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ORIGINAL CONTRIBUTION

J. Nakarapanich
T. Barameesangpet
S. Suksamranchit
A. Sirivat
A. M. Jamieson

Rheological properties and structures of cationic surfactants and fatty alcohol emulsions: effect of surfactant chain length and concentration

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J. Nakarapanich · T. Barameesangpet
S. Suksamranchit · A. Sirivat (✉)
Petroleum and Petrochemical College
Chulalongkorn University
Bangkok 10330, Thailand
e-mail: anuvat.s@chula.ac.th
Tel.: +66-2-2184131
Fax: +66-2-6117221

A. M. Jamieson
Department of Macromolecular Science
Case Western Reserve University
Cleveland, Ohio 44106-2699, USA

Abstract Rheological and optical properties of cetyltrimethylammonium chloride (CTAC)/fatty alcohol (FA), behenyltrimethylammonium chloride (BTAC)/FA and CTAC/FA/hydroxyethyl cellulose (HEC) emulsions have been studied with particular emphases on the effects of FA content, the difference in the chain length of the hydrophobic groups between CTAC and BTAC, and the addition of a water soluble polymer, HEC. The effects of the FA content are to accelerate the structure development during the aging period and to increase the storage modulus, the yield stress, and the zero-shear-rate viscosity in the three emulsion systems investigated. At a low FA content of 2% w/w, lamellar and vesicular aggregates and isolated multilamellar vesicles can be observed in the CTAC/FA and BTAC/FA emulsions, respectively. At a high FA content of 6% w/w or with an excess of FA present, networklike structures and sunflower-like structures form, respectively, instead, inducing a higher entanglement storage modulus and a higher yield stress relative to those emulsions with a low FA content. The effect of adding HEC to the CTAC/FA emulsion is to reduce the entanglement storage modulus and the yield stress, consistent with the optical observation that the presence of the polymer disrupts the formations of lamella and vesicular aggregates and network structures.

Key words Emulsion · Fatty alcohol · Cationic surfactants · Rheology · Optical properties

Introduction

In formulating many pharmaceutical and cosmetic products for skin, rheological properties play an important role in mixing, pumping, packaging, or packing and final consumer appeals [1]. Typically, cosmetic emulsions are mixtures of one or two fatty alcohols (FAs) and ionic surfactants [2, 3], whose rheological properties can be tailored as required by specific needs, by means of adding another component or by varying component compositions.

FA is one type of fatty amphiphiles [4] often used as the emulsifying wax. The alcohol is, in a commercial form, usually a homologous admixture of cetyl and stearyl alcohols, as in cetostearyl alcohol. The pure long-chain alcohols exist in at least three polymorphic forms. The high-temperature α form or α-crystalline polymorph separates first from the melt and is stable over a narrow temperature range. In the α form, the hydrocarbon chains are hexagonally packed and there is a rotation about the long axis of the molecule. In water, this polymorph forms a hydrated crystal in which the thickness of the water layers incorporated between the polar groups is limited by the van der Waals attractive force which balances the osmotic repulsion. In the presence of very small quantities of ionic surfactant, the amount of swelling increases markedly to give a swollen α-crystalline gel phase which is characterized by a lamellar structure of alternating bilayers of emulsifying wax separated by layers of water. The β form, in which the hydrocarbon chains are nonrotating, and the tilted γ form can coexist, although the β form is usually in excess or at a higher proportion. The pure cetyl and stearyl alcohols in water are in the β- and γ-crystalline forms at room temperature, while the mixed homologous alcohols exist as the α-crystalline form.

In its most basic form a surfactant molecule possesses a polar hydrophilic head group and a nonpolar paraffinic group. At sufficiently high concentrations above the critical micelle concentrations, in an aqueous environment, surfactants can form a variety of structures and phases, including spherical and elongated micelles, lamellae, and hexagonal and cubic phases [4–6]. Fundamental studies, of both rheological and structural aspects, of these lyotropic systems are still an active research area [7, 8] because of wide scientific and technical interests.

Whereas rheological and optical properties of ternary systems consisting of water, an FA, and an anionic surfactant have been well investigated, less is understood rheologically and optically about an emulsion consisting of mixed FAs and a cationic surfactant, in particular the role of the surfactant structure. In this article, we present and discuss the effects of FA content, the cationic surfactant chain length, and the addition of a long-chain polymer on the rheological and optical properties of emulsions consisting of water, an admixture of FAs and cationic surfactants, and a water-soluble polymer.

