

## Materials Characterization Using Microwave Technique

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### ABSTRACT

Microwave is nonionizing energy. It does not alter the molecular structure of the materials. Microwave technique involves the measurements of impedance and power. The Ayurvedic medicines are becoming popular worldwide. Ayurvedic medicines includes different plants, minerals and materials of animal and marine origin. They are basically hybrid composite nanomaterials. In the present work interaction of -NH- amino, >C=O carbonyl, -OH hydroxyl, -CN cyanide and -COOH carboxyl groups, which are most common groups in ASAVA of Ayurvedic medicines, with ethanol has been studied using microwave technique. The static dielectric constant, dielectric relaxation time, Bruggeman factor and thermodynamic parameters were reported.

### KEYWORDS

A. Microwave; B. Hybrid Materials; C. Time Domain Reflectometry

### 1.INTRODUCTION

Ayurvedic medicines, 'ASAVA' are the self generated alcoholic preparation with 10% alcohol and 25% sugar. A variety of medicinal herbs and their leaves, flowers, fruits, peels, roots, gum and resins; gems, metals and colors are used in the production of Ayurvedic medicines. The Sanskrit (Indian Language) names of medicinal plants and metals have been used to indicate the standard names of asava. For example, the metal complex used in the production of Lohasava is a calx of iron. The Sanskrit name of calx of iron is Lohabhasma. Asava are prepared by Indian traditional methods.

The solute-solvent molecular interactions between Ayurvedic medicines, ASAVA and associative solvent Ethanol are presented in this paper. Ayurvedic medicines, 'ASAVA' having carbonyl (>C=O) and cyanide (C-N) functional groups are selected to study relative structural changes as well as change in molecular interactions with ethanol molecules.

## 2. EXPERIMENTAL

Ayurvedic medicines, Lodhrasava and Lohasava were obtained from Ayurved rasasala, Pune. Ethanol AR grade (Changshu Yangyuan Chemical, China) 99.9% purity was obtained commercially and used without further purification. The solutions were prepared at different volume percentage of Lodhrasava in Ethanol and Lohasava in Ethanol in steps of 10 vol. %, within a  $\pm 0.01$  % error limit.

The complex permittivity spectra were studied using time domain reflectometry. The Hewlett Packard HP 54750A-sampling oscilloscope [1] with an HP 54754A TDR plug-in module [2] was used. A fast rising step voltage pulse of about 25 ps rise time generated by a tunnel diode was propagated through a flexible coaxial cable. The asava was placed at end of the coaxial line in the standard military application (SMA) coaxial cell of 3.5 mm outer diameter and 1.35 mm effective pin length.

All measurements were done under open load conditions. The change in pulse on reflection from the sample placed in cell was monitored by the sampling oscilloscope. The reflected pulse without sample  $R_1(t)$  and with sample  $R_x(t)$  were digitized in 1024 points and stored on disc.

The temperature controller system with a water bath and a thermostat has been used to maintain the constant temperature within the accuracy limit of  $\pm 1^\circ\text{C}$ . The sample cell was surrounded by an insulating container through which, the constant temperature water was circulated.

## 3. DATA ANALYSIS

The time dependent data were processed to obtain complex reflection coefficient spectra  $\rho^*(\omega)$  using Fourier transformation (Samulon [3]; Shannon [4]) as

$$\rho^*(\omega) = \frac{c p(\omega)}{j\omega d q(\omega)}$$

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 the angular frequency, and  $d$  is the effective pin length.

The complex permittivity spectra  $\varepsilon^*(\omega)$  were obtained from reflection coefficient spectra  $\rho^*(\omega)$  by using the bilinear calibration method (Cole [5] et al.). A sample  $\varepsilon^*(\omega)$  spectrum for Lodhrasava-Ethanol system at  $20^\circ\text{C}$  is shown in figure 1. The Cole-Cole plot for Lodhrasava and Ethanol system at  $20^\circ\text{C}$  is shown in figure 2. The Cole-Cole [7] plot follows the Debye semicircle, which indicates that relaxation process in Asava-Ethanol system can be explained with single relaxation time. The experimental values of  $\varepsilon^*(\omega)$  are fitted with the Debye equation (Havriliak and Negami [6]; Cole and Cole [7]; Davidson and Cole [8])

$$\varepsilon^*(\omega) = \varepsilon_\infty + \frac{\varepsilon_0 - \varepsilon_\infty}{(1 + j\omega\tau)}$$

with  $\epsilon_0$ ,  $\epsilon_\infty$  and  $\tau$  as fitting parameters. A nonlinear least- squares fit method (Bevington [9]) was used to determine the values of dielectric parameters. The values of static dielectric constant( $\epsilon_0$ ) and dielectric relaxation time( $\tau$ ) are listed in table I&II.

