JOURNAL OF INNOVATION AND TECHNOLOGY
eISSN:2805-5179 Vol. 2023, No.17

Hexyltrimethylammonium Bromide Solutions at 278.15: A Dielectric Investigation on Micelle Relaxation and Hydration

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Received: *7 November 2023;* ***Accepted:*** *9 December 2023;* ***Published:*** *11 December 2023*

Abstract: We have examined the dielectric relaxation of hexyltrimethylammonium bromide solutions at 278.15 K and at 298.15 K in order to investigate the impact of hydrophobic and hydrophilic hydration on temperature. The dynamics of micelles and their hydrophilic and hydrophobic hydration of n-hexyltrimethylammonium bromide solutions are investigated at a low temperature of 278.15 K using dielectric measurements. The concentration ranged from 0.0982 to 0.8534 mol•dm-3, and the frequency range covered from 0.2 to 89 GHz. Four Debye processes added together fit all the spectra. Micelle relaxation processes are visible below the critical micelle concentration, which denotes premicellar aggregation. The relaxation mechanisms were investigated with the aid of Grosse's hypothesis. Due to the radial and tangential diffusion of unbound and bound counter ions around the charged micelles, the two low frequencies are consequently ascribed to these processes. The hydrophilic hydration, in which the water molecules are entirely immobilised, is shown to be temperature independent. On the other side, at lower temperatures, the hydrophobic hydration rises.

Keywords: Dielectric relaxation, hexyltrimethylammonium bromide, micelle dynamics, hydration

Introduction

N-hexyltrimethylammonium bromide lies between tetraalkylammonium salts and alkyltrimethylammonium surfactant (C_6TAB). In our prior study (Hone, Whitney, & Zettl, 1999) at 298.15 K, it was demonstrated that C6TAB functions similarly to higher chain surfactants when micelle relaxations are taking place. On the other hand, when it comes to hydration, C_6TAB is more comparable to tetraalkylammonium salts. Additionally, the outcome demonstrates that loose aggregates rather than conventional micelles with strong bonds form. As the temperature drops, the aggregates should bind strongly and form true classical micelles. The complex permittivity measurements of C_6TAB solutions as functions of concentration ($0.0982 \leq c/\ \mathrm{mol.dm^{-3}} \leq 0.8534$) over the frequency range of $\sim 0.2 \leq \nu/\mathrm{GHz} \leq 89$ at 278.15 K are summarised in this paper. The hydration behaviour varies between 298.15 and 278.15 K, according to the results.

Methodology

Earlier studies by Rohman & Mohiuddin, 2021; Rohman, Mohiuddin, & Khan, 2022 have provided details on the experimental methods. Under reduced pressure, C_6TAB (98%, Fluka, Switzerland, Purum) was dried over P_2O_5. Densities are measured using an Anton Paar DMA60 vibrating tube density metre, which has an accuracy of ± 0.05 kg.m-3. The electrical conductivity measurements were performed with a high-precision AC bridge and two electrode capillary cells, was 0.5%. A homemade time domain reflectometer (TDR) was used to measure the complex permittivity, $\hat{\varepsilon}(\nu)$ of all the solutions. The equipment and experimental methods are described in detail elsewhere (Barker, Gonsalves, Macpherson, Slevin, & Unwin, 1999). The temperature stability was kept within ± 0.0015 K by mounting the TDR in a precision thermostat. Four waveguide interferometers were used to cover the frequency range of $8.5 \leq \nu(\text{GHz}) \leq 89$ (J. Barthel, Bachhuber, Buchner, Hetzenauer, & Kleebauer, 1991; J. Barthel, Hetzenauer, & Buchner, 1992; Buchner, Wachter, & Hefter, 2019).

