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Micellar size of drag reducing cationic surfactants

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Abstract Dynamic light scattering was employed to determine the effects of surfactant nature, concentration and counterion ratios, and shear on the hydrodynamic radii of micelles of commercial cationic surfactants which are powerful drag reducers in turbulent flow at high temperatures. Such surfactants are potentially useful for reducing pumping energy losses in district heating and cooling systems.

Key words Surfactant micelle size – hydrodynamic radius – shear history – dynamic light scattering

Introduction

Intensive research has been carried out on the influence of certain cationic, anionic and non-ionic surfactants, in some cases with counterion salts, on drag reduction in turbulent flow in pipes. The observed drag reduction has been ascribed to the presence of rod-like micelles in the solution. Surfactant drag reducers may lose their drag reduction ability when subject to high shear but quickly regain their effectiveness when flowing in a regime of lower shear, allowing them to be used in recirculation systems where polymer additives which mechanically degrade irreversibly cannot be used. This is believed to be due to the ability of micelles to form long threads or networks which break up under high shear but reform when shear is removed.

During the last few years much effort has been given to developing cationic surfactants which are effective over a wide range of temperatures such as are encountered in district heating or cooling systems. Quite a few results have already been published on physical–chemical, hydraulic and rheological properties of such surfactants and their possible usage, see for example refs. [1–5]. In this paper, we want to show and discuss one of the principal properties of single micelles, namely their size.

Ohlendorf et al. [6] showed that the length of rod-like micelles of hexadecyl trimethyl ammonium bromide with sodium salicylate (C_{16} TA Sal) determined by electric birefringence decreases with rising temperature and increases with surfactant concentration. Micelle lengths increased from 30 nm to 70 nm at 30 °C when concentration increased from 0.2 to 0.4%. At 50 °C, the lengths were 18 and 30 nm at the same concentrations. For tetradecyl trimethyl ammonium salicylate (C_{14} TA Sal) micelles at 30 °C, the lengths were 30 and 40 nm at the same two concentrations. Kalus et al. [7] reported that micelles of 5 mM tetradecyl trimethyl ammonium salicylate in D_2O at 25 °C are approximately 30 nm long and their diameter is approximately 4 nm based on small angle neutron scattering measurements. Using electrical birefringence measurements, Bewersdorff and Ohlendorf [8] found that the length of micelles of C_{14} TA Sal decreases with decrease in salt content and with increase in temperature.

Interesting results on cetyl trimethyl ammonium/sodium salicylate micelles were reported by Nemoto and

Kuwahara [9]. The lengths of these micelles are unusually large, ranging from 600 nm to 1870 nm based on experiments in a broad range of salt/surfactant ratios, from 1 to 41, with fixed concentration of the surfactant, 10 mM. These authors found that the length decreased at 25° and 33 °C and increased at 50 °C with increase in sodium salicylate concentration, while at 40 °C the salicylate ratio had no influence. Since 50 °C is near the upper temperature limit for effective drag reduction for this system and above 60 °C the micelles revert to spherical shape, and additional sodium salicylate extends this limit and prolongs the rod-like shape to higher temperatures, the 50 °C result is not surprising.

Using flow birefringence measurements, Hoffman and Hofmann [10] observed that the length of mixed micelles can be varied in the tetradecyl dimethyl aminoxide/sodium dodecylsulfate (C_{14}DMAO/SDS) system by varying the ratio of the two surfactants in 100 mM solutions. With the ratio under 1:1, the length is about 10 nm or smaller while at a ratio of 2:1, the length is 19 nm. Earlier Hofmann et al. [11] had also reported that the dimensions of tetradecyl dimethyl aminoxide/sodium dodecylsulfate micelles are dependent on the ratio of the two surfactants based on small angle neutron scattering measurements. The length varied from 7 to 360 nm for concentration ratios of 2:3 to 10:0. Micelle diameter was about 4 nm independent of concentration ratio. The influence of total concentration of the same surfactant mixture is presented in ref. [12]. Micelle length increased from 5 to 20 nm when concentration increased from 20 to 150 mM at a ratio of 6:4 and a temperature of 25 °C. With increasing concentration of SDS, the length of the micelles decreased; the rate of decrease depended on the particular value of the ratio C_{14}DMAO to SDS. Rehage et al. [13] also noted that anisometric micelles generally grow in length as surfactant concentration increases.

Hu, Wang and Jamieson [14] reported on the hydrodynamic diameter of micelles of tetradecyl dimethyl aminoxide/sodium dodecylsulfate at a concentration of 6/4 mM plus 24 mM of NaCl based on light scattering measurements. This surfactant mixture had micelles with a hydrodynamic diameter of $2Rh = 22$ nm. The authors estimate that this corresponds to a rod-like micelle of length 50 nm, which is approximately $4.5Rh$. A number of methods for the study of micellar shape and size are mentioned in ref. [15] with a list of references.