Experimental

Materials

Cetyltrimethylammonium chloride [CTAC, $C_{16}H_{33}N(CH_3)Cl$] and behenyltrimethyl ammonium chloride [BTAC, $C_{22}H_{45}N(CH_3)Cl$] were obtained from Unilever Holdings and were used as received. Both surfactants are cationic but with one essential difference: CTAC has 16 carbon atoms in the alkyl chain, whereas BTAC has 22 carbon atoms. Hydroxyethyl cellulose (HEC), a long-chain nonionic polymer, was also obtained from Unilever Holdings and was used as received. The FA or amphiphile, a homologous admixture of cetyl alcohol (15%) and stearyl alcohol (85%), was obtained from Unilever Holdings and was used as received. These amphiphiles exist in the α-crystalline form at room temperature [3] and its melting temperature is about 54 °C. Deionized, distilled water was used as the solvent in preparing the emulsions studied.

Sample preparations

For the emulsions of CTAC/FA and BTAC/FA, the preparation was as follows. A weighed amount of FA was heated to 75 and 85 °C, respectively, before a weighed amount of surfactant was added. The mixture was stirred and poured into a main mixer consisting of water preheated to 75 °C. The mixture was homogenized by a blade mixer at 110 rpm for 2 min and at 210 rpm for 15 min. The resultant emulsion was cooled to and maintained at 45 °C and was stirred at 110 rpm for 10 min as an appropriate amount of water was added.

To prepare an HEC solution, water was stirred vigorously until a vortex occurred and a known amount of HEC was very slowly added so that the HEC particles did not form lumps. The solution was stirred by a blade mixer at 550 rpm for 30 min at 25 °C.

For the emulsions of CTAC/FA/HEC, the preparation was as follow. A weighed amount of FA was heated to 75 °C before a weighed amount of surfactant was added. The mixture was stirred and poured into a main mixer consisting of an aqueous HEC solution preheated to 75 °C. The mixture was homogenized by a blade mixer at 110 rpm for 2 min and at 210 rpm for 15 min. The resultant emulsion was cooled to and maintained at 45 °C and was stirred at 110 rpm for 10 min as an appropriate amount of water was added.

Instruments

Laser scanning confocal microscope

A laser scanning confocal microscope (Zeiss, model LSM 410) was used to study emulsion optical properties [9]. The system consists of a laser (argon ion, $\lambda = 514.3$ nm), a scanner unit, and a detector unit. A laser beam is redirected by a beam splitter and is scanned horizontally over the sample via the scanner and the objective lens. A pinhole located in the focal plane in front of the detector ensures that only light reflected or emitted from a particular and very narrow focal plane will reach the detector. The pinhole effectively suppresses light emanating from other planes from passing through to the photodetector, so it greatly improves the limited depth of focus normally available with a typical optical microscope. By using the scanning technique, images of one focal plane can be generated one at a time. Repeated scannings give three-dimensional images after proper digital signal processing. A magnification of 1000 times was used throughout in obtaining the micrographs shown. This optical technique allows us to avoid the conventional approach of relating the microstructures of a gel to its rheological properties by using a freeze-fracture transmission electron microscope [11–14], which may introduce artifacts such as ice-crystal formation.

Rheometer

A rheometer (Rheometrics, model ARES) was used to obtain emulsion rheological properties. The system consists mainly of a motor, two transducers, and a temperature control unit, which allows the testing temperature to be varied between −150 and 600 °C with an accuracy of ± 0.1 °C. The fixture used was a 50-mm cone and plate, with a gap spacing of 0.051 mm and a cone angle of 4°. The testing temperature was normally set at 26 °C. Two sets of measurements were carried out. In the oscillatory test, a strain sweep test was carried out to determine the proper strain to be used; the lower limit was set by the transducer used and the upper was set by the extent of the linear viscoelastic regime of the emulsion. In this test, we measured G', the storage modulus, and G'' the loss modulus, as functions of frequency, which was varied between 0.1 and 100 rads^{-1}. Because emulsion G' functions do not have apparent plateaus at high frequencies, G_N^0, the liquidlike entanglement storage modulus, is then estimated as $G'(\omega = 100\ \text{rads}^{-1})$. In the steady-state test, we measured the shear stress and viscosity versus strain rate, which was varied from 0.01 to 100 s^{-1}. In most of the emulsions investigated, the viscosity versus strain rate curves do not have apparent Newtonian plateaus at low frequencies; therefore, η_0, or the zero-shear-rate viscosity is estimated as $\eta(\gamma = 0.01\ \text{s}^{-1})$. From the stress versus strain rate curve, the Bingham stress is obtained from the extrapolation of the stress towards the zero-strain rate.

