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Research Article

SURFACTANT AND OIL EFFECTS ON THE LIQUID CRYSTAL FORMATION OF SUCROSE STEARATE

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ABSTRACT

Surfactant and oil effects on the formation and the rheological properties of lypotropic liquid crystals formed by sucrose stearate (S1570) were investigated by means of phase diagrams, rheological techniques. It shows that sucrose stearate can form liquid crystal phase at much lower concentration and lower temperature by adding oleyl polyoxyethylene (20) surfactant (Brij98). Inducing isopropyl myristate （IPM）, the liquid crystal phase formed by Brij98/S1570 system transformed from hexagonal liquid crystal to lamellar liquid crystal. The rheological experiments indicate that all the liquid crystal samples have strong fake plastic property and follow a shear-thinning behavior. Dynamic measurements show that the hexagonal liquid crystal meets the general Maxwell model, exhibiting viscoelastic behavior. In the investigated frequency range, lamellar liquid crystal exhibiting the loss modulus is always lower than the storage modulus, performing as an elastomer. In addition when the mass ration of Brij98/S1570 is 5/5, shear stress and platform modulus of the lamellar liquid crystal get increased as the IPM concentration increases. Whereas at the Brij98/S1570 mass ratio of 3/7, these values decrease with this change.

Key Words: *Sucrose Stearate; Liquid Crystals; Phase Behavior; Rheological Properties; Polarizing Microscope.*

INTRODUCTION

Sucrose ester are biodegradable surfactants and used as a raw material in the variety of industry processes (Garti *et al.*, 1999). Surcrose esters possessing a wide range of hydrophilic-lipophilic balance (HLB) values, can be an outstanding emulsifier and be used in food, medicine and cosmetics (Szuts *et al.*, 2010). However, the lypotropic liquid crystal of surcrose stearate are rarely used in these fields. It may because that surcrose stearate is hardly miscible in water at a temperature lower than 44^{o}C at any concentration, unless mixed with other excellent performanced surfactants such as ethoxylated surfactants (Fanun, 2007).

Usually, mixed surfactants systems are more surface active than the individual surfactant. Such as for some purpose, in some micellar aggregates using mixed surfactants to increase the solubilization of oil and water (Kunieda *et al.*, 2001). Moreover, mixed surfactants can assemble into rich micellar aggregates than single surfactant, exhibiting different geometrical shapes and frequently fluid-like rheological properties (Wang *et al.*, 2009). For example, in a special process, it may be need to obtain a particular aggregate in a given surfactant concentration, temperature and oil additives. By inputting the second surfactant, one can adjust the phase behavior in the desired direction (Brooks *et al.*, 1994; Ghoulam *et al.*, 2004).

Recently, the mixed systems of a polyoxyethylene surfactant and a sugar based surfactant in the presence of alkanes or a complex mixture of alkanes and alkycyclohexanes have been studied intensively (Oh *et al.*, 1995). In the pseudo-ternary-component systems, the self-assembly structures of lyotropic liquid crystal are also highly affected by the oil phase. It was found that adding oil phase to the water/surfactants systems can decrease the temperature and surfactant concentration needed for the aggregate formation (Fanun *et al.*, 2006). When the oil solubilized into the palisade layer, a self-assembly structure transition from rod-shaped micelle to layer structure usually happened. While, a transition often occurred from rod-shaped micelle to spherical micelle, when the oil mainly solubilized into the hydrophobic core (Alam *et* *al.*, 2008). Therefore, the designated self-assembly structure can be obtained by either using the surfactant mixture or incorporate a proper oil component. In this work, the effects of oleyl polyoxyethylene (20) and isopropyl myristate on the formation and properties of liquid crystals formed by sucrose stearate were studied. Some interesting results were obtained.

MATERIALS AND METHODS

Materials

The sucrose stearate (S1570) was obtained from Mitsubishi-Kasei Food Corp. (Mie, Japan). The sucrose stearate compositions are 70% monoester and 30% di, tri, or polyester, and the HLB is 15. Oleyl polyoxyethylene (20) ($C_{18}EO_{20}$, Brij98) and Isopropyl myristate (IPM) were purchased from Sigma-Aldrich Chemical Company. All chemicals were used as received.

