CHAPTER - IV

ULTRASONIC VELOCITY AND ABSORPTION STUDIES IN AQUEOUS SOLUTIONS OF SODIUM TAUROCHOLATE

4.1 INTRODUCTION:

In aqueous solutions, the surfactant molecules have a tendency to collect at the interfaces. At very low surfactant concentrations of 10^{-4} - 10^{-2} mol dm^{-3}, the behaviour of ionic surfactants in aqueous solutions resembles that of strong electrolytes. On increasing the concentration of surfactant, however, a pronounced deviation from the `ideal' behaviour is observed. This behaviour is attributed to the association of the surfactant monomers to form high molecular weight aggregates, known as micelles. The concentration at which micelles form is called the Critical Micelle Concentration (CMC). When micelles are formed in aqueous solutions, the CMC is one of the most readily obtainable parameters [1,2]. It is now generally accepted that below CMC only monomers are present in the solutions whereas above CMC both the monomers and the micelles are present in the solutions and the monomeric surfactant concentration is reasonably constant [2].

The bile salts are naturally occuring detergents which form miceller aggregates in aqueous solutions [3]. Bile salts are synthesised in the liver of human beings. They form aggregates (micelles) which help to solubilize and disperse dietary lipids in the small intestine. Bile salts have also been studied in recent years as alternatives to

conventional detergents for chemical analysis. Specific areas of application include chemical separations [4] and luminescence analysis [5]. The bile salt micelles are smaller and more rigid than those of conventional detergents resulting in unique aggregation behaviour with respect to self association as well as solubilisation of hydrophobic molecules in aqueous solutions [6-8]. Bile salts are nearly flat molecules with a hydrophobic and a hydrophilic surface. Oakenfull and Fisher [9] have carried out equivalent conductance and apparent molar volume studies on aqueous and aqueous-ethanolic solutions of sodium cholate, sodium deoxy cholate and sodium lithocholate. Their studies established that the first stage in the formation of bile salt micelles is the formation of hydrogen bonded dimers and the hydrogen bonding is the major interaction associated with the formation of dimers. Information about the formation and structure of these micelles is needed to develop a further understanding of the physiological role of the bile salts.

From a survey of literature available, it can be seen that most of the ultrasonic studies are only on aqueous and aqueous-alcoholic solutions of some ionic and non-ionic surfactants [9-17].

The ultrasonic velocity studies of Varma et al. [11] in aqueous solutions of barium soaps show that the ultrasonic velocity decreases below CMC with increase of soap concentration. Further increase of concentration of barium soaps above CMC, results in an increase of the ultrasonic velocity, thus exhibiting a break or change of

slope in the velocity concentration curve at CMC. The observed decrease in the compressibility and free length of the soap solutions above CMC has been explained as due to the fact that ions in the solution are surrounded by a layer of solvent molecules firmly bound and oriented towards the ion by the influence of electrostatic field of ions. This orientation results in an increase of internal pressure and in the lowering of compressibility. A similar variation of the ultrasonic velocity with soap concentration was reported in the solutions of praseodymium linoleates, neodymium linoleates [20] and thorium soaps in benzene-methanol mixture [12] and in aqueous solutions of alkaline earth metal butyrates [21].

The ultrasonic relaxation studies of Aicart et al. [16] on mixed micelles of the cationic surfactant Decyltrimethylammonium bromide (DTAB) and alcohol shows that the micelle kinetics of DTAB is affected by the presence of propanol. Their results indicate that the exit rate of DTAB monomer from micelles decreases with the addition of propanol. This behaviour is believed to be due to the decrease in the charge density at the surface of the mixed micelle which may be due to the presence of propanol around the surface of the micelle. A similar relaxation studies of Kato et al. [10] in octyl, decyl, and tetradecyltrimethylammonium bromide in aqueous solution over the frequency range 0.2 - 210 MHz revealed that the ultrasonic relaxation spectra show a single relaxation process for all the solutions and at all concentrations

investigated. The observed relaxation process was ascribed to fast relaxation due to an exchange process of a surfactant monomers between micelle and the surrounding bulk solution.