By means of rheological and SANS measurements, Schmitt et al. [16] reported that aqueous solutions of cetylpyridinium chlorate with 5 mM sodium chlorate contain micelles only 8.6 nm long at 20% of $CPClO_3$ and 6.0 nm long at 34% of $CPClO_3$; the length decreasing slightly with concentration at this high concentration. Using SANS, Appell and Marignan [17] determined the micelle length of 0.25% $CPClO_3$ in 1 to 4 M brine at 35 °C to be 17 ± 5 nm with 5 nm diameter. The length was insensitive to the salt concentration. Berret et al. [18] using light scattering and rheological measurements found that the length of micelles of cetylpyridinium chloride with sodium salicylate is 15 nm. Large micellar sizes were estimated from rheological measurements by Khatory et al. [19] for 0.35 M hexadecyltrimethyl ammonium bromide (CTAB) with potassium bromide. Increasing salt concentration by fivefold from 0.4 to 2.0 M gave a smooth increase of the apparent micelle length, from 170 to 710 nm at 31 °C and from 110 to 490 nm at 35 °C. Variations in CTAB concentration at 1.5 M potassium bromide gave lengths of 800 nm to 580 nm in the CTAB concentration range of 0.1 M to 0.4 M.

In summary, the lengths of micelles are mostly reported to be several tens of nanometers, but micelles of cetyltrimethyl ammonium with sodium salicylate and hexadecyltrimethyl ammonium bromide with potassium bromide were reported to be much longer, in one case even over 1 μm. Clearly, the lengths of micelles vary very much with micelle composition, temperature and salt concentration.

Experimental

Surfactants

Four of the surfactants used in this investigation were commercial products, while those described in the literature were pure. One pure surfactant was also tested to see if any differences in behavior are caused by impurities in the commercial products.

1) Habon G (Hoechst AG), which is hexadecyl-dimethyl-hydroxyethyl ammonium 3-hydroxy-2-naphthoate (molecular weight $Mw = 501$).

2) Ethoquad T13-50 (Akzo Chemical) is tallow-tris-hydroxyethyl-ammonium acetate [tallow-$N(C_2H_2OH)_3$ Ac] ($Mw = 454$), referred to as ET 13. The tallow is 3% C_{14} (saturated), 27% C_{16} (saturated), 3% C_{16} (mono-unsaturated), 18% C_{18} (saturated), 42% C_{18} (mono-unsaturated) and 4% C_{18} (di-unsaturated).

3) Ethoquad 0-12 (Akzo Chemical) is oleyl bis-hydroxyethyl-methyl-ammonium chloride [Oleyl-$N(CH_3)(C_2H_4OH)_2Cl$] ($Mw = 403$), referred to as ETO below. The oleyl is 2% C_{14} (saturated), 4% C_{16} (saturated), 4% C_{16} (mono-unsaturated), 14% C_{18} (saturated), and 75% C_{18} (mono- and di-unsaturated).

4) Arquad 16-50 (Akzo Chemical) is approximately $C_{16}H_{33}N(CH_3)_3Cl$] ($Mw = 319$), referred to as AR below. The alkyl group is 12% C_{14} (saturated), 75% C_{16} (saturated), 11% C_{18} (saturated), and 1% C_{20} (saturated).

5) Cetyl trimethyl ammonium chloride, which is $C_{16}H_{33}N(CH_3)_3Cl$ ($Mw = 318$) referred to as CTAC below was purchased as pure chemical (25% aqueous solution) from Aldrich Chemical Company.

Habon G, donated by Hoechst AG was received as 53.5% active surfactant with 10.2% isopropanol and 36.3% water.

Ethoqaud T13-50, Ethoquad 0-12 and Arquad 16-50, donated by Akzo Chemical (now Akzo Nobel), were received as 50% surfactant in a water–isopropanol solution.

All concentrations listed are of active surfactant.

The salt containing salicylate counterion used with surfactants 2, 3, 4 and 5 was sodium salicylate, $Mw = 160$, referred to as NA below. Habon G reaches maximum drag reduction effectiveness at about 1 mM concentration [2, 4] while solutions of ET13/NA, ETO or AR/NA require approximately 5/12.5 mM concentration for maximum drag reduction [1, 3].

Rheological data on ET13, ETO and AR16 have been presented in refs. [20–22]. Habon G rheological data will be reported in a forthcoming paper [23].