Results and discussion

Effect of aging time

Viscosity measurements of CTAC/FA, BTAC/FA, and CTAC/FA/HEC emulsions were carried out at room temperature at 1, 4, 7, 14, 21, and 28 days after preparation in order to monitor the change in viscosity as a function of aging time. For the CTAC/FA and BTAC/FA emulsions at a weight ratio of 1.0/2.0, the zero-shear-rate viscosities, approximated as the viscosity at a shear rate of 0.01 s^{-1}, reach their equilibrium values within an aging period between 14 and 18 days. For the CTAC/FA and BTAC/FA emulsions at a weight ratio of 1.0/6.0, the zero-shear-rate viscosities attained their equilibrium values within aging periods of 7 and 14 days, respectively. The larger amount of FA present in the emulsions and the shorter surfactant chain length seem to accelerate the aging process of the emulsions studied.

For the CTAC/FA/HEC emulsions at weight ratios of 1.0/2.0/0.5 and 1.0/6.0/0.5, the zero-shear-rate viscosities remain unchanged at their initial values over an aging period of 7 days, thereafter they increase dramatically by an order of magnitude over an aging period between 7 and 10 days, and finally reach the equilibrium values after an aging period of 21 days. The presence of HEC delays the aging process of both emulsions.

Effect of FA content and surfactant type

To investigate the effect of FA concentration and surfactant type, both oscillatory and steady-state measurements were carried out to determine $G'(\omega)$, $G''(\omega)$ and $\eta(\gamma)$ of the CTAC/FA and BATC/FA emulsions at weight ratios between 1.0/1.0 and 1.0/8.0. $G'(\omega)$ and $G''(\omega)$ of both CTAC/FA and BTAC/FA emulsions exhibit a typical power-law dependence on frequency with respective scaling exponents less than 2 and 1, the characteristic values for the terminal zone of a liquid [15]. Furthermore, within the range of the FA concentration investigated, $G'(\omega)$ is always greater than $G''(\omega)$. This experimental evidence suggests that the emulsions investigated are gel-like [1], even though the dispersed phase concentrations are rather low. Furthermore, for the emulsions at high FA contents, both $G'(\omega)$ and $G''(\omega)$ no longer obey the power-law dependence at high frequencies; these are the characteristics of $G'(\omega)$ and $G''(\omega)$ in the transition zone.

G_N^0, the liquidlike entanglement storage modulus, is shown in Fig. 1 versus N_{FA}/N_{CTAC} and N_{FA}/N_{BTAC}, the FA and surfactant molar ratios, of CTAC/FA and BTAC/FA emulsions. G_N^0 of both emulsions are increasing functions of the molar ratios. For the emulsions with molar ratios lesser than 4, the measured G_N^0 values of the two emulsion systems are comparable or nearly the same. At molar ratios above 4, G_N^0 of the CTAC/FA emulsions are significantly greater than those of the BTAC/FA emulsions. The saturated G_N^0 values, at a molar ratio of 10, are approximately 2×10^4 and 5×10^3 Pa for the CTAC/FA and BTAC/FA emulsions, respectively.

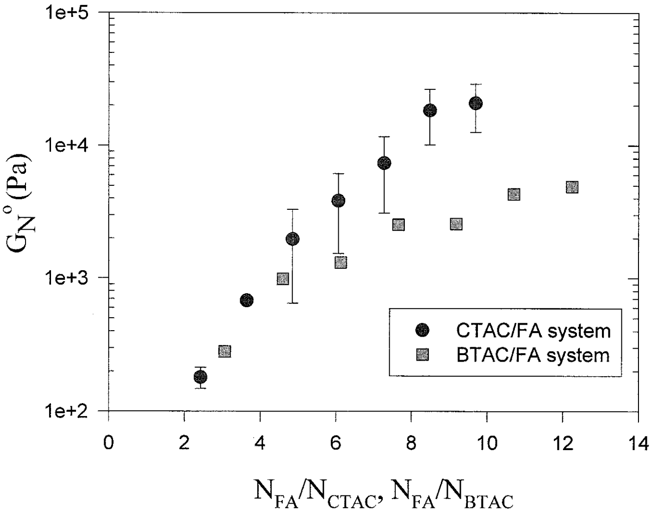

Fig. 1 The liquidlike entanglement storage modulus, G_N^0, versus N_{FA}/N_{CTAC} and N_{FA}/N_{BTAC} or the fatty alcohol (*FA*)/cetyltrimethylammonium chloride (*CTAC*) and FA/behenyltrimethylammonium chloride (*BTAC*) molar ratios of CTAC/FA and BTAC/FA emulsions at 26 °C