The modified Bruggeman [10] mixture formula, given below, is used to fit experimental data.

$$f_B = \left( \frac{\epsilon_{om} - \epsilon_{ow}}{\epsilon_{os} - \epsilon_{ow}} \right) \left( \frac{\epsilon_{os}}{\epsilon_{om}} \right)^{1/3} = 1 - [a - (a - 1) * V] * V = 1 - f(V)$$

with “a” as a fitting parameter and ‘f’ can be interpreted as fraction of effective volume V. The relative change in value of “a” reveals amount of interaction between solute and solvent. The values of Bruggeman factor are listed in table III & IV.

The thermodynamic parameters [11] molar enthalpy of activation  $\Delta H$  and the molar entropy of activation  $\Delta S$  were obtained using equation

$$\tau = \frac{h}{KT} \exp[(\Delta H - T\Delta S) / RT] \quad \text{and are listed in table V.}$$

#### 4. RESULTS AND DISCUSSION

The static permittivity is measure of effective dipole moment per unit volume in mixture. Relaxation time of biological material can be related to the size of molecule, mobility of molecules in liquid, molecular volume, viscosity, and temperature. Decrease in relaxation time can be correlated to decrease in size of molecule as well as to increase in mobility of molecules in liquid. If the polar solute molecules are spherical, and large by comparison with the solvent molecules, then the orientation relaxation of the solute molecules can usefully be described using Debye’s model. In this model the dipolar solute molecules are considered as spheres whose rotation is opposed by the viscosity of the surrounding solvent medium.

When value of numerical fitting parameter “a” is unity, modified Bruggeman mixture formulae reduces to original Bruggeman mixture formulae. Decrease in value of “a” below unity shows increase in effective volume fraction of solvent in mixture.

The value of activation enthalpy ( $\Delta H$ ) gives an idea about nature of compactness in molecules of liquid. The variation in ( $\Delta H$ ) with change in solute concentration provides us information about relative change in amount of hydrogen bonding in mixture.

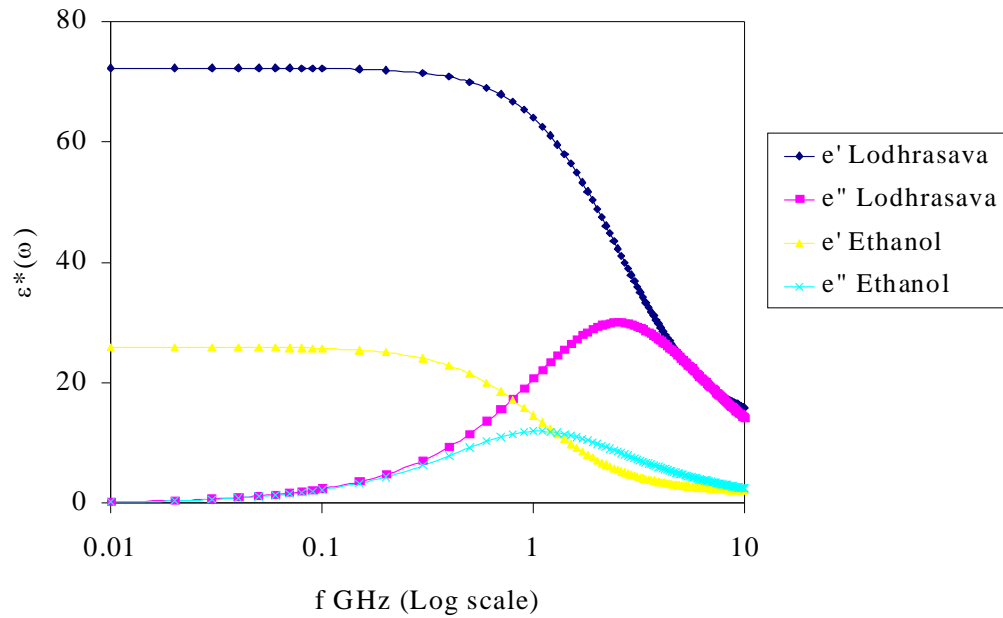
#### 5. CONCLUSION

The static permittivity increases with addition of *ASAVA* in ethanol. Decrease in permittivity with increasing temperature is also observed for all concentrations. This raise in permittivity with increasing amount of solute indicates linear increase in effective dipole moment of the system.