Result and Discussion

• Data analysis

The total complex permittivity of the examined solutions at varying surfactant concentrations is shown in Figures 1a and b as the real, $\varepsilon'(\nu)$ and loss, $\varepsilon''(\nu)$ components, respectively. The cooperative dynamics of bulk water are what cause the majority of the absorption at ~ 11 GHz. A "slow water" process with a time constant of ~ 34 ps is also observable (Lima, Chaimovich, Cuccovia, & Buchner, 2013). They are referred to as "slow water" since they have reduced dynamics than pure water. These water molecules are experiencing hydrophobic hydration because they are close to the surfactant's hydrophobic tail (Baar, Buchner, & Kunz, 2001; Bakulin, Pshenichnikov, Bakker, & Petersen, 2011). Low frequency processes called micelle relaxation are detected between 1-2 GHz. It is possible to split the micelle processes into two distinct processes. The entire spectrum is thus fitted to a total of four separate Debye processes, taking into account two solvent processes and two micelle processes:

$$\hat{\varepsilon}(\nu) = \frac{\varepsilon_1-\varepsilon_2}{1+i2\pi\nu\tau_1} + \frac{\varepsilon_2-\varepsilon_3}{1+i2\pi\nu\tau_2} + \frac{\varepsilon_3-\varepsilon_4}{1+i2\pi\nu\tau_3} + \frac{\varepsilon_4-\varepsilon_\infty}{1+i2\pi\nu\tau_4} + \varepsilon_\infty \tag{1}$$

Eq (1) considers the sum of four separate relaxation processes with individual relaxation time of τ_j and dispersion amplitudes of $S_j (= \varepsilon_j - \varepsilon_{j+1})$, which together contribute to the entire spectrum, $\hat{\varepsilon}(\nu)$. The high frequency permittivity limit, which results from intramolecular polarizability, is given by the symbol ε_∞.

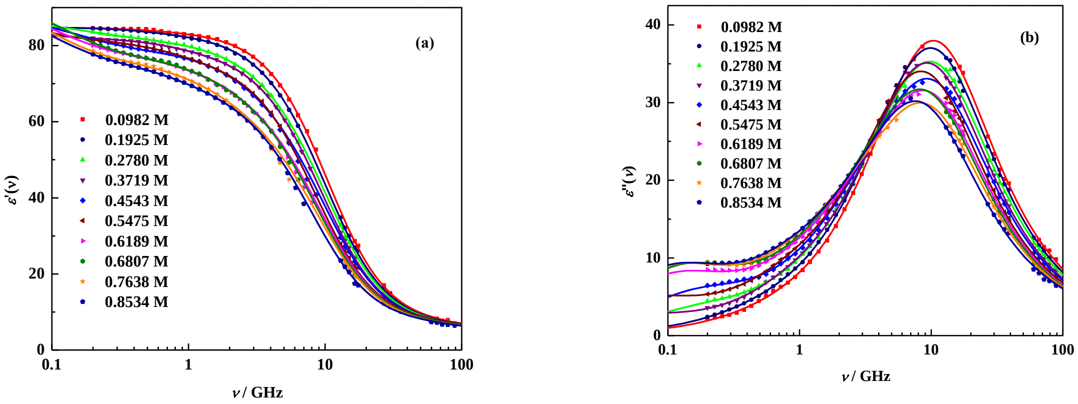

Figure 1. Dielectric permittivity

Figure 1 illustrated the dielectric permittivity, ε'(ν) and loss, ε"(ν) spectra for different concentrations of aqueous hexyltrimethylammonium bromide solutions at 278.15 K. The symbols denote experimental data points, solid lines depict fitted curves that were obtained using the **Table 1** and eq (1) parameters.

Table 1. Densities, ρ, specific conductivities, κ, and relaxation parameters, (ε_i,τ_i) obtained from eq (1) for aqueous hexyltrimethylammonium bromide solutions at 278.15 K.

c (mol.dm^{-3})	ρ (kg.m^{-3})	κ (S.m^{-1})	ε_1	τ1 (ps)	ε_2	τ2 (ps)	ε_3	τ3 (ps)	ε_4	τ4 (ps)	ε_∞	χ_r^2
0.09817	1003.72	0.5004			84.71	253.1	83.35	34.74	79.88	14.99	5.397	0.0696
0.1925	1006.86	0.8753			84.83	280.5	82.63	38.45	75.00	15.19	5.275	0.0395
0.2429	1009.80	1.155	85.69	2159	82.39	147.4	79.53	40.00	71.87	14.96	5.004	0.0915
0.2780	1013.15	1.421	86.37	974.8	81.90	169.2	79.93	42.55	70.09	15.04	5.177	0.045
0.3719	1016.20	1.626	85.98	3075	81.86	166.7	77.33	30.03	67.50	15.83	5.300	0.039
0.4543	1019.76	1.828	87.15	1005	78.61	178.1	77.52	52.90	65.18	15.48	5.173	0.118
0.5475	1022.59	1.965	89.36	2570	81.20	181.8	74.94	29.50	63.00	17.22	5.298	0.069
0.6189	1025.10	2.071	90.45	1448	77.25	156.5	72.33	50.14	61.33	16.33	5.125	0.101
0.6807	1028.58	2.196	91.86	1276	76.71	111.7	69.93	39.18	60.00	16.97	5.208	0.092
0.7638	1032.47	2.308	90.44	1481	75.33	136.2	68.53	52.99	58.00	16.75	4.932	0.103

