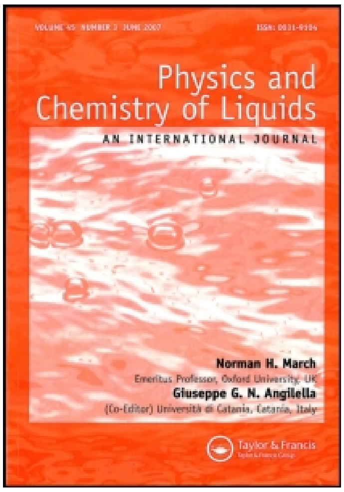

Physics and Chemistry of Liquids

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

Study of Dielectric Response of Aqueous Solutions of Copper Sulphate Using Microwave Cavityspectrometer

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To cite this Article Johri, G. K. , Gupta, D. C. , Johri, M. and Roberts, James A.(2002) 'Study of Dielectric Response of Aqueous Solutions of Copper Sulphate Using Microwave Cavityspectrometer', Physics and Chemistry of Liquids, 40: 1, 1 — 7

To link to this Article: DOI: 10.1080/00319100208086645

URL: http://dx.doi.org/10.1080/00319100208086645

Phys. Chem. Liq., 2002, Vol. 40(1), pp. 1–7

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STUDY OF DIELECTRIC RESPONSE OF AQUEOUS SOLUTIONS OF COPPER SULPHATE USING MICROWAVE CAVITY SPECTROMETER

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(Received 13 June 2000)

Dielectric relaxation study of aqueous solutions of copper sulphate ($CuSO_4$) using microwave cavity spectrometer is reported. A cylindrical microwave cavity operated in TM_{010} mode was loaded. The variations of the width of the resonance profile and shift of the resonance frequency were achieved by changing the volume of the sample in the cavity as a function of probe length at 29.567 GHz. Slater perturbation equations have been used to analyse the data for permittivity, dielectric loss and relaxation time. This work provides information about ion–ion interactions.

Keywords: Aqueous solutions; Dielectric relaxation; Copper sulphate; Microwave cavity

1. INTRODUCTION

The ionic property and relaxation processes within a sample of material loading a resonant cavity can be related to the change in the resonant frequency and change in the quality factor Q of the cavity when samples are put in place. The microwave cavity spectrometer has been used [1–6] due to its advantage for instant accuracy and

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ISSN 0031-9104 DOI: 10.1080/00319100290005631

the quantity of sample needed is very small. We have measured the width of the resonance profile, shift in the resonance frequency and relaxation time for aqueous solutions of copper sulphate ($CuSO_4$) and pure water using microwave cavity spectrometer at 29.567 GHz for different sample volumes by advancing the sample holder in the cavity at room temperature (298°K) and different concentrations.

2. EXPERIMENTAL

The aqueous solutions of copper sulphate are prepared in units of gm/cm^3 in the range of 0.00033 to 0.1248 gm/cm^3. The apparatus used to conduct the experiment has been discussed elsewhere [1, 2].