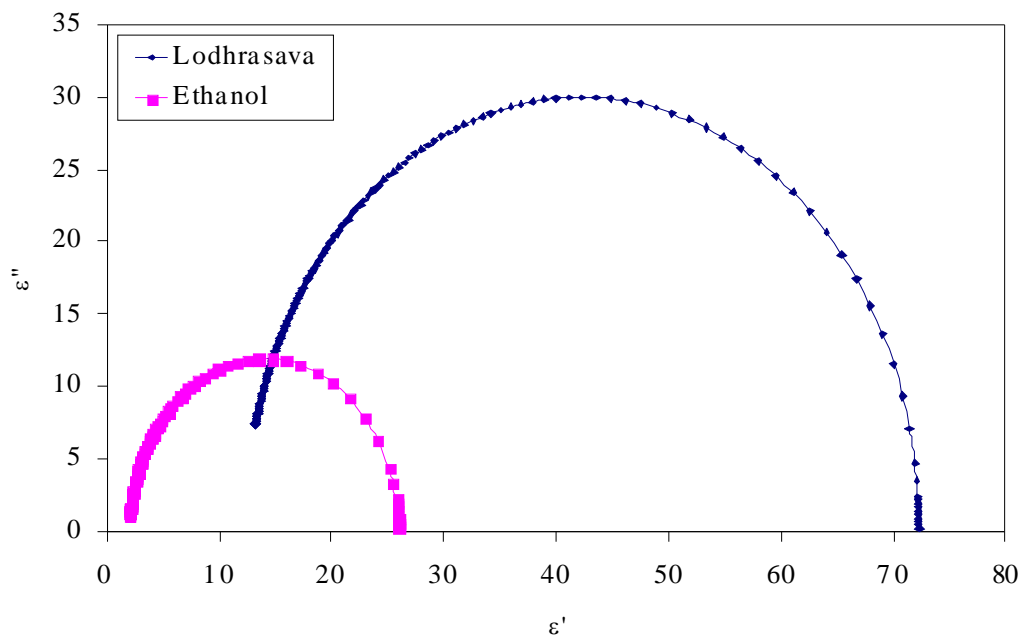
The variation in relaxation time shows that, the decrease in amount of hydrogen bonding between solute and solvent molecules, which leads to smaller molecular structures rotating fast.

The values of numerical fitting parameter 'a' are below unity shows increase in effective volume fraction of solvent in mixture, with decreasing size of *ASAVA* showing weaker solute solvent interactions.

The activation enthalpy ( $\Delta H$ ) decreases with increase in volume fraction of *ASAVA* in mixture, which shows decrease in amount of hydrogen bonding between molecules of liquid.



**Fig.1:**  $\epsilon^*(\omega)$  spectrum of Lodhrasava-Ethanol at 20<sup>0</sup>C.



**Fig.2:** Cole-Cole plot for Lodhrasava-Ethanol at 20<sup>0</sup>C.

**Table I:** Temperature dependent dielectric parameters for

Lodhrasava-Ethanol mixture

Volume fraction	10 <sup>0</sup> C		20 <sup>0</sup> C		30 <sup>0</sup> C		40 <sup>0</sup> C	
	$\epsilon_0$	$\tau$ ps	$\epsilon_0$	$\tau$ ps	$\epsilon_0$	$\tau$ ps	$\epsilon_0$	$\tau$ ps
0.0	26.85	167.04	25.91	150.77	25.23	128.08	24.52	111.29
0.1	31.00	147.25	30.43	125.87	29.39	106.55	28.37	95.73
0.2	35.96	119.40	35.17	96.69	34.57	91.73	33.74	85.87
0.3	40.95	105.41	40.19	93.01	39.08	94.91	38.12	78.13
0.4	46.74	98.15	45.34	85.97	44.03	78.22	43.44	71.01
0.5	52.67	87.56	51.36	79.83	50.11	69.05	49.14	64.33
0.6	58.08	83.83	55.64	74.83	54.44	65.88	53.78	63.77
0.7	63.47	78.80	60.88	67.63	59.61	24.91	58.76	62.17
0.8	70.58	68.34	68.38	65.65	66.44	62.07	64.16	61.13
0.9	71.85	67.16	70.03	64.27	68.29	61.13	67.57	59.99
1.0	74.16	65.00	72.27	63.65	69.57	60.41	68.33	59.70