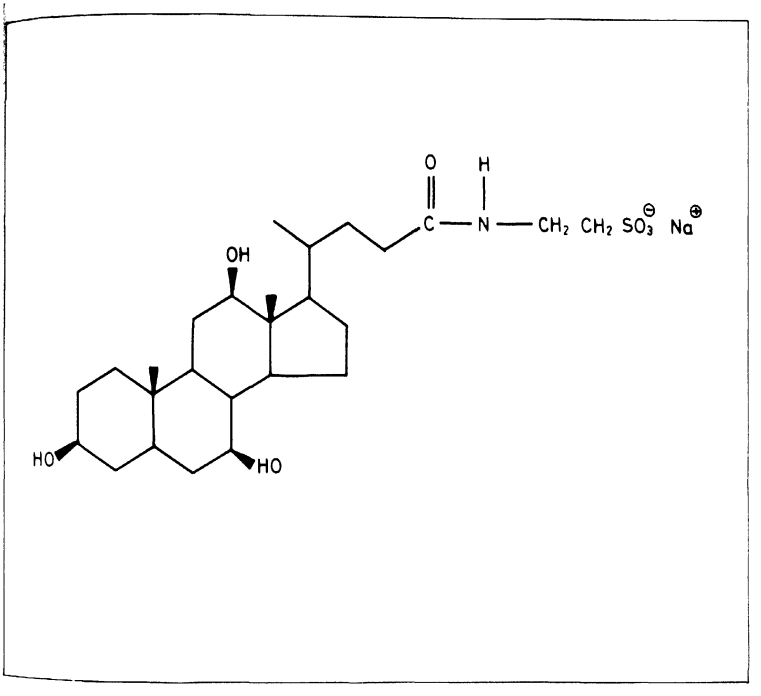
4.2 Estimation of kinetic parameters for micellar formation:

The kinetic parameters for the fast exchange process, described above can be estimated by applying Teubner theory [18].

Teubner derived the following expression for the relaxation time of the fast ultrasonic relaxation due to surfactant monomer exchange process between micelles and the bulk solution, using approximations introduced by Aniansson and Wall [19,25]

$$\frac{1}{\tau} = 2\pi f_r = \frac{k_{-1}}{\sigma^2} + \frac{k_1}{m}\left[\frac{C}{C_1} - 1\right] \qquad (4.1)$$

Where C and C_1 represent the total and monomer concentrations of surfactant, respectively, and usually C_1 is assumed to be equal to critical micelle concentration. The symbols m and σ^2 represent the mean aggregation number and the variance of size distribution on proper micelles, respectively, and k_{-1} is the mean of the dissociative rate constant and k_1 is the associative rate constant for the exchange process. He further derived an expression for the absorption maxima per wavelength $(\alpha\lambda)_{max}$, as follows



FIG·4·1. SODIUM TAUROCHOLATE

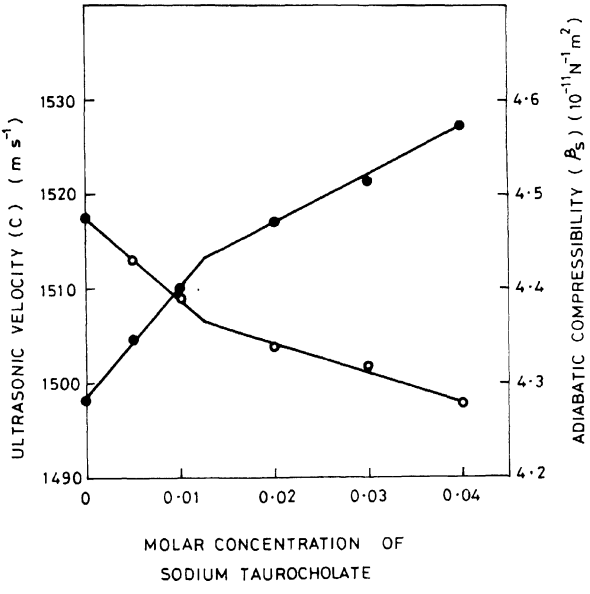
$$(\alpha\lambda)_{max} = \frac{\pi \rho v^2 (\Delta V_s)^2 C_1 \frac{\sigma^2}{m} (C/C_1 - 1)}{2RT\left\{ 1 + \frac{\sigma^2}{m} \left(\frac{C}{C_1} - 1 \right) \right\}} \qquad (4.2)$$

where $(\alpha\lambda)_{max}$ is defined as equal to 1/2 Av f_r. Here ΔV_s represents the change in the molar volume due to the exchange process, ρ is the density of the solution, R is the gas constant and T is the temperature.

