase in relaxation time may be due to the increase in effective radius of the rotating unit.

Table 1. Value of dielectric constants and relaxation times and activation energy for the various weight fractions of the mixture of Aniline with 2-methoxy Ethanol in different solvents

Ratio	Weight fraction W_2	ε_0	ε'	ε''	ε_∞	Relaxation Time (ps) using Higasi's Method			Activation energy	
						τ_1	τ_2	τ_0	□□□□	□□f□
				Aniline + 2-Methoxy Ethanol + Benzene						
1:3	0.0304	2.6576	2.4021	0.1667	2.2568	19.84	25.47	22.48	14.21	16.92

1:2	0.0299	2.7743	2.4073	0.1911	2.2579	22.10	31.92	26.56	14.59	17.53
1:1	0.0289	2.9179	2.4105	0.2150	2.2604	24.76	39.19	31.15	14.82	17.86
2:1	0.0280	2.8004	2.4085	0.1910	2.2632	22.74	34.09	27.84	14.26	17.34
3:1	0.0274	2.6576	2.4050	0.1688	2.2656	20.99	24.87	22.85	14.13	16.58

Aniline + 2-MethoxyEthanol + Carbon Tetrachloride

1:3	0.0167	2.7549	2.6210	0.1824	2.1435	19.48	27.52	21.27	12.50	14.22
1:2	0.0165	2.8977	2.6680	0.1976	2.3451	22.10	33.86	29.83	12.85	14.73
1:1	0.0159	2.9743	2.6389	0.2949	2.3941	25.24	40.73	36.18	12.93	15.82
2:1	0.0154	2.9515	2.6373	0.2536	2.4144	24.57	36.12	32.85	12.63	14.35
3:1	0.0151	2.8156	2.6432	0.1873	2.4948	20.65	30.17	25.36	12.44	14.11

Aniline + 2-MethoxyEthanol + 1,4-Dioxane

1:3	0.0258	2.6024	2.8317	0.0313	2.3010	20.39	24.12	21.72	10.94	11.23
1:2	0.0258	2.6306	2.8329	0.0291	2.3101	22.54	27.53	26.89	11.36	11.53
1:1	0.0245	2.8698	2.8810	0.0396	2.3764	28.11	42.19	34.56	11.47	11.59
2:1	0.0237	2.7837	2.8790	0.0375	2.3639	26.89	37.54	31.23	11.24	11.31
3:1	0.0232	2.6527	2.8562	0.0347	2.3547	24.63	31.17	23.64	10.76	11.18

Aniline + 2-MethoxyEthanol + Toluene

1:3	0.0307	2.5321	2.3285	0.1639	2.2073	18.26	17.59	20.58	13.37	15.63
1:2	0.0302	2.6513	2.4725	0.1702	2.2127	21.47	18.78	23.76	13.75	16.38
1:1	0.0292	2.8297	2.5401	0.2391	2.2281	23.85	36.17	32.15	13.89	16.53
2:1	0.0282	2.7412	2.4947	0.1810	2.2327	21.63	31.84	26.74	13.58	16.25
3:1	0.0278	2.6425	2.3610	0.1739	2.2652	19.79	27.56	21.58	13.26	15.47

Table 2. Value of dielectric constants and relaxation times and activation energy for the various weight fractions of the mixture of Aniline with 2-ethoxy Ethanol in different solvents

Ratio	Weight fraction W_2	ε_0	ε'	ε''	ε_∞	Relaxation Time (ps) using Higasi's Method			Activation energy	
						τ_1	τ_2	τ_0	$\square\square\square\square$	$\square\square f\square$
Aniline + 2-Ethoxy Ethanol + Benzene										
1:3	0.0309	2.7354	2.3917	0.1632	2.2576	21.11	35.00	27.18	14.27	17.96
1:2	0.0209	2.8525	2.3954	0.1889	2.2586	23.95	40.22	31.03	14.62	18.58
1:1	0.0313	2.9703	2.3962	0.2057	2.2612	26.43	46.38	35.01	14.78	18.64
2:1	0.0314	2.8525	2.3942	0.1874	2.2642	25.04	40.65	31.9	14.48	18.43
3:1	0.0311	2.7483	2.3922	0.1646	2.2667	22.83	35.95	28.65	14.11	17.68