Apart from the first two solutions, where the lower frequency may have changed to a much lower frequency than the experimental coverage, the entire dielectric spectrum has been fitted to a summation of four-Debye processes. Based on eq. (1), the dielectric parameters ε_i and τ_i have been determined for every solution and are given in Table S1. Figures S1 and S2 illustrate how (ε_i, τ_i) and the dispersion amplitude, $S_i = \varepsilon_i - \varepsilon_{i+1}$, vary with surfactant concentration. The selected model is supported by the smooth variation of the estimated parameters (ε_i, τ_i) with concentration (**Figure 2**). **Figure 3** shows the four Debye resolved modes of various processes.

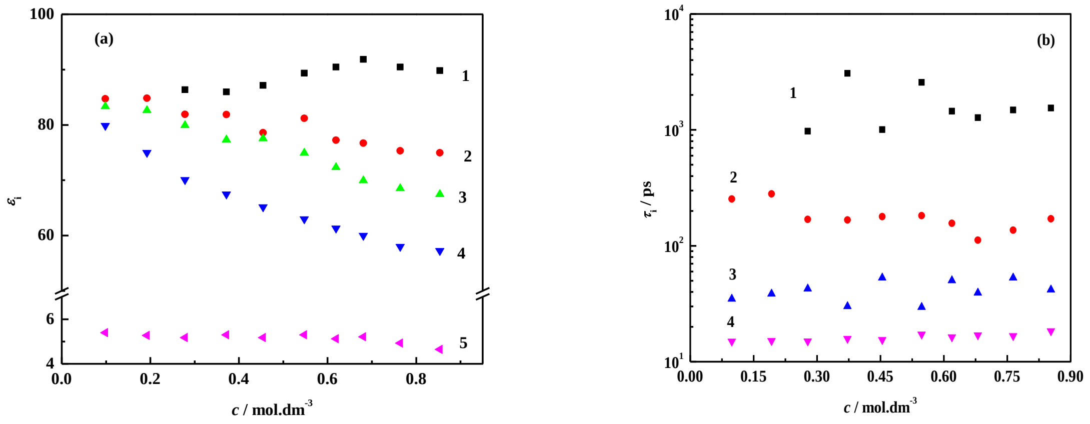

Figure 2. Relaxation parameters, (ε_i,τ_i) of aqueous hexyltrimethylammonium bromide solutions at 278.15 K.

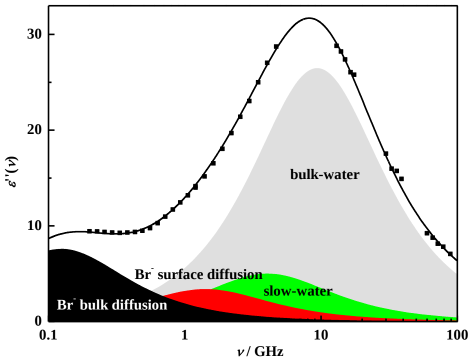

Figure 3. The various relaxation processes for an aqueous solution of 0.4543 mol.dm^{-3} hexyltrimethylammonium bromide at 278.15 K are represented by four-Debye resolved modes.