An exmamination of equation (4.1) indicates that a graph of $1/\tau$ vs. (C/C_1-1) should give a linear correlation with the intercept and slope of the line providing values for k_{-1}/σ^2 and k_{-1}/m respectively.

If the values of CMC and the micellar aggregation number are available, kinetic parameters k_{-1} and σ^2 can be estimated from the values of $k_{-1}/\sigma^2$and k_{-1}/m. The values of the associative rate constant k_1 is further estimated on the basis of the assumption, $k_1=k_{-1}/C_1$.

The ultrasonic studies on aqueous solutions of biological surfactants, such as bile salts are scanty and hence it may be worthwhile to study the molecular interactions in these solutions using a non-destructive technique like ultrasonic method. Therefore, the present ultrasonic velocity and absorption studies have been undertaken in order to investigate the kinetics of micellar formation in the aqueous solutions of sodium taurocholate in the molar concentration ranging from 0.01 to 0.04. The ultrasonic velocity studies were carried out at 2 MHz frequency using multi-frequency variable path ultrasonic



FIG·4·2. ULTRASONIC VELOCITY AND ADIABATIC COMPRESSIBILITY VS CONCENTRATION OF SODIUM TAUROCHOLATE IN WATER

interferometer. The absorption measurements were done over a frequency range of 2-30 MHz and at a fixed temperature of 298 K. The temperature of the solutions were maintained within ±0.1K. The ultrasonic absorption studies were carried out using MATEC MBS-8000 measurement system.

4.3 RESULTS AND DISCUSSION:

4.3.1 Ultrasonic velocity studies:

Aqueous solutions of sodium taurocholate were prepared by taking AR (BDH) chemicals and ultrasonic velocity and absorption measurements were carried out in various solute concentration ranging from 0.01 molar to 0.04 molar at a fixed temperature of 298K. The ultrasonic velocity was determined at 2 MHz frequency using multi frequency variable path interferometer as described in chapter-II. The densities of the solutions were determined using a graduated dilatometer as described in chapter -II. From the measured values of velocity and density, the parameters like adiabatic compressibility and intermolecular free length are computed using the standard relations given in chapter - I. The ultrasonic velocites, adiabatic compressibilities and intermolecular free lengths of the aqueous solutions of sodium taurocholate in the molar concentration range 0.01 - 0.04. are given in table 4.1. The variation of ultrasonic velocity and adiabatic compressibility with solute concentration is shown graphically in figure 4.2.

From the figure 4.2, it can be seen that the ultrasonic velocity in aqueous solutions of sodium taurocholate increases with increasing concentration. The variation of ultrasonic velocity with concentration exhibits a change in slope at 0.013 molar concentration of sodium taurocholate.

The adiabatic compressibility of the aqueous solutions of sodium taurocholate decreases with increase in concentration of sodium taurocholate in the concentration range 0.01 molar to 0.04 molar. The compressibility vs concentration curve also exhibits a change in slope at 0.013 molar sodium taurocholate similar to the velocity curve.

The variation of intermolecular free length in the concentration range 0.01 to 0.04 molar of sodium taurocholate given in table 4.1 shows that the free length decreases with increasing concentration of sodium taurocholate.

The salient features of the velocity studies are given as follows:

1. The ultrasonic velocity increases with increasing concentration of sodium taurocholate and exhibits a sharp change in slope at 0.013 molar concentration of sodium taurocholate.
2. The adiabatic compressibility decreases with increase in concentration of sodium taurocholate. Similar to the velocity behaviour, the adiabatic compressibility vs concentration curve also shows a

change in slope at 0.013 molar sodium taurocholate concentration.

3. The intermolecular free length decreases with increasing concentration of sodium taurocholate in the concentration range studied.