τ_B, the Bingham stress, is shown in Fig. 2 versus N_{FA}/N_{CTAC} and N_{FA}/N_{BTAC}. The Bingham stresses of both emulsion systems increase monotonically with the respective molar ratios. Similar to the G_N^0 behavior described, at molar ratios less than 4, the measured τ_B values of both emulsion systems are comparable. At molar ratios greater than 4, the measured τ_B values of the CTAC/FA emulsions become greater than those of the BTAC/FA emulsions; the saturated τ_B values are 3×10^2 and 1×10^2 Pa, respectively.

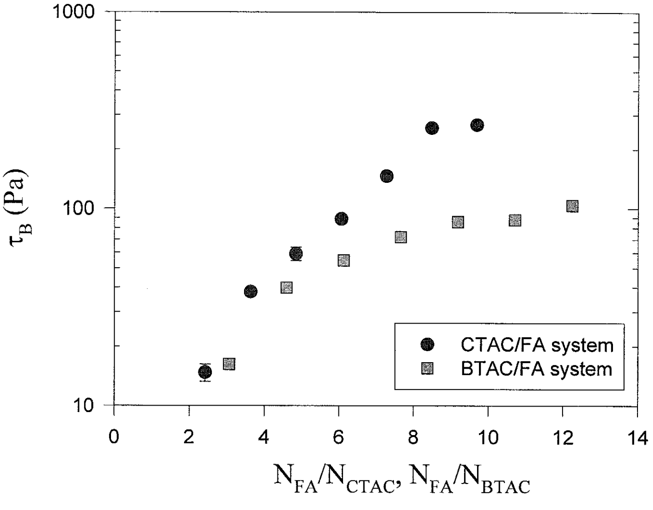

Fig. 2 The Bingham stress, τ_B, versus N_{FA}/N_{CTAC} and N_{FA}/N_{BTAC} or the FA/CTAC and FA/BTAC molar ratios of CTAC/FA and BTAC/FA emulsions at 26 °C

Plots of η_0, the zero-shear-rate viscosity, versus N_{FA}/N_{CTAC} and N_{FA}/N_{BTAC} are shown in Fig. 3. η_0 of both systems increase with FA content. At any given molar ratio, the zero-shear-rate viscosity of the CTAC/FA emulsion is always greater than that of the BTAC/FA emulsion. The saturated value of η_0 of the BTAC/FA system is approximately 5×10^3 Pas, whereas the corresponding value for the CTAC/FA system cannot be determined from this figure.

The observed quantitative differences in rheological properties, G_N^0, τ_B, and η_0 between the CTAC/FA and BTAC/FA emulsions can be explained through examining their structures. Optical micrographs taken from the CTAC/FA and BTAC/FA emulsions at an FA content of 2% w/w and at 26 °C are shown in Fig. 4a and b. For the CTAC/FA emulsion (Fig. 4a), the structures observed were isolated lamellar and vesicular aggregates, whereas for the BTAC/FA emulsion (Fig. 4b), the structures observed were isolated multilamellar vesicles appearing as a nearly concentric structure [13].

The interactions between the cationic surfactants, CTAC or BTAC, and the FA give rise to the swollen α-crystalline gel phase [3]. The gel phase is characterized by a lamellar structure of bilayers of the surfactant molecules and the emulsifying FA molecules separated by layers of water. The interaction forces between the ionic surfactants and an emulsifier such as an FA mainly consist of the electrostatic force between charges and the hydrophobic force of the hydrophobic portion. The formation of the lamellar and vesicular aggregate structure for the CTAC/FA emulsions at low FA contents ($N_{FA}/N_{CTAC} < 4$) occurred through fusions of small vesicles and lamellar bilayers, where the main binding force was electrostatic in nature. CTAC has the chemical composition of $C_{16}H_{33}N(CH_3)Cl$, whereas the BTAC components are $C_{22}H_{45}N(CH_3)Cl$. BTAC has a longer hydrocarbon chain than CTAC and can interact with an FA at a lesser degree. The interaction between carbon chains of BTAC due to the hydrophobic force is greater than that of CTAC and the chains tend to avoid water. These two interactions in the BTAC/FA emulsion system are the possible causes which resulted in the production of the multilamellar vesicle structure rather than the lamellar and vesicular aggregates.