Method of measurement

The measurements of surfactant size were made using the dynamic light scattering technique. The apparatus consists of a home built goniometer, and a Coherent Innova-70 Argon laser, operated at a wavelength of 514 nm. The correlator used was the ALV-5000E multi-bit, multi-tau correlator which covers approximately 11 decades in delay time range.

The measured correlation functions were mathematically analyzed using a nonlinear inverse Laplace transformation technique proposed by Jakeš [24] which yields the distribution of decay rates, Γ.

The relaxation time distribution can be transformed into a distribution of diffusion coefficients only if the particles meet the conditions for diffusion movement, that is, if the relation

$$\Gamma = D\left(\frac{4\pi n}{\lambda}\right)^2 \sin^2\left(\frac{\theta}{2}\right) \qquad (1)$$

is satisfied. Here Γ is the relaxation (decay) rate, D the diffusion coefficient, λ the wave length of light in vacuum, n the refractive index, and θ the scattering angle. If the dependence of Γ on q^2, the square of the scattering vector, is linear, the process is diffusive, with diffusion coefficient, $D = \Gamma/q^2$. Here, $q = (4\pi n/\lambda)\sin(\theta/2)$.

The hydrodynamic radius, Rh, of a particle can then be extracted from D using the Stokes–Einstein equation:

$$Rh = kT/6\pi\eta D \qquad (2)$$

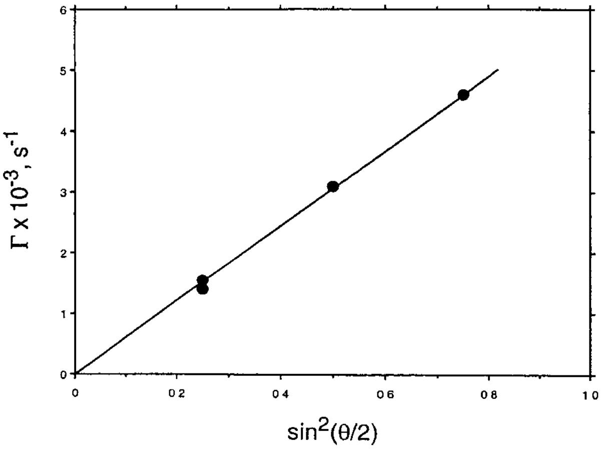

Fig. 1 Dependence of the decay rate on the scattering angle in the solution of Ethoquad T13/50 (ET13) with sodium salicylate (NA) with concentration ET13/NA equal 5/7.5 mM

where k is the Boltzmann constant, T the absolute temperature and η the viscosity of the solvent. The calculated radius, Rh, is the radius of a sphere which has the same diffusion coefficient as the measured particle even though the true shape of the particle may differ from a sphere. $2Rh$ would thus be a diameter of a spherical particle with diffusion coefficient equal to the diffusion coefficient of a rod-like micelle. It is assumed that the micelles are separated and do not interact.

The dependence of the decay rate, Γ, on the scattering angle, θ, was examined for several solutions. In all cases Γ as a function of $\sin^2(\theta/2)$ was a linear function passing through the origin. This demonstrates that the dynamics observed are diffusive and that it is possible to extract a diffusion coefficient, D, from the measured decay rate. Figure 1 illustrates this finding for a solution of ET13/NA at a concentration of 5/7.5 mM.

All the measurements were repeated several times at 20 °C, usually 10 times and average values of dimensions and distributions were determined. The distribution curves are plots of intensities of scattered light $A(Rh)$ against the radius ($\log Rh$). The estimated width, w, of the distribution is measured as the width at half height of its peak in logarithmic units. It represents 69 to 74% of all particles in these experiments. The hydrodynamic radius, Rh, is calculated as an average from the integral of the curve. As a rule in our measurements, it is a little smaller than the coordinate of the maximum of the peak.

Two types of measurements are reported here. The first type is on solutions prepared with gentle stirring. These are referred to fresh solutions. The second type is on solutions which were vigorously sheared with a propellor mixer for two hours. These solutions are referred to as sheared.

With the exception of Habon G, the distributions of relaxation times (and therefore also the distributions of other quantities) for fresh samples were observed to be unimodal which means that the solutions contained only one kind of particle, but the distributions were not monodisperse, the width depending on the concentration.

Results and discussion

Concentration influence

The dependence of *Rh* of micelles in ET13/NA in distilled water on the surfactant concentration (at fixed molar ratio) of both components and the distribution of *Rh* at a concentration 1.25/1.87 mM are shown in Figs. 2 and 3. (This molar concentration corresponds to a total concentration of ET13 of 1.135 g/l (50% active matter) and 0.3 g/l of NA). *Rh* increased from 20.5 nm to 25.5 nm when concentration increased from 1.25/1.87 mM to 5/7.5 mM. This 25% change is not large for the fourfold concentration range. Extrapolation to infinite dilution yields *Rh* = 19.5, but is not physically meaningful as rod-like micelles would not form at low concentration. The distributions of hydrodynamic radii are rather large. At 1.25/1.87 mM, *Rh* ranges from 11 nm to 46 nm and the width, *w*, of the distribution is 61% of a decade. The estimated width increases with concentration, showing a 45% increase at 5.0/7.5 mM as listed in Table 1.