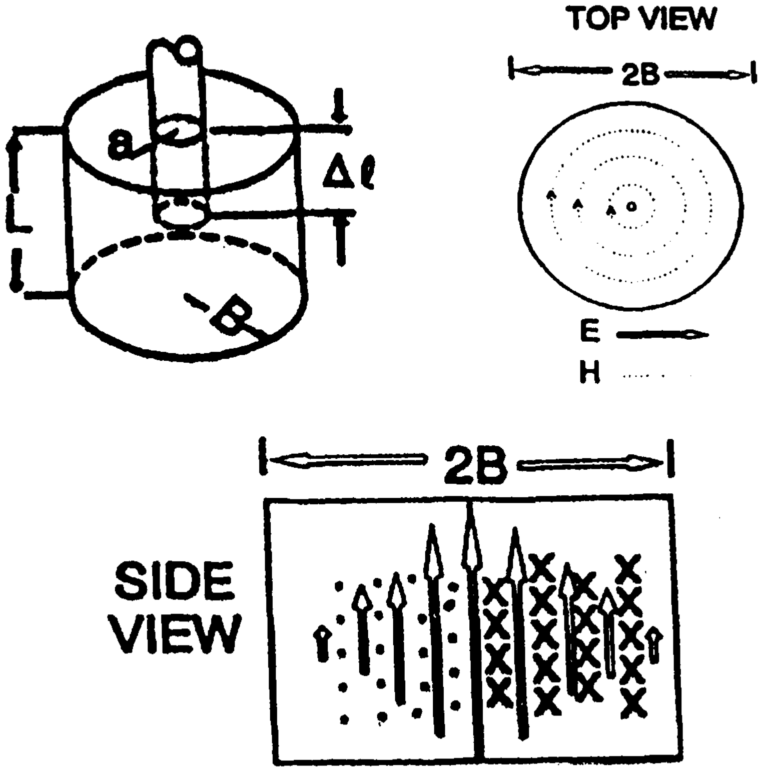

FIGURE 1 A sketch of the resonance cavity with assumed fields in TM_{010} mode pattern. Δl is controlled by a micrometer drive mechanism attached to the exterior of the cavity.

The addition of a calibrated micrometer drive to advance the sample into the cavity in known increment has enabled the volume dependency of frequency shift and resonance width. A cylindrical microwave cavity is shown in Figure 1. Each sample of the solution studied is placed in a thin walled pyrex test tube of 1.5 mm diameter. The samples were then advanced into the microwave cavity which is operated in TM_{010} mode. Measurement of amplitude of resonance profile, shift in resonant frequency and width of resonance profile were measured for each inserted length of the pyrex tube in the cavity. The frequency chosen to see the change in dielectric response was near 29.567 GHz with respect to the frequency of the driving microwave field.

The relative behavior of the permittivity ε' and the dielectric loss ε'' can be obtained from the measured frequency change (Δf) and the change in the width of resonance profile (Δw) at half power maximum *verses* depth of penetration of the sample into the resonant cavity. The trend of variation in the two parameters ε' and ε'' can be compared *via* the loss-tangent for each sample obtained. The relaxation time is deduced from $\tan\delta$ using Debye's mechanism.

3. DATA ANALYSIS

The Slater perturbation equations [4] for a resonant microwave cavity, which were used to analyse the data can be written in the following forms, after real and imaginary terms have been separated

$$\frac{\Delta f}{f_0} = -\left[\frac{(\varepsilon' - 1)}{2}\right] F(\mathbf{E}) \tag{1}$$

for the real part of the frequency shift and,

$$\Delta\left(\frac{1}{Q}\right) f_0 = \frac{\Delta w}{f_0} = \varepsilon'' F(\mathbf{E}) \tag{2}$$

for the imaginary part of the frequency shift. Hence, the loss tangent can be written as

$$\tan\delta = \frac{\varepsilon''}{\varepsilon'} \approx \frac{\Delta w}{2\Delta f}, \quad \text{for } \varepsilon' \gg 1. \tag{3}$$

Therefore the relaxation time is obtained from the following relation

$$\tau = \frac{1}{\omega} \times \left(\frac{\Delta w}{2\Delta f}\right) \tag{4}$$

where $\omega = 2\pi f$.