- **Micelle relaxation**

A micelle in a micelle solution with a particular surface conductivity is surrounded by a layer of ions with a mainly one sign. As a result, when subjected to fields, the two types of ions in the micelle solution behave differently. As the counterion approaches the particle, charge is easily transferred to the opposite side when it comes into touch with a region of dense counterions. A co-ion must either move around the particle or stay on its original side while being affected by electric and diffusive forces since it lacks this highly conductive channel. As a result, while considering the motion of co-ions, a charged particle and its counterion layer behave as an insulator, and when considering the motion of counterions, they behave as a conductor. Regardless of whether the thickness of the conducting shell is believed to be infinitely thick or to have a definite thickness, single (Grosse & Foster, 1987) or double (Grosse, 1988) relaxation occurs. When the particle's radius, R, is significantly larger than the Debye screening length,

$$\chi^{-1} = \left(\frac{\varepsilon_m \varepsilon_0 D}{\kappa_m}\right)^{\frac{1}{2}} \tag{2}$$

The two relaxations can only be separated from one another in frequency by a wide margin. In eq (2), ε_0 is the vacuum's permittivity, ε_m is the medium's static permittivity, and D is the counter ions', Br- bulk diffusion coefficient. The two relaxation processes yielded the following analytic equation for dispersion, S_i and relaxation times, τ_i (Grosse, 1988):

$$\tau_1 \approx \frac{R^2}{D} \tag{3}$$

$$S_1 = \frac{9\phi\varepsilon_m\left(\frac{2\chi\lambda_s}{\kappa_m}\right)^4}{16\left[\frac{2\chi\lambda_s}{\kappa_m}\left(\frac{2\lambda_s}{R\kappa_m}+1\right)+2\right]^2} \tag{4}$$

$$\tau_2 = \frac{\varepsilon_0\varepsilon_m\left(\frac{\varepsilon_p}{\varepsilon_m}+2\right)}{\kappa_m\left(\frac{2\lambda_s}{R\kappa_m}+2\right)} \tag{5}$$

$$S_2 = \frac{9\phi\varepsilon_m\left(\frac{2\lambda_s}{R\kappa_m}-\frac{\varepsilon_p}{\varepsilon_m}\right)^2}{\left(\frac{\varepsilon_p}{\varepsilon_m}+2\right)\left(\frac{2\lambda_s}{R\kappa_m}+2\right)^2} \tag{6}$$

Here, the micelle is modelled as a spherical particle with a permittivity, ε_p that is frequency independent. The micelle is surrounded by a conducting shell of counterions with a finite value of surface conductance, λ_s. The micelles are considered as immersed in a conducting medium with specific conductivity, κ_m. ϕ is the volume fraction of micelles. The experimental data's τ_1, $\tau_{2,}$ S_1 and S_2 are fitted to eqs. (3-6), and for each solution, the unknown parameters R, ϕ, λ_s and the reciprocal Debye parameter, χ, are evaluated. The micelle dielectric core's constant,(Sen, Anicich, & Arakelian, 1992) ε_p = 2, and the medium's conductivity, κ_m is the experimental conductivity of the solution. **Table 2** contains a list of estimated parameters. Evidently, R is more than the hydrophobic core radius calculated by Tanford: = 0.15 + 0.1265n = 0.909 nm. The so-called Stern layer, which is made up of a hydrophilic head group, bound counter ions, and hydration water, is thought to be responsible for the discrepancy.

Table 2. Parameters of eq.(3)- eq.(6) for aqueous hexyltrimethylammonium bromide solutions at 278.15 K.

c **(mol.dm^{-3})**	***R*** **(nm)**	**λ_s (nS)**	**ϕ**	**χ (10^9 m^{-1})**
0.2780	1.09	3.81	0.009	3.43
0.3719	1.94	6.38	0.024	1.21
0.4543	1.11	3.09	0.007	5.87
0.5475	1.77	4.41	0.049	1.52
0.6189	1.33	4.06	0.035	2.88
0.6807	1.25	6.19	0.035	2.61
0.7638	1.35	4.80	0.046	2.61
0.8534	1.37	3.13	0.080	2.36

We have determined the conductivity of micelle particles, κ_p by using the equation, $\kappa_p = \kappa_m + 2\lambda_s R^{-1}$, proposed by O'Konski (O'Konski, 1960), where κ_m is the medium's conductivity, λ_s is the surface conductivity of micelle particles, and R is the micelle's radius. The following eq (7) was suggested by Maxwell-Wagner (Wagner). According to the mixing rule,

$$\frac{\kappa-\kappa_m}{\kappa+2\kappa_m} = \phi\frac{\kappa_p-\kappa_m}{\kappa_p+2\kappa_m} \tag{7}$$

where κ is the total conductivity of the solution and ϕ is the volume fraction of particles. With the knowledge of the values of, , κ_p, and ϕ, and, we have estimated κ_m. The plots of κ_m, κ, and κ_p with surfactant concentration are shown in **Figure 4**.