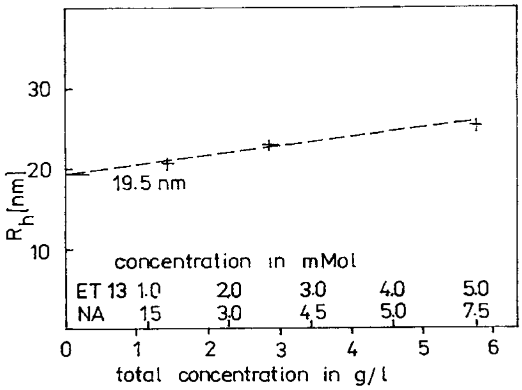

Fig. 2 Dependence of hydrodynamic radius, *Rh*, of the solution of Ethoquad T13/50 with sodium salicylate on total concentration of both components; the ratio of concentrations of both components C(ET13)/C(NA) is constant

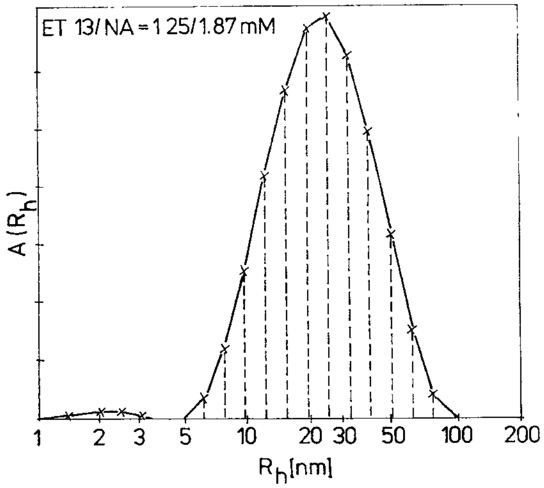

Fig. 3 Plot of scattered light, *A*(*Rh*), vs. hydrodynamic radius, *Rh*, in ET13/NA (1.25/1.87 mM) solution in distilled water

Table 1 Effect of ET13/NA Concentration on *Rh* and *w*

Surfactant/NaSal Concentration	*Rh*, nm	Distribution Width, *w* In%
1.25/1.87 mM	20.5	61%
2.50/3.75 mM	23.0	70%
5.00/7.50 mM	25.5	88%

Measurements of 3 mM ET13 with 5.0, 7.5 and 12.5 mM NA in distilled water gave an increase of the mean hydrodynamic radius of only 10% (Figs. 4 and 5). The distilled water samples contained some impurities and for that reason each was measured twenty times. Only results which were not affected by scattering from impurities are shown. The width of the distributions decreased by 33% as NA/surfactant ratio increased. Thus, with decreasing ET13/NA ratio, the dispersion of the micelles tends to monodispersity.

Similar influence of the salt content in the sample was found with AR/NA solutions in distilled water and shown in Figs. 6 and 7. Table 2 shows the increase of mean *Rh* values and the decrease of *w* values with the decrease of surfactant/salt ratio, i.e., with increase of sodium salicylate content, C(NA), at constant concentration of the surfactant, C(AR):

In this case there is a 13% increase of *Rh* over a 1.9 fold concentration ratio while *w* decreases 29%. The extrapolation to zero concentration C(NA) yields *Rh* = 27 nm.

The results with AR/NA and ET13/NA solutions are very similar though their molecular weights differ considerably. In general, these results showing the influence of the concentration of the mixture and that of the ratio of surfactant/salt are in accordance with results from the investigations using pure chemicals presented in refs. [6–8, 10–13].