The form factor $F(\mathbf{E})$ can be expressed as

$$F(\mathbf{E}) = \frac{\int_v \mathbf{E}_s \cdot \mathbf{E}_a dv}{\int_v \mathbf{E} \cdot \mathbf{E}_a dV} \tag{5}$$

where Δf is the frequency shift, Δw is the change in the resonance width. $F(\mathbf{E})$ is the functional form of the field interacting with the

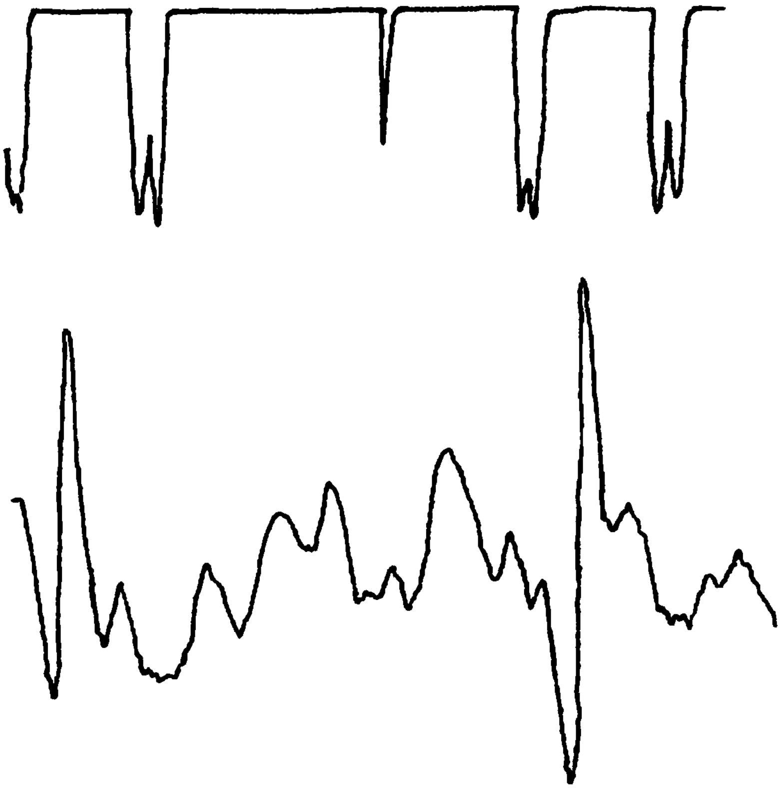

FIGURE 2 A dual-pen chart recorder trace of the second order derivative of cavity resonance.

sample loading the cavity and energy stored per cycle in the cavity. v is the sample volume, V is the cavity volume, $\mathbf{E}_s$ the electric field in the sample, $\mathbf{E}_a$ is the unperturbed radiation component of the electric field in the cavity volume before the sample is in place and $\mathbf{E}$ is the electric field applied to the cavity.

Frequency calibration was achieved by the use of a general radio frequency standard set composed of an 1112A and 1112B system good to $1/10^6$. Thus, the frequency measurements are accurate to a few KHz for each reading of the chart on the dual-pen chart recorder. The data were taken by displaying the cavity resonance profile on one scan of a dual trace chart recorder while calibration markers generated from a mixer-multiplier displayed on the other scan. Each frequency interval was partitioned by using an interpolation radio receiver set for the desired frequency interval in this experiment. The separation of the markers was set at 4 MHz. The center frequency of the resonance was set at $f_0 \pm \Delta f$, where the range of Δf was determined by how much shift ensued when each sample was placed into the cavity. A permanent record of the signal and markers were produced on a dual-pen chart recorder as shown in Figure 2. The overall uncertainty of the measurements is less than 5%.

4. RESULTS AND DISCUSSION

The data of the amplitude of the resonance profile (Δh), shift in resonance frequency (Δf), width of resonance profile (Δw) and relaxation time (τ) for aqueous solution of copper sulphate ($CuSO_4+H_2O$) for concentrations 0.00033, 0.045, 0.075, 0.10 and 0.1248 gm/cm^3 and pure water are plotted as a function of sample volume measured by changing the probe length. It is revealed from the Figures 3(a)–(f) that (Δh), (Δf) and (τ) have non-linearities, however nearly opposite trend of variation between (Δh) and (Δf) is observed. The variation in the width of resonance profile (Δw) keeps its value to be nearly constant for aqueous solutions of copper sulphate while there is non-linearity observed in the case of pure water, which may be due to ion–ion interactions.