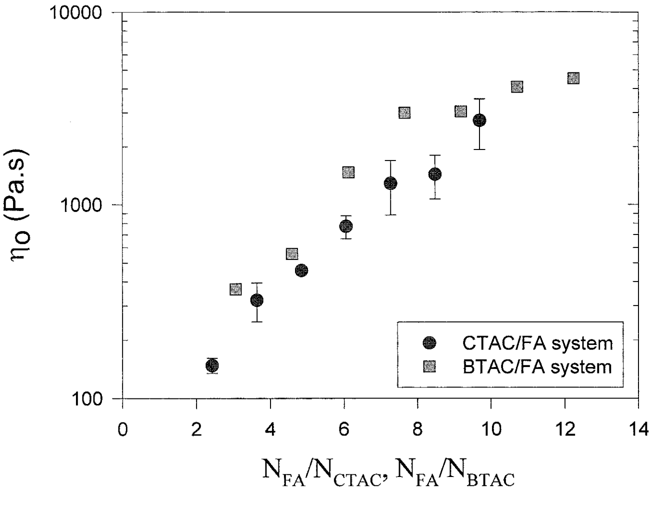

Fig. 3 The zero-shear-rate viscosity, η_0, versus N_{FA}/N_{CTAC} and N_{FA}/N_{BTAC} or the FA/CTAC and FA/BTAC molar ratios of CTAC/FA and BTAC/FA emulsions at 26 °C

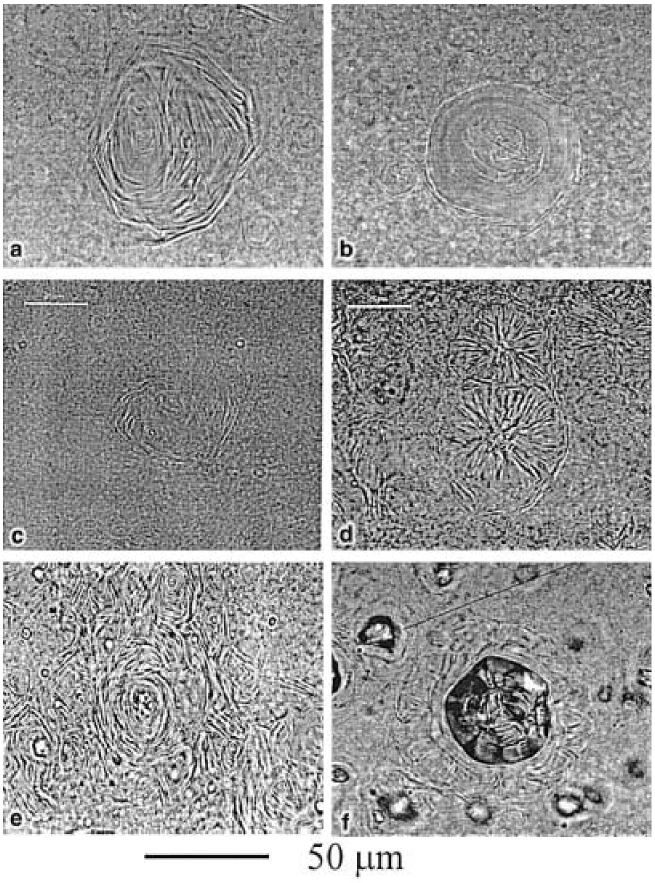

Fig. 4 Micrographs of equilibrium structures in the emulsions at 26 °C: **a** CTAC/FA = 1.0/2.0% w/w; **b** BTAC/FA = 1.0/2.0% w/w; **c** CTAC/FA = 1.0/4.0% w/w; **d** BTAC/FA = 1.0/4.0% w/w; **e** CTAC/FA = 1.0/4.0% w/w; **f** BTAC/FA = 1.0/4.0% w/w