Another effective drag reducing surfactant is ETO with a molecular weight between those of AR and ET13. ETO/NA solution with 3.0/12.5 mM concentration in distilled water has an average mean hydraulic radius of

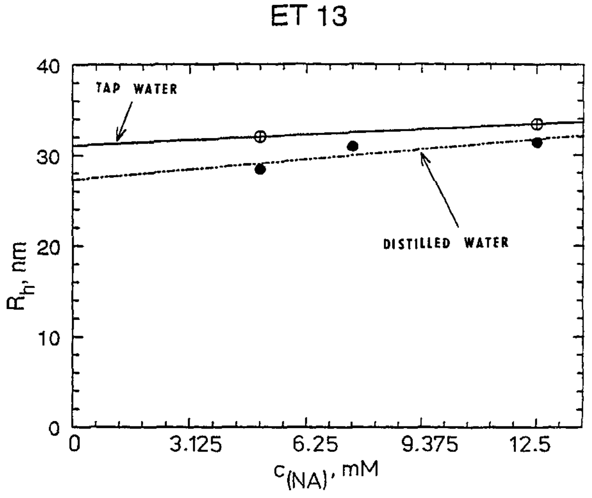

Fig. 4 Dependence of *Rh* on changing content of NA in ET13/NA solution in distilled and tap water (the influence of changing ratio of both components) with constant 3 mM concentration of ET13

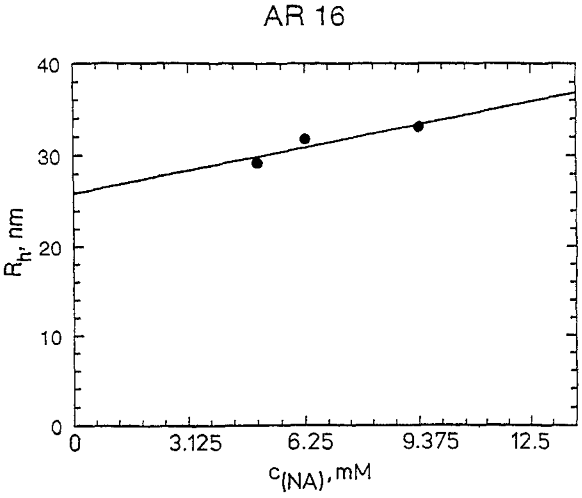

Fig. 6 Dependence of *Rh* on changing content of NA in AR16/NA solution in distilled water with constant concentration of AR16 of 3 mM

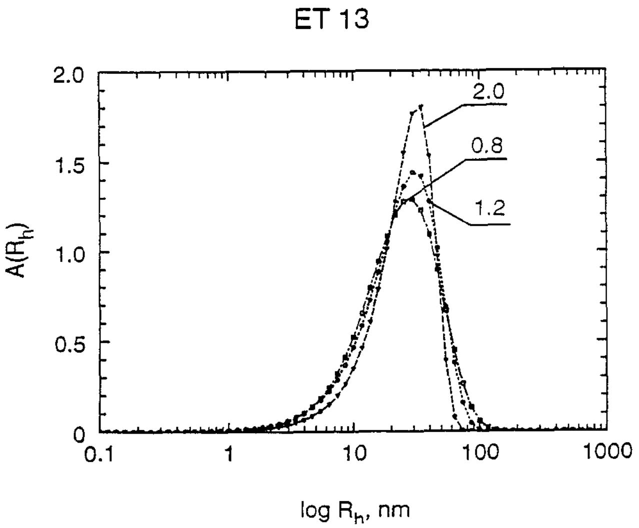

Fig. 5 Distribution functions in ET13/NA solutions in distilled water with constant ET13 total concentration C(ET) = 2.7 mg/l (= 3 mM of effective matter) and changing content of NA: 0.8 g/l (= 5 mM), 1.2 g/l (7.5 mM) and 2 g/l (12.5 mM)

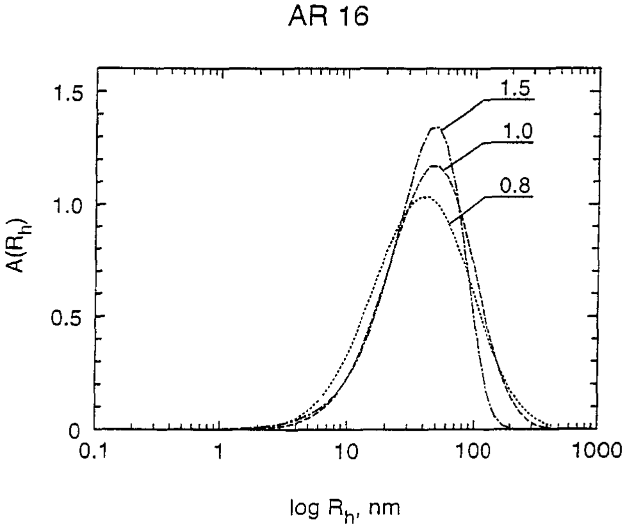

Fig. 7 Distribution functions in AR16/NA solutions with constant AR16 total concentration 1.9 g/l (= 3 mM of effective matter) and concentration of NA changing from 0.8 g/l (5 mM) to 1.5 g/l (9.37 mM) in distilled water

Table 2 Effect of AR/NA Ratio on *Rh* and *w*

Concentration AR 16 in mM	Concentration NaSal in mM	*Rh*, nm	Distribution width *w*, in%
3.00	5.00	29.3	91%
3.00	6.25	31.8	82%
3.00	9.37	33.1	65%

34.1 ± 0.6 nm with $w = 45\%$ of a decade. The size distribution data are plotted in Fig. 8. The *Rh* distributions correspond to those of the highest concentrations of both ET and AR.