Phase diagram determination

Binary phase diagram draw process as that mixed surfactants and water were homogenized with suitable weight rations at a temperature 60-70 ºC. The water phase was added sequentially and the samples were mixed using a vortex mixer and repeated centrifugation. Temperature was increased gradually in the range of 10-70 °C. Ternary phase diagram draw process as that twenty pre-weighed mixtures of surfactants and oils were well stirred at a temperature of about 60–70 ◦C, with weight ratios varying from 0:10 to 10:0; then the water phase was added sequentially and the samples were mixed using a vortex mixer and repeated centrifugation. Samples inside and outside the phase boundary line were stored in a thermostat for 1 week for equilibration (Wang *et al.*, 2006).

Phase equilibria were determined by visual observation of the samples in normal light and also observed with a cross polarizer for anisotropy. The types of liquid crystals were identified by polarized optical microscopy.

Polarization microscopy

XP-10 polarization microscope with a maximum magnification of 1000× was applied in the microscopic observation. The relevant pictures were digitalized using a charge-coupled device camera and the proper computer hardware.

Rheological measurements

Rheological measurements were made with an American AR-2000ex rheometer. A cone-plate sensor was used with a diameter of 20 mm and a cone angle of 2°. The sample thickness in the middle of the sensor was 0.105 mm. Samples were kept in saturated water vapor during the measurements. The sample was gently inserted in to the top of the plate of the sensor, and then the plate was slowly elevated to its measuring position with constant velocity. The sample squeezed out from the sensor system was then gently removed. Measurements were carried out after 10 minutes to allow for the stress relaxation. The maximum permitted deviation in temperature was ± 0.1 °C during the measurements. Initially, controlled stress measurements were performed with all samples at 1.0 Hz, in order to determine the extent of the linear viscoelastic region. Later, frequency sweep measurements were performed at a constant stress, which was found to be in the linear viscoelastic domain in all cases when the amplitude of the deformations was very low. The frequency was varied from 0.06 to 300 $rad{\cdot}s^{-1}$. Subsequently, steady shear measurements were performed on all the samples in which the shear rate was varied (Zhao *et al.*, 2011).

RESULTS AND DISCUSSION

Phase Behavior

Oleyl polyoxyethylene 20 effect on phase behavior: According to literature (Fanun, 2007), the phase diagram of the S1570/water system was drawn in Figure 1a. A small area of liquid crystal phase was formed at the sucrose stearate concentration between 45 wt% and 70 wt% and the temperature beyond 44 °C. As presented in Figure 1b and 1c, for the two systems of Brij98/S1570/water with different mass ratio of Brij98/S1570, a large hexagonal liquid crystal area was formed at lower temperature and lower surfactant concentration. All hexagonal liquid crystal samples have the similar fan-shaped textures at different temperature, the typical picture shown in Figure 2a. Meanwhile the Brij98/S1570/water system at the Brij98/S1570 mass ratio of 5/5, formed a more larger hexagonal liquid crystal area than the system of Brij98/S1570/water which the mass ratio of Brij98/S1570 is 3/7. This phase behavior may result from that Brij98 has the highly emulsifying ability than S1570 at the low temperature range.