In both emulsion systems, the entanglement storage moduli and the Bingham stresses of the two emulsion systems at low FA contents ($N_{FA}/N_{CTAC} < 4$) can be expected and were found to be nearly the same. The finding can be attributed to two factors: both structures observed consist of the same basic component, the lamellar bilayer; and the comparable dispersed sizes of the CTAC/FA and BTAC/FA emulsions at a 1.0/2.0 weight ratio are 27 and 32 μm, respectively. On the other hand, the slight difference in the viscosity values between the CTAC/FA and BTAC/FA emulsions at low FA contents (N_{FA}/N_{CTAC} < 4) can be attributed to the physical difference in the structures. The nearly concentric multilamellar vesiclular structure can resist shear deformation better than the lamellar and vesicular aggregate which consists of fusions between vesicles and lamellar bilayers.

Optical micrographs taken from the CTAC/FA and BTAC/FA emulsions at an FA content of 6% w/w and at 26 °C are shown in Fig. 4e and f. For the CTAC/FA emulsions, a nearly continuous network of lamellar and vesicular aggregates can be observed; the excess FA exists as small droplets inside the vesicles or as sheets between the lamellar bilayers. The structure is evidently similar to the structures observed in the CTAC/cetyl alcohol emulsion [16, 17].

For the BTAC/FA emulsion, an isolated structure consisting of an FA droplet surrounded symmetrically by lamellar and vesicular aggregates, or a sunflower-like structure, appeared. The larger amount of excess FA present in the BTAC/FA emulsion clearly provides solid evidence that the longer carbon chains of BTAC interact with FA molecules to a lesser degree and, therefore, a smaller number of FA molecules are required to create the pseudoequilibrium structure observed.

At an N_{FA}/N_{CTAC} ratio above 4 G_N^0 and τ_B values of the CTAC/FA emulsions are greater than those of the BTAC/FA emulsions, as shown in Figs. 1 and 2. This finding is simply the consequence of the formation of the nearly continuous network of lamellar and vesicular aggregates in the CTAC/FA emulsions, instead of the sunflower-like structures which exist in isolation in the BTAC/FA emulsions. Since longer BTAC carbon chains tend to interact amongst themselves through the weak hydrophobic force rather than with FA molecules through the electrostatic force, it can be expected that G_N^0 and τ_B of the BTAC/FA emulsions at an N_{FA}/N_{CTAC} ratio above 4, or with excess FA, have substantially lower values.

The larger values of η_0 observed for the BTAC/FA emulsions at an N_{FA}/N_{CTAC} ratio above 4 can be attributed to some physical differences in the structures observed: excess FA droplets seen in the BTAC/FA emulsions were crystalline domains whose molecules were held together by the electrostatic force.

Effect of polymer additive

Additional experiments were carried out to investigate the effect of adding a water-soluble polymer, HEC, on the rheological and optical properties of CTAC/FA emulsions. Plots of G_N^0 and τ_B of CTAC/FA and CTAC/FA/HEC emulsions versus N_{FA}/N_{CTAC} or the FA/CTAC molar ratio are shown in Figs. 5 and 6. Both G_N^0 and τ_B increase with the FA content in nonlinear manners. The effect of the additive polymer, HEC, on the CTAC/FA emulsions is twofold. At an N_{FA}/N_{CTAC} ratio less than 4, both G_N^0 and τ_B are reduced; at an N_{FA}/N_{CTAC} ratio of 4, they decrease by a factor of 2. Similarly, at an N_{FA}/N_{CTAC} ratio greater than 4, both G_N^0 and τ_B are reduced, more than a factor of 2 at an N_{FA}/N_{CTAC} ratio of 8.5. On the other hand, for the emulsion with an N_{FA}/N_{CTAC} ratio of approximately 4, G_N^0 and τ_B retain the same values with the addition of HEC at 0.5% w/w. The CTAC/FA emulsion with an N_{FA}/N_{CTAC} ratio of approximately 4 represents an emulsion with a stoichiometric ratio between CTAC and