Measurements with Habon G

The surfactant, Habon G, is a one component product. The counterion, 3-hydroxy-2napthoate, is reacted with the ammonium. The influence of concentration and also the

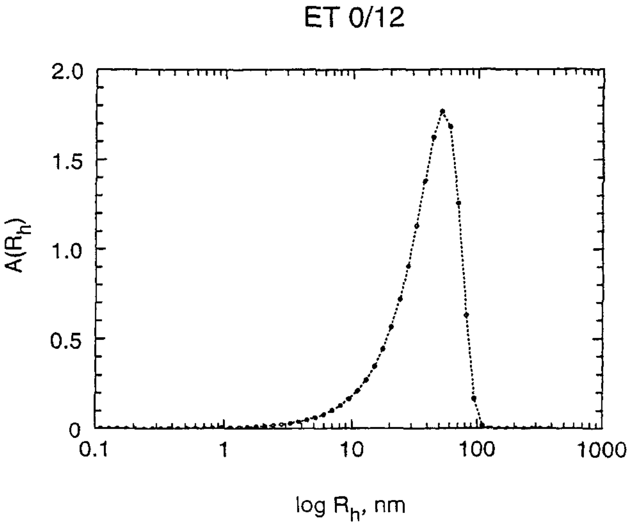

Fig. 8 Distribution function of Ethoquad 0/12 with sodium salicylate in distilled water; the concentration of ET0/NA = 3/12.5 mM

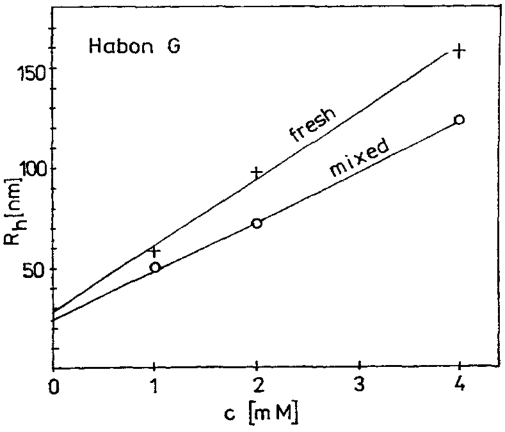

Fig. 9 Dependence of *Rh* on concentration of Habon G

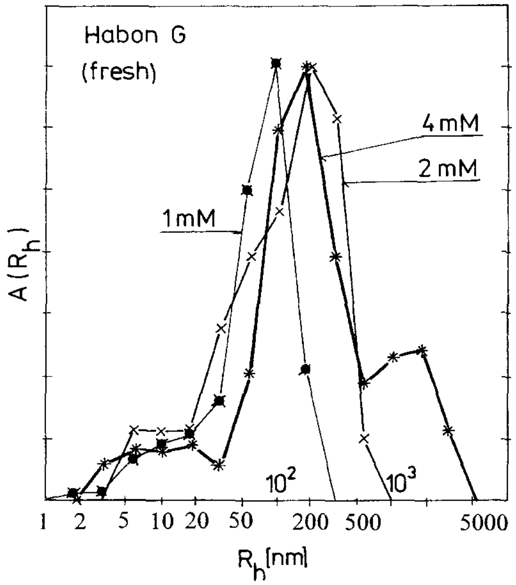

Fig. 10 Distribution function in fresh Habon G solutions of 1, 2 and 4 mM concentrations in distilled water

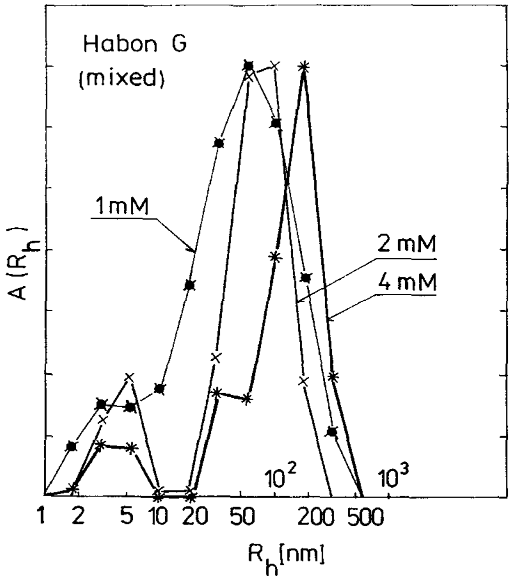

Fig. 11 Distribution function in sheared Habon G solution of 1, 2 and 4 mM concentrations in distilled water

influence of intense shearing by mixing in a beaker for two hours (which was applied to simulate the effect of pumping on the breakdown or degradation of the micelles) were investigated. Light scattering measurements were made several days after mixing.