From the velocity graph (fig.4.2), it is observed that the ultrasonic velocity is higher in sodium taurocholate solutions as compared to water. The increase in ultrasonic velocity, generally, indicates interaction between solute and solvent molecules [22]. The association in the present case may be due to the hydrogen bonding between the free hydroxyl groups of sodium taurocholate with water. Further, the plots of ultrasonic velocity and adiabatic compressibility versus concentration are characterised by an intersection of two straight lines at a definite concentration of 0.013 molar which corresponds to the critical micelle concentration (CMC) of sodium taurocholate in water. A similar behaviour was also reported by Upadhyaya [20] in the solutions of praseodymium and neodymium linoleates in benzene-methanol mixture and by Mehrotra [21] in aqueous solutions of alkaline earth metal butyrates. The present study in aqueous solutions of sodium taurocholate shows that the CMC is 0.013 molar. This value of CMC is in good agreement with the CMC value of 8-12 millimolar reported by Meyerhoffer and McGown [23].

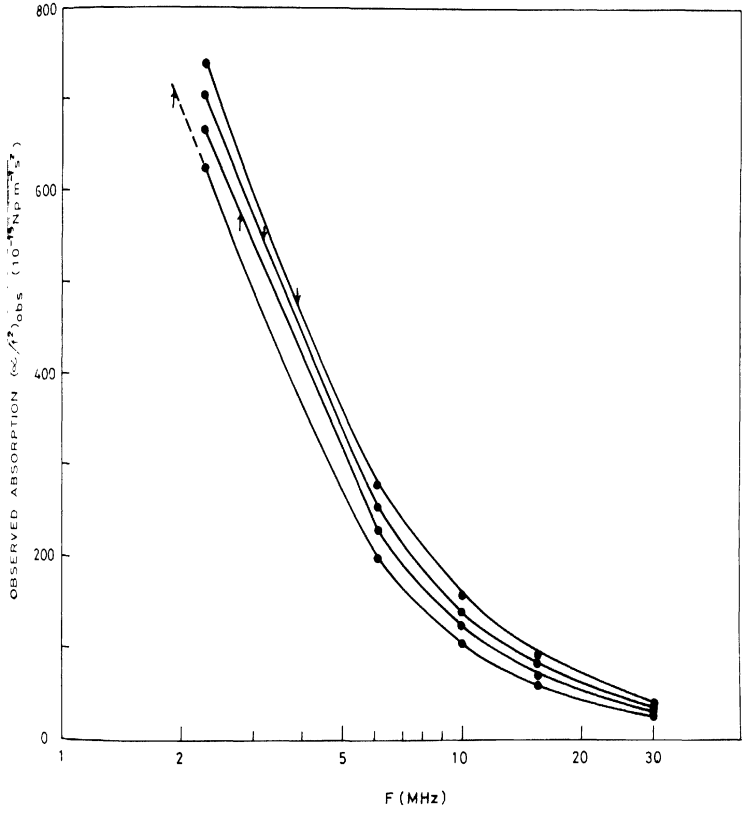
On increasing the concentration of sodium taurocholate above CMC, the ultrasonic velocity also increases. This may be due to the micelle formation

occuring in the aqueous solutions of sodium taurocholate. According to Oakenfull and Fisher [9], the first stage in the formation of bile salt micelle is the formation of hydrogen bonded dimers. Further aggregation of micelle can take place by polyfunctional hydrogen bonding. Since the dimers have residual hydrogen bond donor and acceptor groups, polyfunctional hydrogen bonding may be possible and this, perhaps leads to the formation of higher aggregates. This increased aggregation leads to an increase in ultrasonic velocity and decrease in adiabatic compressibility as reported by Mehrotra et al. [12] and Varma et al [11]. Such an increase in ultrasonic velocity is observed in the present case when the concentration of sodium taurocholate in water is increased above critical micelle comcentration value. Hence the explanation proposed above holds good for the present study also.

The intermolecular free length decreases with increase in concentration of sodium taurocholate in water. The decrease in intermolecular free length with concentration generally indicates strong interaction between solute and solvent molecules [24]. Hence the variation of free length with concentration of sodium taurocholate in water generally supports the explanation offered for the variation of ultrasonic velocity in the present system.