Aniline + 2-Ethoxy Ethanol + Carbon Tetrachloride										
1:3	0.0174	2.6179	2.1635	0.0192	2.0935	21.91	35.58	35.12	12.26	14.37
1:2	0.0115	2.7329	2.2717	0.0218	2.1832	25.58	41.37	48.80	12.63	14.56
1:1	0.1728	2.9613	2.4231	0.0305	2.3241	27.34	48.40	55.46	12.75	15.11
2:1	0.0172	2.8725	2.3857	0.0272	2.4351	26.35	45.36	51.45	12.49	14.43
3:1	0.0171	2.6952	2.2086	0.0197	2.5687	23.17	37.18	41.71	12.14	14.32
Aniline + 2-Ethoxy Ethanol + 1,4-Dioxane										
1:3	0.0268	2.1679	2.1635	0.0192	2.0915	21.81	35.58	29.01	10.84	11.23
1:2	0.0277	2.3329	2.2717	0.0217	2.1892	25.71	41.37	36.71	11.58	11.43
1:1	0.0245	2.6613	2.4231	0.0306	2.3251	27.24	48.40	42.46	11.81	11.65
2:1	0.0266	2.5725	2.3857	0.0272	2.4331	26.46	45.36	38.45	11.24	11.37
3:1	0.0263	2.4952	2.2086	0.0196	2.5647	23.27	37.18	32.80	10.65	11.2
Aniline + 2-Ethoxy Ethanol + Toluene										
1:3	0.0319	2.6125	2.43.17	0.1632	2.2413	20.36	38.74	25.24	13.26	14.93
1:2	0.0211	2.7319	2.4518	0.1798	2.2427	21.63	39.51	27.33	13.34	15.74
1:1	0.0317	2.8370	2.4759	0.2367	2.2531	25.34	45.73	34.09	13.57	15.88
2:1	0.0317	2.7529	2.4751	0.2351	2.2540	24.72	40.19	30.56	13.30	15.49
3:1	0.0549	2.6476	2.4389	0.1674	2.2656	21.54	39.18	26.38	13.17	14.85

Table 3. Value of dielectric constants and relaxation times and activation energy for the various weight fractions of the mixture of Aniline with 2-butoxy Ethanol in different solvents

Ratio	Weight fraction W_2	ε_0	ε'	ε''	ε_∞	Relaxation Time (ps) using Higasi's Method			Activation energy	
						τ_1	τ_2	τ_0	ΔH_ε	ΔH_f
Aniline + 2-Butoxy Ethanol + Benzene										
1:3	0.0340	2.7874	2.3936	0.1619	2.2681	22.46	40.43	30.14	14.17	17.23
1:2	0.0347	2.9179	2.3959	0.1850	2.2694	25.45	46.89	34.55	14.49	17.45
1:1	0.0361	3.1019	2.3974	0.2327	2.2633	30.11	50.32	38.93	14.68	17.52
2:1	0.0761	2.9637	2.3939	0.2072	2.2657	28.11	45.71	35.94	14.23	17.38
3:1	0.0383	2.8264	2.3909	0.1742	2.2673	24.56	41.55	31.94	14.08	17.10
Aniline + 2-Butoxy Ethanol + Carbon Tetrachloride										
1:3	0.0303	2.7450	2.4901	1.1753	2.2725	20.15	37.10	30.49	11.89	14.29
1:2	0.0191	2.9182	2.4941	0.2078	2.2740	23.51	40.94	33.74	12.26	14.91
1:1	0.0199	3.2019	2.4978	0.2683	2.2743	36.17	54.32	40.89	12.43	15.09
2:1	0.0207	2.9761	2.4952	0.2415	2.2750	24.73	50.18	37.21	12.08	14.45
3:1	0.0211	2.8146	2.4918	1.9420	2.2759	21.27	46.17	31.63	11.83	14.16
Aniline + 2-Butoxy Ethanol + 1,4-Dioxane										
1:3	0.0288	2.0218	2.2491	0.1790	2.2103	19.74	40.31	27.43	11.37	10.78