Hydraulic radii as a function of concentration for the fresh and the sheared solutions are plotted in Fig. 9. Over a fourfold range of concentration (1 to 4 mM), *Rh* shows a strong dependence on concentration, varying from 58 nm to 157 nm in the fresh solution which is a 170% increase. The growth of micelle size with concentration is large compared with other surfactants. Intensive shear reduced the hydraulic radii of the micelles by 16 to 28%, the effect of shear being more pronounced with the larger, higher concentration micelles. If we calculate a volume change of hypothetical spheres with diameter 2*Rh*, this decrease of *Rh* corresponds to a significant 41 to 63% decrease in volume of the micelles. In the limit of infinite dilution both series of samples (fresh and mixed solution) approach a hydrodynamic radius, *Rh* ~ 25 nm, which is close to the value obtained with ET13/NA.

The particle size distributions for the fresh and degraded Habon G solutions are shown in Figs. 10 and 11.

There are a significant number of particles with an approximate hydraulic radius around 10 nm for the fresh solutions and about 5 nm for the sheared solutions. These cannot be neglected as they constitute about 10% of the intensity which corresponds to 16 times more of these smaller particles than the larger ones. Spherical micelles of cationic surfactants of this type typically have diameters of about 5 nm so these smaller particles for the fresh solutions are not spheres. The components with sizes of 1000 nm or more in Fig. 10 for fresh 4 mM Habon G may correspond to aggregates in a network of micelles or might be caused by some impurities. These large particles disappeared in the sheared solution (Fig. 11). Thus Habon G which is far more effective as a drag reducing additive, has micelles considerably larger than both Ethoquads and the Arquad. The micelles grow very quickly with increase in concentration and they are broken down somewhat by intensive shearing.

Comparative measurements

We also tested the influence of tap water on the size of micelles of 3 mM ET13/NA to compare with results with 5 mM and 12.5 mM NA in distilled water. The results are shown in Fig. 4. There is an increase in *Rh* of 10 to 15% when tap water replaces distilled water. An increase in first normal stress differences at high shear rates for tap water over distilled water was also noted for a similar cationic surfactant system at high shear rates [25].

Finally, we investigated the influence of vigorous mixing on micelle size in solutions with ET13, AR and chemically pure CTAC, the latter being similar in composition to AR but without any impurities. These micelles are much smaller than those of Habon G. The results are given in Figs. 12–14 and are summarized in Table 3. The lower concentration CTAC solution had slightly smaller micellar size than the AR solution as expected. The width of its distribution was narrower, however, probably reflecting the narrower molecular weight distribution of the alkyl group compared with the commercial AR. In other rheological experiments, AR16 and CTAC solutions give almost identical results at equal concentrations [26].

The shearing (mixing) had no effect on the average hydrodynamic radius of ET13/NA and only small but significant effects on AR/NA, and CTAC/NA, the change in the latter two being 6 and 7%. The width of distributions in the sheared solution decreased only a little in the ET13/NA solution, which is probably within the scatter of data, but with the small decrease of *Rh* in the AR/NA solution there was a 25% decrease in *w*. The distribution of the fresh CTAC solution was narrower with no change in *w* after shear.

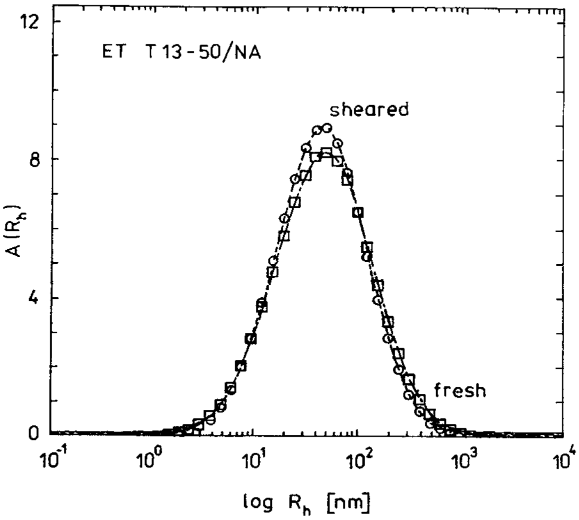

Fig. 12 Distribution functions in ET13/NA solutions with 5/12.5 mM concentration; the influence of 2 h of intensive shearing