4.3.2 Ultrasonic absorption studies:

The ultrasonic absorption was measured over a wide frequency range of 2-30 MHz using MATEC MBS-8000 measurement system as described in chapter - II. These absorption data



FIG·4·3. OBSERVED ABSORPTION VS F(MHz) FOR AQUEOUS SOLUTIONS OF SODIUM TAUROCHOLATE AT DIFFERENT CONCENTRATIONS

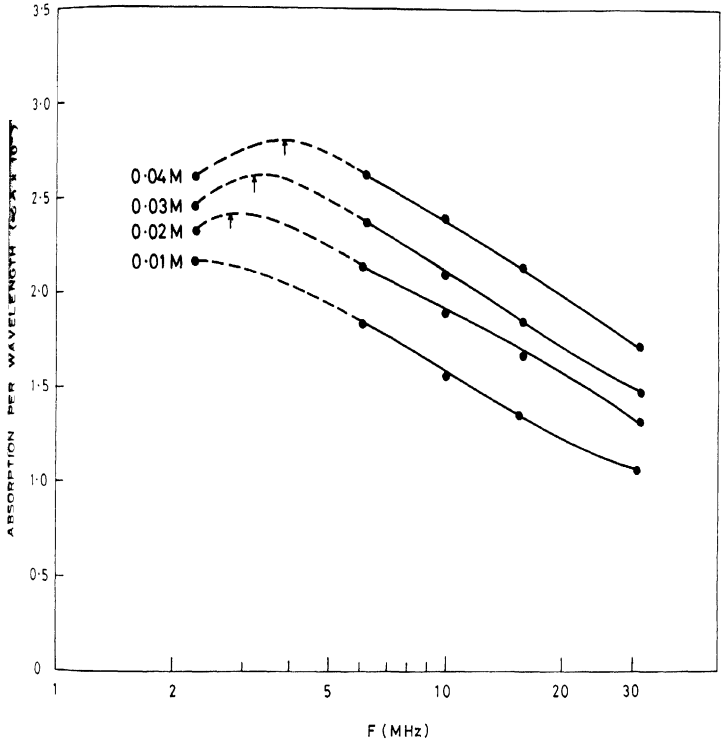


FIG · 4 · 4. ABSORPTION PER WAVELENGTH VS F(MHz) FOR AQUEOUS SOLUTIONS OF SODIUM TAUROCHOLATE AT DIFFERENT CONCENTRATIONS

were fitted to the equation (1.22) for a single relaxation in the frequency range studied using a fortran program "SINGLE" given in the appendix-I. The values of relaxation frequency (f_r), maximum absorption per wavelength $(\alpha\lambda)_{max}$, and the relaxation amplitudes (A & B) thus obtained are reported for the concentrations from 0.01 molar to 0.04 molar in tables 4.2 and 4.3. The kinetic parameters namely, the mean dissociative rate constant (k_{-1}), associative rate constant (k_1), the variance of size distributions on proper micelles (σ^2) and the volume change due to the exchange process (ΔV_s) are estimated using the expressions (4.1) and (4.2) and are listed in table 4.4. The aggregation number (m) for sodium taurocholate in water is not available in the literature. So for the present calculation of the kinetic parameters, m assigned a value equal to 100. This is the aggregation number of the anionic surfactant sodium hexadecyl sulphate. The variation of observed absorption $(\alpha/f^2)_{obs}$ and the absorption per wavelength ($\alpha\lambda$) with frequency for all concentrations studied are shown graphically in figures 4.3 & 4.4.

Figure 4.3 shows the variation of observed absorption with frequency for 0.01 molar solution of sodium taurocholate in water. The observed absorption decreases with increasing frequency. The variation of observed absorption shows a similar behaviour with increasing concentration of sodium taurocholate. It can also be seen that the values of observed absorption increases in

magnitude with increase in concentration of sodium taurocholate.

The variation of absorption per wavelength ($\alpha\lambda$) with increasing frequency is shown in figure 4.4. From the figure 4.4, it can be seen that the absorption per wavelength for the aqueous solutions of sodium taurocholate increases with increasing frequency and reaches a maximum value at a particular frequency called the relaxation frequency (f_r) of that particular concentration and then decreases with further increase in frequency. For any particular frequency, the absorption per wavelength increases to a higher value with increase in concentration of sodium taurocholate. Also, the relaxation frequency (f_r) increases to a higher value with increase in concentration of sodium taurocholate. The experimental absorption between the frequencies 2.25 and 5 MHz could not be measured, as the fundamental frequency of the transducer used in the present study is 2.25 MHz, so its odd harmonics will be 6.75 MHz etc.

The salient features of the absorption studies are summarised as follows:

1. The observed absorption $(\alpha/f^2)_{obs}$ decreases with increase in frequency for any particular concentration of sodium taurocholate.
2. The values of observed absorption increases to a higher value with increase in concentration of sodium taurocholate in water.