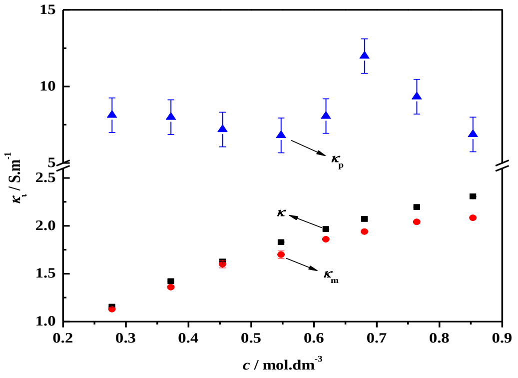

Figure 4. For aqueous hexyltrimethyalammonium bromide at 278.15 K, plots of conductivity of micelles, κ_p medium, κ_m and the total, κ against concentration of surfactant, c. The standard error is shown by the vertical lines.

- **Hydration**

The dispersion amplitude of bulk water decreases with increasing surfactant concentration as shown in **Figure 5**. Three factors contribute to this: (a) the volume effect, where water concentration decreases as surfactant concentration rises; (b) hydration, where some water molecules are tightly bound to ions, ion-pairs, or surfactant head groups and are therefore non-rotational and unresponsive to applied fields; and (c) the kinetic depolarization, which results from the coupling of ion transport with solvent viscosity.

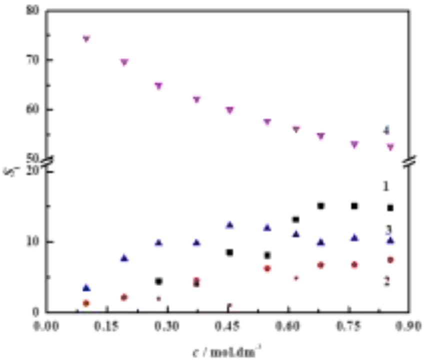

Figure 5. Dispersion amplitude, S_i of various processes in aqueous hexyltrimethylammonium bromide solutions at 278.15 K.

A more useful fact is that the bulk water dispersion is temperature independent after kinetic depolarization has been corrected, while the slow water dispersion is temperature dependent, as seen in **Figure 6**. As it continues, the slow water reaches a plateau at nearly cmc and doesn't change. In the case at 298.15 K, the slow water mode does not occur until after cmc.

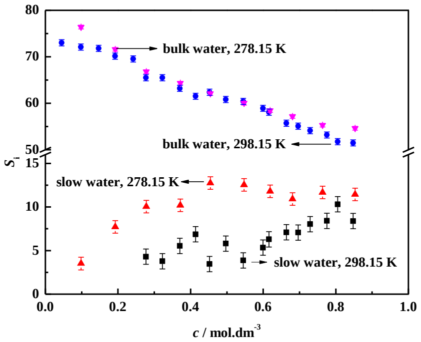

Figure 6. Kinetic depolarization corrected dispersion, S_i of bulk and slow water at 278.15 and 298.15 K for aqueous hexyltrimethylammonium bromide solutions at various concentration, c.

The normalised Cavell equation is used to compute the apparent concentration of slow water, $c_w^{app,s}$, and bulk water, $c_w^{app,b}$, which contribute to the solvent dispersions (J. Barthel et al., 1992; Cavell, Knight, & Sheikh, 1971):

$$c_w^{app,i}(c) = c_w(0)\left[\frac{\varepsilon_w(0)}{\varepsilon_w(c)}\right]\left[\frac{2\varepsilon_w(c)+1}{2\varepsilon_w(0)+1}\right]\left[\frac{1-\alpha_w f_w(c)}{1-\alpha_w f_w(0)}\right]^2\left[\frac{S_w(c)}{S_w(0)}\right] \qquad (8)$$

where the dielectric constant, dispersion amplitude, and polarizability of water are represented by ε_w, S_w and α_w, respectively. The input values are, $c_w(0)$ = 55.51 mol·dm^{-3}, $\varepsilon_w(0)$ = 85.89, $S_w(0)$ = 82.07 and $4\pi\varepsilon_0\alpha_w$ = 1.607 x 10^{-40} m^3. The reaction field factor, $f_w(c)$ is obtained by using the eq (9):

$$f_w^i(c) = \frac{3}{4\pi r_w{}^3}\frac{A(1-A)[\varepsilon_w^i(c)-1]}{\varepsilon_w^i(c)+[1-\varepsilon_w^i(c)]A} \tag{9}$$

where, $A = \frac{1}{3}$ for spherical reaction field and r_w = 142.5 pm.