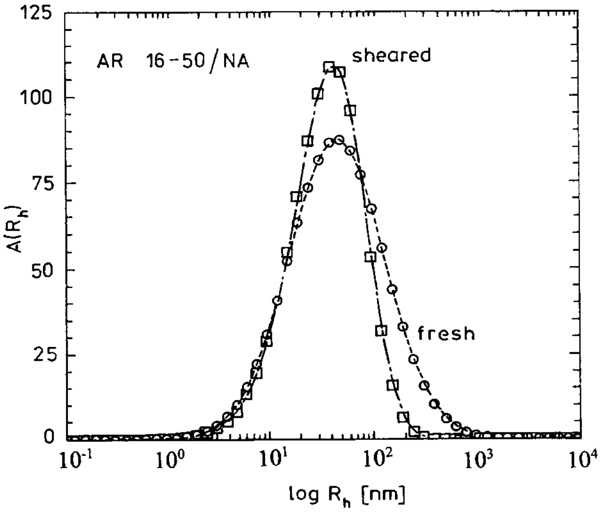

Fig. 13 Distribution functions in AR16/NA solutions with 5/12.5 mM concentration in distilled water; the influence of shearing

Comparison of the AR and ET13 5/12.5 mM fresh solutions to results with ET13/NA = 5/7.5 mM and AR/NA = 3/9.37 mM plotted in Fig. 3 and in Fig. 7 shows that both of these more concentrated solutions contain somewhat larger micelles and exhibit somewhat larger widths of distributions.

Electron microscope photographs of ET13/NA micelles in solution were published by Myska and Stern [20]. Although the particles shown in the photographs were dried which could affect the micelle structure, their size is in accordance with the measurements presented here. The photographed particles are mostly anisotropic

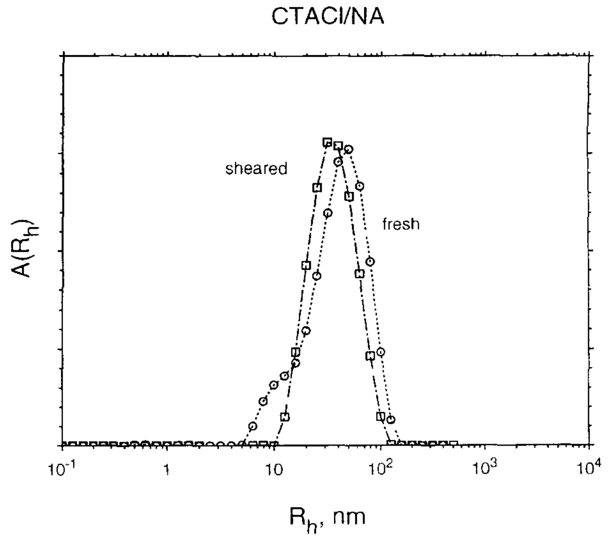

Fig. 14 Distribution functions in CTAC/NA solutions with 1.25/12.5 mM concentration in distilled water, the influence of shearing

Table 3 Effect of Shear on *Rh* and *w*

	Rh, nm	Distribution width, *w*, In%
ET13/NA = 5/12 5 mM		
Fresh solution	35.4	107%
Sheared solution	35.8	101%
AR/NA = 5/12.5 mM		
Fresh solution	33.5	107%
Sheared solution	31.6	80%
CTAC/NA = 1.25/12.5 mM		
Fresh solution	31.6	58%
Sheared solution	29.3	57%

with average lengths of 90–130 nm. Polydispersity can also be seen in the micrographs.

The hydrodynamic radii of ET13/NA micelles obtained from these light scattering measurements range from about 20–35 nm. These radii are consistent with the electron microscope lengths given in [20] as Hu, Wang and Jamieson [14] estimate that the relation between *Rh* and the length of the micelle gives a micelle length approximately four times *Rh*.

Conclusions

1) Micelle size, represented by hydrodynamic radius, increases with the concentration of the solution as well as with increase in counterion concentration. *Rh* increases when tap water replaces distilled water.

2) Micelles of Habon G are approximately four times larger than micelles of Ethoquad T13 or Arquad 16. Habon G solutions reach maximum drag reduction effectiveness at about one-fifth of the concentration for the other surfactants.

3) Considerable polydispersity was found in these surfactants. A purer sample similar in composition to AR had similar average size but a narrower distribution than the commercial sample.

4) Intensive shearing (mixing) reduces the size of large micelles in Habon G solutions while the sizes of the smaller micelles in solutions of Ethoquad T13, Arquad 16 and pure CTAC are not greatly affected by shearing.

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