3. For any particular concentration of sodium taurocholate, the variation of absorption per wavelength ($\alpha\lambda$) shows a maximum at the relaxation frequency of that particular concentration.
4. The values of absorption per wavelength increases in magnitude with increasing concentration of sodium taurocholate.
5. The relaxation frequency shifts towards a higher value with increase in concentration of sodium taurocholate.

In aqueous surfactant solutions containing micelles, there is a dynamic exchange process occurring between surfactant monomers in the bulk solution surrounding the micelles and the aggregated surfactant monomer in the micelle. The perturbation of this equilibrium leads to the so called fast relaxation time τ_1 [25-27]. This relaxation occurs in the time range of 10^{-6} - 10^{-9} seconds. The relaxation spectra of micellar solutions are actually characterised by two distinct relaxation times τ_1 and τ_2. The slow relaxation time (τ_2) lies in the time scale of second to millisecond region which is up to three orders of magnitude less than that of τ_1. The relaxation time τ_2 is identified as due to the step-wise build up of micelles from monomers and the dispersion of micelles into monomers [28].

In the present study on aqueous solutions of sodium taurocholate, the absorption data obtained in the frequency range 2-30 MHz fit very well to the equation for a single relaxation which is clear from the figures 4.3 and 4.4.

Thus indicating that the solutions of sodium taurocholate show a single relaxation bahaviour. This behaviour is similar to the relaxation studies undertaken by Aicart et al. [18] in aqueous and aqueous-ethanolic mixtures of DTAB, where a single relaxation behaviour was observed in 0.12 molar aqueous solutions of DTAB. This relaxation behaviour is attributed to the exchange of surfactant monomer between the mixed micelle and the surrounding bulk solution.

A similar result of single relaxation behaviour was reported by Kato et al. [10] for aqueous alkyltrimethyl-ammonium bromides in the frequency range 0.2 - 210 MHz. This study also established that the observed relaxation process was due to the exchange process of a surfactant monomer between the alkyltrimethylammonium bromide micelles and the surrounding bulk solution. Also, in the present study on aqueous solutions of sodium taurocholate the relaxation frequency (f_r) shifts to a higher value with increasing concentration of the sodium taurocholate. This behaviour is in agreement with the studies of Kato et al [10]. Hence, it may be inferred that the observed single relaxation behaviour in the aqueous solutions of sodium taurocholate may be due to the exchange of sodium taurocholate monomer between the sodium taurocholate micelle and the surrounding bulk solution.

Further it can be seen from the table 4.2 that, the obtained values of relaxation time (τ) lies in the time range of 10^{-8} seconds. The relaxation time which occurs in

the time range of 10^{-8} - 10^{-9} seconds is associated with monomer micelle exchange [29-32]. This confirms that the single relaxation process observed in aqueous solutions of sodium taurocholate in the frequency range of 2 - 30 MHz may be due to the monomer exchange between the sodium taurocholate micelle and the surrounding bulk solution. For the observed exchange process, the various kinetic parameters were estimated by applying Teubner theory [18]. The estimated values of kinetic parameters agree well in magnitude with the values reported in the literature for other surfactant systems [10,16]. Hence, it may be concluded that, Teubner theory explains successfully the observed relaxation process in the aqueous solutions of sodium taurocholate.

4.4 Conclusion

The ultrasonic velocity studies in aqueous solutions of sodium taurocholate indicate that the ultrasonic velocity increases with increasing concentration of sodium taurocholate. The plot of ultrasonic velocity against concentration shows a change in slope at 0.013 molar concentration of sodium taurocholate, which corresponds to the critical micelle concentration of sodium taurocholate in water. The increase in ultrasonic velocity may be due to the formation of sodium taurocholate micelle in water. The possible interaction in the formation of sodium taurocholate micelle may be due to the polyfunctional hydrogen bonding.

The variation of adiabatic compressibility and intermolecular free length supports the explanation offered for velocity variation.

The ultrasonic absorption studies carried out in the frequency range 2 - 30 MHz shows that the ultrasonic absorption decreases with increasing frequency and increases with increasing concentration. The absorption variation follows a single relaxational behaviour. The possible relaxation mechanism in the present case may be due to the exchange of sodium taurocholate monomer between sodium taurocholate micelle and the surrounding bulk solution. The various kinetic parameters for the observed relaxation process are also estimated by applying Teubner theory.