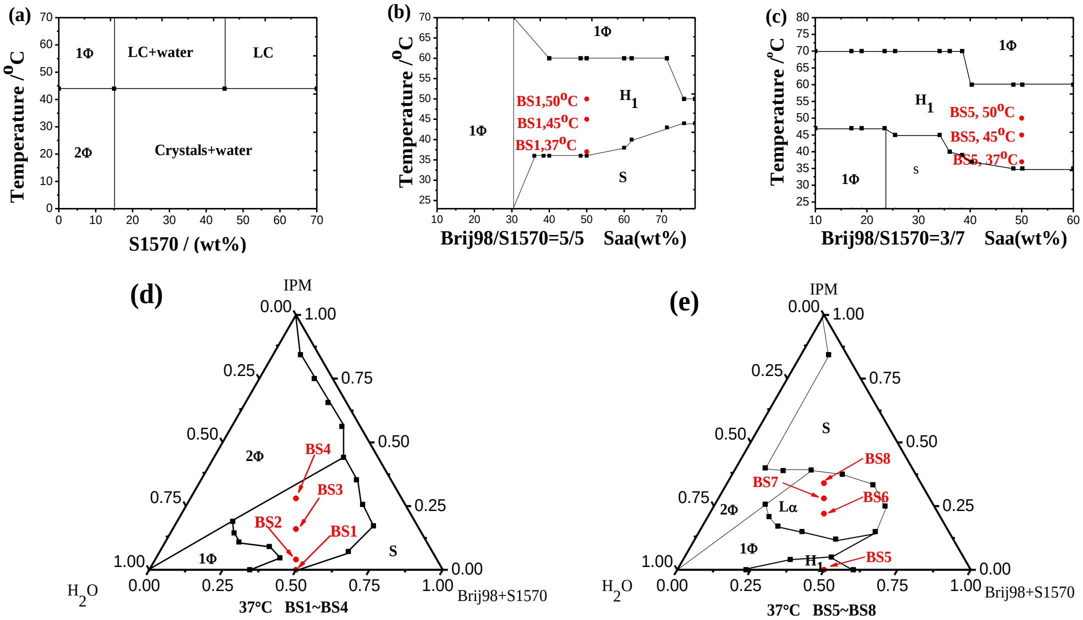

Figure 1: Phase diagrams (a) S1570/water system. (b) Brij98/S1570/water system. (c) Brij98/S1570/water system. (d) Brij98/S1570/IPM/water system, at Brij98/S1570=5/5 (w/w) and 37 °C. (e) Brij98/S1570/IPM/water system, at Brij98/S1570=3/7 (w/w) and 37 °C. 1□ isotropic solution phase; 2□, two phase region; Lα, lamellar liquid crystalline phase; H_1, hexagonal liquid crystalline phase; LC, lypotropic liquid crystalline phase; S, solid phase.

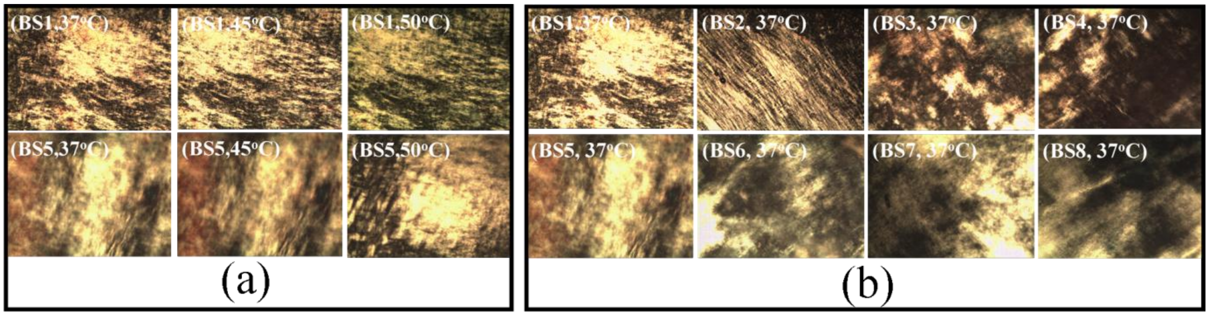

Figure 2: Textures of liquid crystals formed by the (a) Brij98/S1570/water systems at different temperature and (b) Brij98/S1570/IPM/water systems at 37 °C.

Isopropyl myristate effect on phase behavior: According to the Figure 1d and 1e, in the Brij98/S1570/water systems, inputting the oil phase of isopropyl myristate induces a transition from hexagonal liquid crystal to lamellar liquid crystal. The composition of some liquid crystal samples chosen in the above systems are listed in Table 1. Polar optical microscope investigations were done on these samples. As shown in Figure 2b, the hexagonal liquid crystal (BS1, BS5) have the typical rock-like textures. And the lamellar samples (BS2, BS3, BS4, BS6, BS7, BS8) have the oily streaks textures or marbling textures.