**Table II:** Temperature dependent dielectric parameters for

Lohasava-Ethanol mixture

Volume fraction	10 <sup>0</sup> C		20 <sup>0</sup> C		30 <sup>0</sup> C		40 <sup>0</sup> C	
	$\epsilon_0$	$\tau$ ps	$\epsilon_0$	$\tau$ ps	$\epsilon_0$	$\tau$ ps	$\epsilon_0$	$\tau$ ps
0.0	26.85	167.04	25.91	150.77	25.23	128.08	24.52	111.29
0.1	32.31	129.28	31.09	117.08	30.25	105.04	29.24	88.97
0.2	37.32	104.83	36.30	94.73	35.43	81.43	34.38	71.48
0.3	42.25	91.23	41.56	78.62	40.80	68.98	39.40	64.08
0.4	48.80	79.37	48.07	64.01	46.95	61.08	44.54	57.70
0.5	55.45	71.63	54.46	56.33	53.22	47.75	50.56	43.22
0.6	62.25	59.13	60.23	52.34	57.70	39.45	55.55	33.53
0.7	69.82	49.54	67.17	40.27	64.77	32.13	62.23	30.65
0.8	73.87	42.00	72.25	31.12	69.66	26.90	68.69	26.78
0.9	79.01	26.69	76.35	22.32	75.70	20.48	73.07	20.00
1.0	82.89	22.09	81.21	21.79	80.80	19.95	77.66	19.10

Volume fraction of Lohasava	Ideal	10 <sup>0</sup> C, a=0.645		20 <sup>0</sup> C, a=0.649		30 <sup>0</sup> C, a=0.723		40 <sup>0</sup> C, a=0.734	
		Expt.	Theor.	Expt.	Theor.	Expt.	Theor.	Expt.	Theor.
0.0	1.00	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000
0.1	0.90	0.932	0.946	0.932	0.931	0.925	0.925	0.924	0.932
0.2	0.80	0.857	0.872	0.856	0.871	0.844	0.840	0.842	0.866
0.3	0.70	0.774	0.812	0.774	0.795	0.758	0.766	0.756	0.764
0.4	0.60	0.685	0.695	0.685	0.686	0.666	0.654	0.664	0.653
0.5	0.50	0.589	0.584	0.588	0.590	0.569	0.579	0.566	0.565
0.6	0.40	0.485	0.467	0.484	0.477	0.466	0.468	0.464	0.453
0.7	0.30	0.374	0.344	0.374	0.354	0.358	0.352	0.356	0.351
0.8	0.20	0.257	0.244	0.256	0.246	0.244	0.242	0.242	0.243
0.9	0.10	0.132	0.133	0.132	0.129	0.125	0.125	0.124	0.123
1.0	0.00	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000

**Table III:** Ideal, experimental and theoretical values of Bruggeman factor ( $F_B$ )  
 for Lodhrasava-Ethanol mixture.

Volume fraction of Lodhrasava	Ideal	10 <sup>0</sup> C, a=0.545		20 <sup>0</sup> C, a=0.554		30 <sup>0</sup> C, a=0.504		40 <sup>0</sup> C, a=0.497	
		Expt.	Theor.	Expt.	Theor.	Expt.	Theor.	Expt.	Theor.
0.0	1.00	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000
0.1	0.90	0.941	0.961	0.940	0.962	0.945	0.977	0.945	0.986
0.2	0.80	0.873	0.940	0.871	0.933	0.879	0.944	0.880	0.924
0.3	0.70	0.796	0.815	0.794	0.799	0.804	0.816	0.806	0.822
0.4	0.60	0.709	0.776	0.707	0.700	0.719	0.715	0.721	0.723
0.5	0.50	0.614	0.612	0.612	0.615	0.624	0.626	0.626	0.627
0.6	0.40	0.509	0.490	0.507	0.409	0.519	0.494	0.521	0.502
0.7	0.30	0.396	0.363	0.394	0.375	0.404	0.379	0.406	0.377
0.8	0.20	0.273	0.245	0.271	0.254	0.279	0.266	0.280	0.266
0.9	0.10	0.141	0.117	0.140	0.130	0.145	0.125	0.145	0.118
1.0	0.00	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000

**Table V:** Activation enthalpy and entropy of Asava-Ethanol

Volume fraction	Lodhrasava + Ethanol		Lohasava + Ethanol	
	$\Delta H$ KJ/mole	$\Delta S$ J <sup>0</sup> /K mole	$\Delta H$ KJ/mole	$\Delta S$ J <sup>0</sup> /K mole
0.0	7.6740	-0.0303	7.6740	-0.0303
0.1	8.2885	-0.0269	6.5431	-0.0321
0.2	5.2651	-0.0355	7.0945	-0.0285
0.3	4.8306	-0.0363	6.3258	-0.0298
0.4	5.3865	-0.0337	4.9931	-0.0331
0.5	5.4115	-0.0328	9.9997	-0.0148
0.6	4.5427	-0.0354	9.9111	-0.0061
0.7	3.1175	-0.0397	9.8678	-0.0121
0.8	1.3147	-0.0487	8.6846	-0.0145
0.9	0.2934	-0.0486	4.6080	-0.0254
1.0	0.2061	-0.0502	3.3723	-0.0357

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