From the work reported in this paper it is obvious that the sample volume in the microwave cavity makes a significant change in the

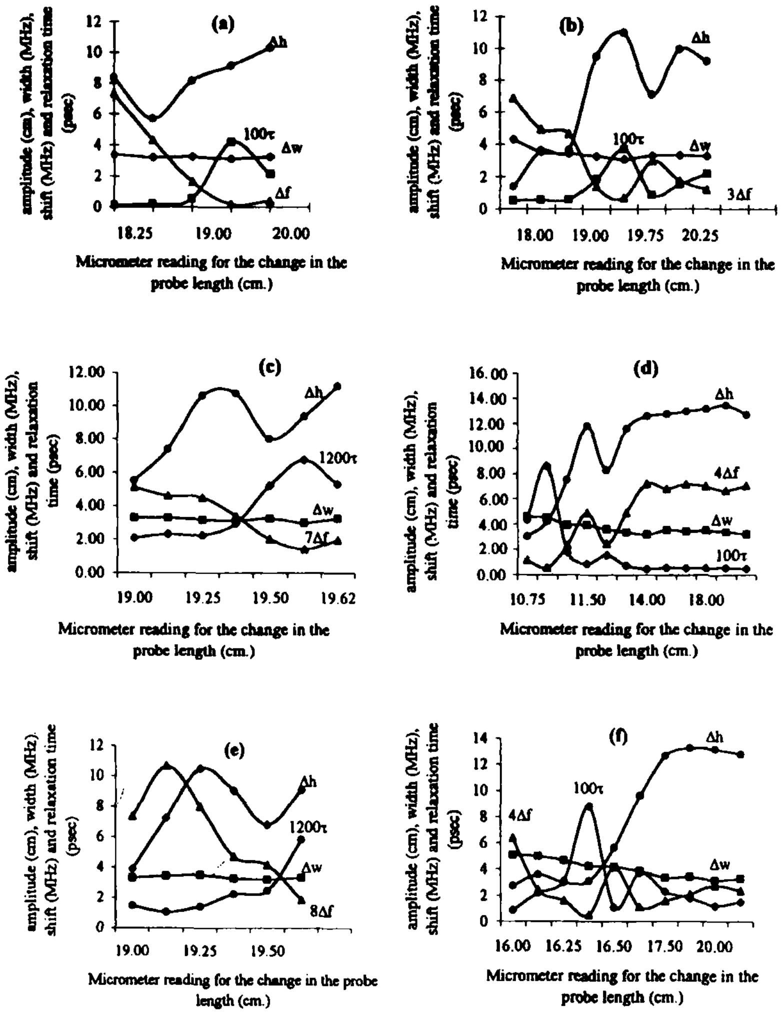

FIGURE 3 Variation in the amplitude (Δ*h*), width (Δ*w*), shift (Δ*f*) and relaxation time (τ) at different probe length for different concentrations in gm/cm^3 (a) $CuSO_4 + H_2O$ at 0.00033 gm/cm^3; (b) $CuSO_4 + H_2O$ at 0.045 gm/cm^3; (c) $CuSO_4 + H_2O$ at 0.075 gm/cm^3; (d) $CuSO_4 + H_2O$ at 0.10 gm/cm^3; (e) $CuSO_4 + H_2O$ at 0.1248 gm/cm^3; (f) pure water.

dielectric parameters ε' and ε'' which are equivalent to shift in the resonance frequency and width of resonance profile following the Slater perturbation equations. The microwave cavity spectrometer is a good technique to make relative measurements of ε' and ε'' but exact values of these parameters need evaluations of form factor $F(\mathbf{E})$. This technique gives relaxation time using Debye mechanism by Eq. (4). The ion-ion and ion-solvent interactions give nonlinear characteristics as a function of sample volume even for small perturbations in loading the cavity. The investigations in this paper are fruitful as dielectric response is accurately known and information about interactions is qualitatively achieved.

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