1:2	0.0294	2.1520	2.2512	0.1849	2.2217	23.18	44.58	32.25	11.55	11.21
1:1	0.0306	3.7138	2.2850	0.2914	2.2432	28.89	48.73	36.86	11.74	11.44
2:1	0.0318	2.6539	2.2733	0.2471	2.2411	26.42	45.20	33.74	11.43	11.10
3:1	0.0325	2.3131	2.2609	0.1632	2.2395	21.59	41.66	31.52	11.26	10.69

Aniline + 2-Butoxy Ethanol + Toluene

1:3	0.0349	2.5017	2.4690	0.1690	2.2503	20.73	40.31	28.43	12.78	15.08
1:2	0.0351	2.7529	2.4711	0.1749	2.2517	22.17	42.57	31.59	13.27	15.31
1:1	0.0365	3.9137	2.4759	0.2814	2.2532	27.80	46.74	35.86	13.38	15.43
2:1	0.0380	2.8531	2.4732	0.2371	2.2581	25.43	43.29	32.74	13.15	15.22
3:1	0.0387	2.5130	2.4709	0.1732	2.2595	20.50	41.60	31.57	12.74	14.82

In the this study we have been observed that, the dielectric constants, dielectric loss and relaxation time of ternary mixtures of aniline with 2-alkoxyethanols in different non polar solvents (benzene, CCl4, 1,4-dioxane and toluene) at room temperatures. The value of relaxation times τ_1, τ_2 and τ_0 for all the systems were calculated using the Higasi's method and are shown in Tables 1-3. In all the systems, the values of τ_2 are significantly higher than τ_1 and τ_0. Higher value of τ_2 indicates that the presence of intermolecular or overall molecular relaxation is larger in comparison to intramolecular or individual molecular relaxation in the systems [15]. It is observed that the value of relaxation times τ_1, τ_2 and τ_0 increases with increasing chain length of 2-alkoxyethanols with aniline in the selected solvents. Aniline suggests the solute-solvent interaction in such a way that, the ternary mixture produces a field in which effective dipoles rotates slowly field will resist the rotation of the molecule this may be due to the strength of heterogeneous H-bond increases. Similar results are predicted by Sengwa et al., [16] and Nimkar et al., (2005) [17].This indicates that there is a formation of hydrogen bond between the OH group of 2-alkoxyethanols and the N-H group of aniline.In the system, the complex formation between solute-solute molecules is likely by involving 2-alkoxyethanols with aniline in which a hydrogen bond formed would be between an hydroxylic oxygen and a nitrogen atom of the type =N– enter into complexation components.

Solvents play an important role in the areas of chemical synthesis, as thinners in paint industry, in inks, and in varnishes and lacquers. There are two broad classifications of solvents they are;(i) polar solvents and (ii) non-polar. Which are classified based on their dielectric constant. Polar solvents have a strong dielectric constant and they have one or more electronegative atoms. In the present study, there are four non-polar solvents (benzene, CCl4, 1,4-dioxane and toluene) as they have low dielectric constants, which is less than 5 and are poor solvents for charged species such as anions. Bonds between atoms with similar electro negativities will lack partial charges. These lone pairs are being highly electronegative, polarizable with the neighbor protic substance, which leads to increasing relaxation time. In suitable combination of aniline and 2-alkoxyethanols having an NH group and OH group, there is a possibility of dipolar interaction with non-polar solvents (benzene, CCl4, 1,4-dioxane and toluene). Inter molecular H-bonds are possible between aniline with 2-alkoxyethanols in the solvents is of the order benzene <CCl4 < 1,4-dioxane < toluene.