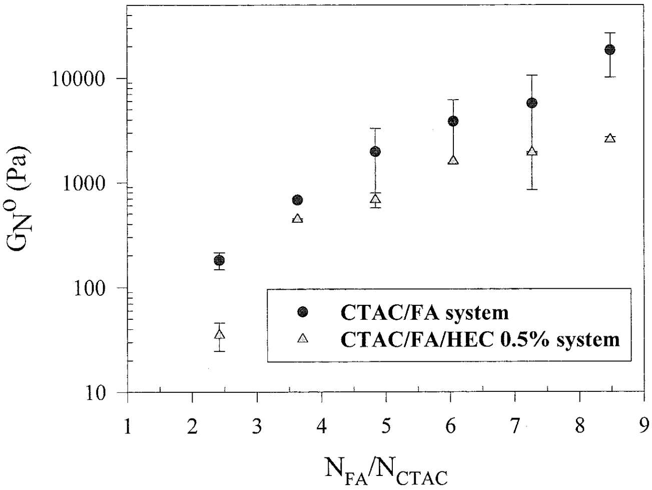

Fig. 5 G_N^0 versus N_{FA}/N_{CTAC} or the FA/CTAC molar ratio of CTAC/FA and CTAC/FA/hydroxyethyl cellulose (*HEC*) emulsions at 26 °C

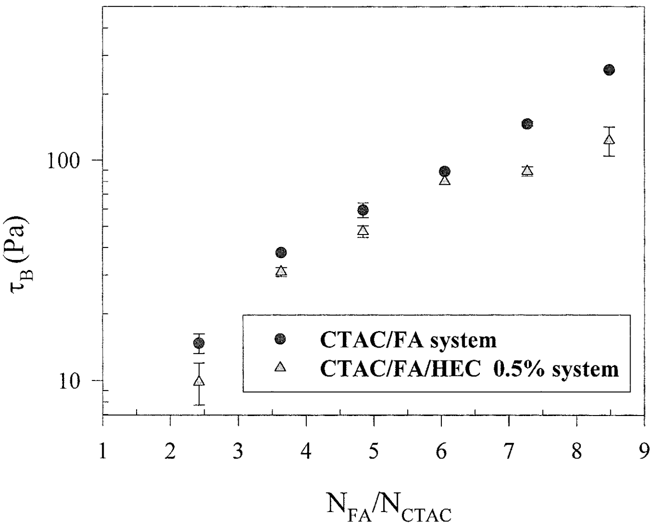

Fig. 6 τ_B versus N_{FA}/N_{CTAC} or the FA/CTAC molar ratio of CTAC/FA and CTAC/FA/HEC emulsions at 26 °C

FA:FA molecules were all consumed in creating the emulsion structures.

The effect of adding HEC into the CTAC/FA emulsions on the zero-shear-rate viscosity is, however, opposite to the effects on G_N^0 and τ_B. A plot of η_0, the zero-shear-rate viscosity, versus N_{FA}/N_{CTAC} or the FA/CTAC molar ratio of CTAC/FA and CTAC/FA/HEC emulsions, is shown in Fig. 7. At an N_{FA}/N_{CTAC} ratio less than 4 or greater than 4, the presence of HEC at 0.5% w/w increases the zero-shear-rate viscosity; however, for the emulsion with an N_{FA}/N_{CTAC} ratio of approximately 4, the zero-shear-rate viscosity retains its value with HEC of 0.5% w/w added.

The reductions in G_N^0 and τ_B of the CTAC/FA emulsions at an N_{FA}/N_{CTAC} ratio less than or greater than 4 can be related to the changes in the structures observed in the optical micrographs. The structures of the CTAC/FA and CTAC/FA/HEC emulsions at an FA content of 2% w/w are shown in Fig. 8a and b, respectively. Adding HEC at 0.5% w/w reduces the size of the isolated lamellar and vesicular aggregates. A lower number of FA molecules are able to interact with CTAC molecules, as evidenced by crystalline droplets of FA which can be seen in Fig. 8b. Since only the domains of these structures contribute toward the emulsion elasticity and yield stress, evidently the reductions in G_N^0 and τ_B can be expected to occur. The structures of the CTAC/FA and CTAC/FA/HEC emulsions at an FA content of 6% w/w are shown in Fig. 8c and d, respectively. The presence of HEC at 0.5% w/w apparently destroys the nearly continuous network of lamellar and vesicular aggregates typically found in the CTAC/FA emulsions at an N_{FA}/N_{CTAC} ratio greater than four. Multilamellar vesicles and lamellar and vesicular aggregates now exist in isolation, as can be seen in Fig. 8d.