TABLE - 4.1

Ultrasonic velocity and related parameters in aqueous solutions of sodium taurocholate at 298 K

Conc. X	ρ Kgm^{-3}	C ms^{-1}	β_s $x10^{-10}\ N^{-1}m^2$	η_s $x10^{-4}Nsm^{-2}$	L_f A^o
water	997.07	1497.00	4.475	8.904	0.422
0.005	997.35	1504.56	4.429	8.777	0.420
0.010	997.92	1510.27	4.393	8.679	0.418
0.020	999.62	1517.50	4.344	8.979	0.416
0.030	1000.47	1521.30	4.319	9.116	0.415
0.040	1002.34	1526.89	4.279	9.548	0.413

X - molar concentration of sodium taurocholate in water;

ρ- density of the solution; C-ultrasonic velocity;

β_s- adiabatic compressibility; η_s- shear viscosity;

L_f- intermolecular free length

TABLE 4.2

Ultrasonic absorption, experimental and calculated absorption per wavelength for aqueous sodium taurocholate at 298K

Conc. X	freq. MHz	$(\alpha/f^2)_{obs}$ 10^{-15}Np $m^{-1}s^2$	$(\alpha/f^2)_{cl}$	$(\alpha\lambda)_{exp}$ $X10^{-3}$	$(\alpha\lambda)_{cal}$ $X10^{-3}$	$(\alpha\lambda)_{max}$ $X10^{-3}$	τ $X10^{-8}$ sec
0.01	2.30	626.99	6.65	2.1670	2.1667	-	8.47
	6.13	198.79		1.8215	1.8192		
	9.97	105.72		1.5614	1.5745		
	15.33	61.27		1.3717	1.3575		
	30.03	25.02		1.0439	1.0473		
0.02	2.30	666.80	6.77	2.3226	2.3235	2.40	5.66
	6.15	230.19		2.1447	2.1403		
	9.98	125.72		1.9015	1.9039		
	15.34	71.84		1.6684	1.6710		
	30.07	29.02		1.3157	1.3145		
0.03	2.30	704.59	6.81	2.4534	2.4547	2.63	4.94
	6.15	255.13		2.3528	2.3452		
	10.00	141.62		2.0974	2.1083		
	15.37	83.58		1.8674	1.8631		
	29.92	36.22		1.4802	1.4803		
0.04	2.31	739.59	7.04	2.6004	2.6001	2.80	4.13
	6.16	280.13		2.6201	2.6223		
	10.01	158.62		2.3994	2.3955		
	15.39	92.58		2.1367	2.1388		
	29.97	39.22		1.7187	1.7184		

X - molor concentration of sodium taurocholate in water; $(\alpha/f^2)_{obs}$ - observed absorption; $(\alpha/f^2)_{cl}$ - classical absorption; $(\alpha\lambda)_{exp}$ - experimentally measured absorption per wavelength; $(\alpha\lambda)_{cal}$ - calculated absorption per wavelength; C - ultrasonic velocity; τ-relaxation time

TABLE 4.3

The fitted values of f_r, A, and B for aqueous solutions of sodium taurocholate at 298K

CONC. X	f_r MHz	A $X10^{15}$ Np $m^{-1}s^2$	B $X10^{16}$ Np $m^{-1}s^2$
0.01	1.88	727.10	30.23
0.02	2.88	778.22	28.75
0.03	3.22	831.72	56.23
0.04	3.85	904.64	25.37

X - molar concentration of sodium taurocholate in water;

f_r - relaxation frequency; A & B - relaxation amplitudes

TABLE 4.4

The kinetic parameters for the aqueous solutions of sodium taurocholate at 298 K.

$\frac{k_{-1}}{m} \times 10^{-7}$	$\frac{k_{-1}}{\sigma^2} \times 10^{-7}$	σ^2	$k_{-1} \times 10^8 s^{-1}$	$k_1 \times 10^{10} M^{-1} s^{-1}$	$\Delta V \times 10^{-6} m^3 mol^{-1}$
0.422	1.517	25.82	4.22	3.25	24.61

k_{-1}- dissociative rate constant; k_1- associative rate constant; m- mean aggregation number; σ^2- variance of size distribution; ΔV- volume change.

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