The dielectric relaxation time shows continuous increase with chain length of 2-alkoxyethanols with aniline and offers barrier to the rotation of the molecule. The relaxation time increases with chain length is expected as hydroxyl group reorientation depends to some extent on the lengthof the 2-alkoxyethanols, and the viscosity of the liquid. Our result shows that the relaxation time is larger at 1:1 molar ratio of aniline with 2-alkoxyethanols. The relaxation time decreases for other molar ratios but it is higher than either of the components. In the present study the relaxation time for dilute solution of 2-alkoxyethanols were observed in the ranges between 22 and 54 ps. The result also shows that the molecular association between aniline and 2-alkoxyethanols is maximum at 1:1 (molar ratio) and lesser in other molar ratios. From this we conclude that the 1:1 complex is dominant in aniline + 2-alkoxyethanols systems.The relaxation time increases with increasing chain length of 2-alkoxyethonls. The relaxation times of aniline with 2-alkoxyethonls are in thefollowing order 2-methoxyethanol< 2-ethoxyethanol < 2-butoxyethonl.

At high concentration of aniline in the mixtures, there are large numbers of 2-alkoxyethonls molecules surrounding the aniline molecules. The associative OH molecules in ethonols act as proton donors enabling hydrogen bonding with aniline molecules. Thus dipole-dipole interaction occurs in such a way that effective dipole moment gets increased and linear α- multimers are formed [19]. The dipole-dipole interaction is the interaction of the –OH group of alcohol with N-H of aniline. At lower concentration of N-H in the mixtures, there are only a lesser number of OH molecules in 2-alkoxyethonls to enable dipole-dipole interaction

throughhydrogen bonding. As a result, intermolecular interaction is weak. The relaxation time increases with increasing alkyl chain length of 2-alkoxyethonls and acidity aniline indicating that the degree of cooperatively for reorientation of the molecules increases with increasing chain length and the bulk of cluster increases. These effects are due to: (i) the increase of viscosity as chain length increases and (ii) the increase of molecular size as the chain length increases [18-20]. The higher value of relaxation time observed for 2-butoxyethanols with aniline suggests that 2-butoxyethanols is more acidic than other selected 2- alkoxyethanol.

The molar free energy of activation for viscous flow Δf_η and the molar free energy of dielectric relaxation Δf_τ are calculated for aniline with 2- alkoxyethanol in different non-polar solvents (benzene, CCl4, 1,4-dioxane and toluene) and presented in Table 1-3. It is evident from our data that the $\Delta f_\eta > \Delta f_\tau$. This is in agreement with the fact that the process of viscous flow, whichinvolves both the rotational and translational forms of motion, faced greater interference from neighbors than dielectric relaxation, which takes place by rotation only [21]. Smyth et al. [22] pointed out that, the relaxation time of a proton donor increases as the acceptor ability of the solvent environment increases. Similarly for a given proton acceptor, the relaxation time must increase with the proton donor ability of the donor solute.

IV.   CONCLUSION

The hydrogen bonded complexes of aniline with 2-alkoxyethonls (2-methoxyethanol, 2-ethoxyethanol, 2-butoxyethonl) in a dilute non polar solvents benzene, CCl4, 1,4-dioxane and toluene using X-band microwave technique suggested by Higasi'set.al. The Different dielectric parameters like dielectric constant (ε') and dielectric loss (ε'') at microwave frequency, and static dielectric constant (ε_0) and dielectric constant (ε_∞) at optical frequency shows significant changes with concentration; the increasing chain length of 2-alkoxyethonls is of the order: 2-methoxyethanols < 2-ethoxyethanols < 2-butoxyethonls. The study also reveals solute-solvent interaction is in the order: benzene < ccl4 < 1,4-dioxane < toluene. The values of average relaxation time (τ_1), the overall dielectric relaxation (τ_2) and the mean relaxation time (τ_0) were found to be maximum at 1:1 molar ratios of aniline with 2-alkoxyethonls. From these studies, it may be concluded that the chain length of 2-alkoxyethonls aniline plays an important role in the determination of strength of hydrogen bond formation.

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