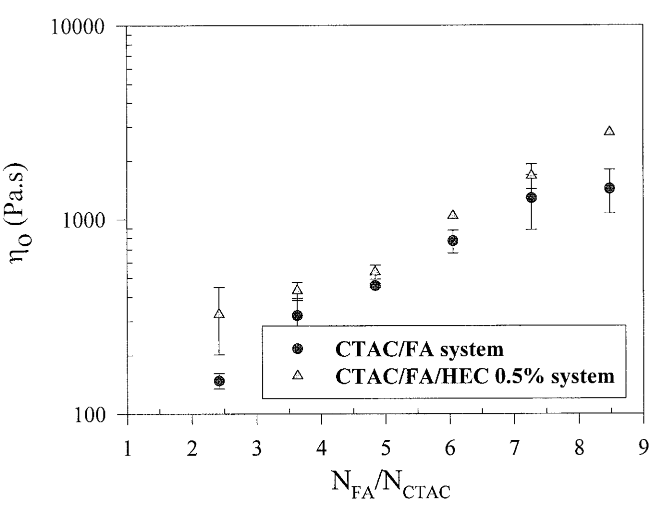

Fig. 7 η_0 versus N_{FA}/N_{CTAC} or the FA/CTAC molar ratio of CTAC/FA and CTAC/FA/HEC emulsions at 26 °C

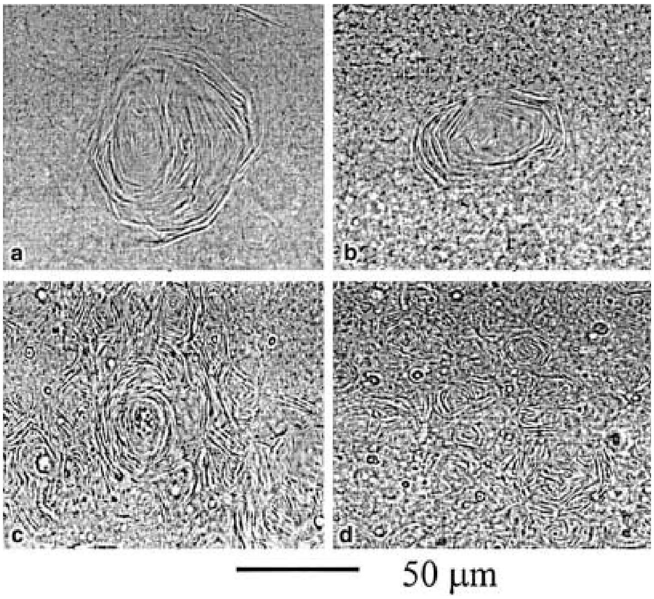

Fig. 8 Micrographs of the equilibrium structures of the emulsions at 26 °C: **a** CTAC/FA = 1.0/2.0% w/w; **b** CTAC/FA/HEC = 1.0/2.0/0.5% w/w; **c** CTAC/FA = 1.0/6.0% w/w; **d** CTAC/FA/HEC = 1.0/6.0/0.5% w/w

The larger zero-shear-rate viscosity of the CTAC/FA/HEC emulsions relative to those of the CTAC/FA emulsions possibly arises from two factors. The addition of HEC generally produces a larger dispersed phase volume. HEC may have acted as tie molecules or as network partitioners between multilamellar vesicles or lamellar and vesicular aggregates. Clearly, more studies are required to fully understand the phenomena observed here.

Conclusion

The effect of aging time, FA content, and surfactant type on rheological and optical properties of three emulsion systems were investigated and reported. The BTAC/FA emulsions require a slightly longer period to reach the pseudoequilibrium states relative to the CTAC/FA emulsions. Emulsions with higher FA content generally need shorter structure formation times. The effect of adding HEC is to disrupt the structure formation and, therefore, to increase the aging time required to reach equilibrium.

The effect of the FA content generally is to increase the storage moduli, the yield stresses and the zero-shear-rate viscosity in both CTAC/FA and BTAC/FA emulsions. The effect of the surfactant chain length on the emulsion optical property is dramatically observed. For a low FA content of 2% w/w, lamellar and vesicular aggregates are observed in the CTAC/FA emulsion, whereas an isolated multilamellar vesicular structure forms in the BTAC/FA emulsion. At a high FA content of 6% w/w, a nearly continuous networklike structure and a sunflower-like structure form in the CTAC/FA and BTAC/FA emulsions, respectively.

The effect of adding HEC is to reduce both G_N^0 and τ_B for the CTAC/FA emulsions at low and high FA contents. The presence of the polymer disrupts the formation of the lamellar and vesicular aggregates and the network structures.

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