By utilising the following relations, $c_w^{app,s}(c)$ and $c_w^{app,b}(c)$ produce the hydrophobic hydration number, $Z_s(c)$, and the nonrotationally bound water molecules, $Z_{ib}(c)$:

$$Z_s(c) = \frac{c_w^{app,s}(c)}{c} \tag{10}$$

$$Z_{ib}(c) = \frac{c_w(c)-\left[c_w^{app,b}(c)+c_w^{app,s}(c)\right]}{c} \tag{11}$$

Figure 7 demonstrates that for 278.15 and 298.15 K, the Z_{ib} is independent of both temperature and surfactant concentration above cmc. In a related study, DRS was used to look at the movements of the water molecules close to tetramethylurea (Bakulin et al., 2011). As concentration increased, it was discovered that the reorientation dynamics of solvating water molecules surrounding the methyl groups were 3–10 times slower than the bulk. They also observed that the water content of this hydrophobic hydration layer reduced with increasing temperature.

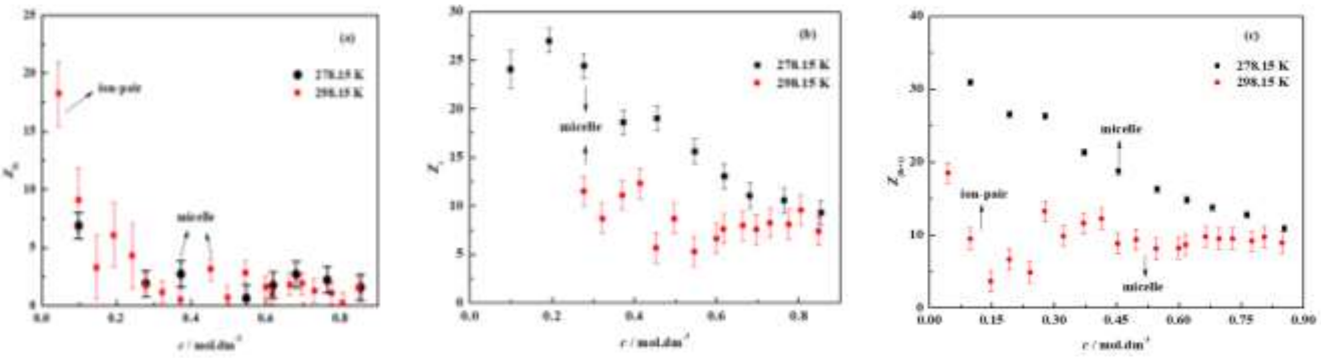

Figure 7. Plots of water molecules, per surfactant molecule against concentration, c at 278.15 K and 298.15 K for aqueous hexyltrimethylammonium bromide solutions. (a) Nonrotationally bound water molecules, Z_{ib}; (b) slow water molecules, Z_s; and (c) total water molecules, Z_{ib+s} per surfactant molecule.

Conclusion

We studied the micelle relaxation and hydration of aqueous solutions of hexyltrimethylammonium bromide at 278.15 K. The low frequency micelle relaxation processes centred at 0.1 and 1.5 GHz are linked to the tangential and radial diffusion of counter ions surrounding the charged micelle. The hydrophilic hydration that immobilises the water molecule at 298.15 and 278.15 K is nearly the same due to strong ion-dipole interaction. Nonetheless, at 278.15 K compared to 298.15 K, there is a greater hydrophobic hydration, which slows down the dynamics of the water molecule.

Acknowledgment

Author gratefully acknowledges the financial support received through a scholarship from the Alexander von Humboldt Foundation. The authors also extend their appreciation to Sultan Qaboos University for sponsoring T.M. through Grant No. SR/SCI/PHYS/18/01.

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