The liquid crystals phase transitions can be explained qualitatively using the surfactant packing parameter *Rp* (Wang *et al.*, 2006). The packing parameter is defined as $Rp = (v_L/a_S l_c)$, where a_S is the area occupied at the interface by a surfactant headgroup, l_c is the hydrophobic chain length, and v_L is the volume of the hydrophobic tail. Different aggregation shapes were formed, when the *Rp* has the diverse values. For example, sucrose stearate aqueous solution always form lamellar liquid crystals (Israelachvili *et al.*, 1976), so the packing parameter ranges of sucrose stearate is $1/2 \leq Rp \leq 1$. And the packing parameter of oleyl polyoxyethylene (20) was estimated to be 0.34. Thus the Brij98/S1570/water systems tends to form hexagonal liquid crystal in aqueous solution, which account for the mixed surfactants packing parameter ranges is $1/3 \leq Rp \leq 1/2$. The hydration of the polyoxyethylene chain decrease with increase isopropyl myristate concentration, which resulting in the effective hydrophilic head group repulsion decrease (Véronique *et al.*, 2004), therefore reducing the effective cross-sectional area per surfactant molecule (a_S). This results in an increase in packing parameter ($v_L/a_S l_c$) and thus the systems occur phase transition from hexagonal liquid crystal to lamellar liquid crystal (BS1→BS2, BS5→BS6).

Table 1: Compositions of lyotropic liquid crystal samples investigated

Sample	S1570 (wt %)	Brij98 (wt %)	IPM (wt %)	H_2O (wt %)	Phase
		mass ratio: Brij98/S1570=5/5（w/w）			
BS1	**25**	**25**	0	**50**	**H_1**
BS2	**24**	**24**	4	**48**	**L□**
BS3	**21**	**21**	16	**42**	**L□**
BS4	**18**	**18**	28	**36**	**L□**
		mass ratio: Brij98/S1570=3/7 (w/w)			
BS5	**35**	**15**	0	**50**	**H_1**
BS6	**27.3**	**11.7**	22	**39**	**L□**
BS7	**25.2**	**10.8**	28	**36**	**L□**
BS8	**23.1**	**9.9**	34	**33**	**L□**

Rheological Properties

Frequency Dependence Spectra. Oscillatory shear frequency sweep measurements were carried out on the liquid crystal samples, in order to obtain the information on the network structure of the liquid crystal phases. The frequency dependence of the storage modulus *G*□ and loss modulus *G*□□ for several liquid crystalline samples is demonstrated in Figure 3. From Figure 3, it can be clearly seen that two different mechanical spectra are shown by the investigated liquid crystal phases. The hexagonal liquid crystal samples (BS1, BS5) show traits of the general Maxwell model and exhibit viscoelastic behavior. The Lamellar liquid crystal samples (BS2, BS3, BS4, BS6, BS7, BS8) have the same type of gel-like rheogram viscoelasticity characteristics and performs as an elastomer at the higher frequencies.

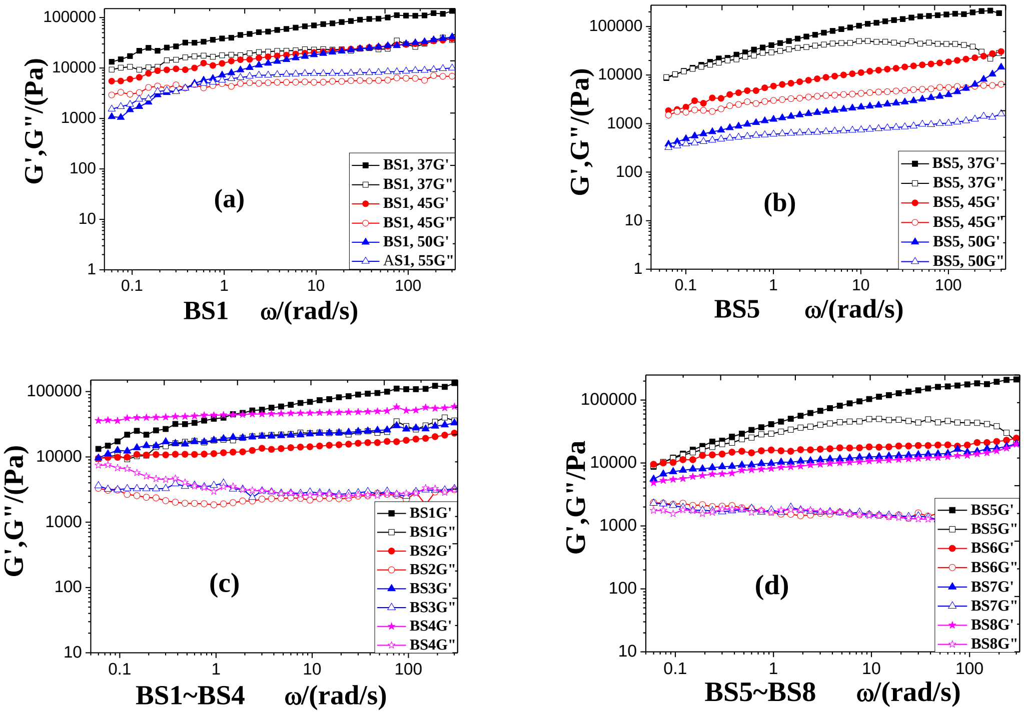

Figure 3: Storage (filled) and loss (hollow) moduli as a function of angular frequency (a) BS1 of the Brij98/S1570/water system at 37 ^{o}C, 45 ^{o}C, 50 ^{o}C. (b) BS5 of the Brij98/S1570/water system at 37 ^{o}C, 45 ^{o}C, 50 ^{o}C. (c)BS1~BS4 of the Brij98/S1570/IPM/water system at 37 ^{o}C.(d) BS5~BS8 of the Brij98/S1570/IPM/water system at 37 ^{o}C.

According to Figure 3a and 3b, for samples BS1 and BS5, the values of the dynamic moduli increase with increasing frequency with different slopes. At low frequencies, *G*″ > *G*′, showing viscous behavior, and at higher frequencies, *G*′ > *G*″, where the elastic response dominates. This means that the hexagonal samples exhibit viscoelastic behavior (Cordobes *et al.*, 1997). And the area of G″ > *G*′not very obvious, that fact must be the frequencies not lower enough. Values of *G*′ and *G*″gradually decrease for the two samples with the temperature increase from 37 to 50 ^{o}C. It can be explained that the speed-up in the Brownian movement of molecules with increase in temperature can cause a larger space between the adjoining rodlike micelles (Durreschmidt *et al.*, 2001).

According to the Figure 3(c) and Figure 3(d), for the Lamellar liquid crystal samples (BS2, BS3, BS4, BS6, BS7, BS8), *G*′ is larger by about one order of magnitude than *G*″ throughout the whole frequency range, and *G*′ presents a weak dependence on the applied frequency (Wang *et al.*, 2005). It is also noticeable that both storage and loss moduli of the two hexagonal samples (BS1, BS5) are higher than those of the lamellar liquid crystal samples (BS2, BS3, BS4, BS6, BS7, BS8). This reflects that the hexagonal liquid crystal have stronger network and more steady internal structure (Wang *et al.*, 2005). The reason of the fact is that the rodlike micelles of hexagonal liquid crystal more closely cumulate in space than the lamellar liquid crystal which is composed by bilayer micelles (Miyoshi *et al.*, 1999).

Steady Rheological Behavior: The variation of the viscosity and complex viscosity (□□) versus shear rate and angular frequency (□□) for the BS1~BS8 samples are given in Figure 4. From the Figure 4, all the samples follow a shear-thinning behavior, because the structure of liquid crystal easily oriented in the direction of the shear force. The viscosity decreases with increasing the shear rate according to the Cross law (Diat *et al.*, 1993). After fitting the rheology data to Cross law, we can notice that the *m* of all samples deviate 1, indicating all the samples behave as fake plastic fluid. In addition we can clearly find that the complex viscosity □* is bigger than the shear viscosity □. All these samples deviate from Cox-Merz rule, which indicates these systems formed liquid crystals but not other aggregates (Nemeth *et al.*, 1998).

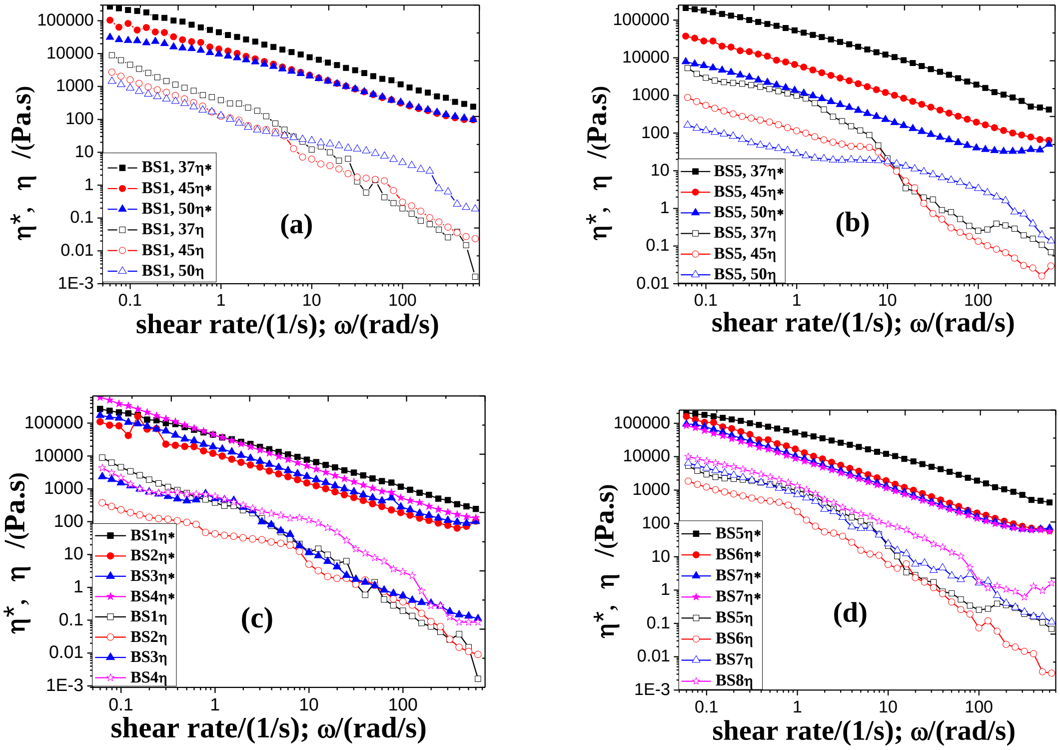

Figure 4: Viscosity and complex viscosity (□□) versus shear rate and angular frequency (□□) of the BS1~BS8 samples. (a) BS1 of the Brij98/S1570/water system at 37 °C, 45 °C, 50 °C. (b) BS5 of the Brij98/S1570/water system at 37 °C, 45 °C, 50 °C. (c)BS1~BS4 of the Brij98/S1570/IPM/water system at 37 °C.(d) BS5~BS8 of the Brij98/S1570/IPM/water system at 37 °C.

Linear Viscoelasticity Range: Oscillatory stress scan tests give the information of the change from linear viscoelasticity domain to nonlinear viscoelasticity domain. The stress sweep spectra of the liquid crystal samples (BS1~BS8) studied are shown in Figure 5. The storage modulus (*G*□) and the loss modulus (*G*□□) of all samples not essentially depend oscillatory stress in the linear viscoelastic range. The values of tan *δ* in the range of 0.07~0.58, and the tan *δ* of hexagonal liquid crystal samples are larger than that of lamellar liquid crystal samples. According to Figure 5a and Figure 5b, the hexagonal liquid crystal samples (BS1, BS5) have the same nonlinear viscoelastic response: *G*□and *G*□□decrease as the shear stress increase outside the linear viscoelastic region. Meanwhile the critical stress and platform modulus of hexagonal liquid crystal samples decrease as the temperature continue rise from 37 °C to 50 °C. That fact also can explain by Brownian movement. As can be observed in Figure 5(c) and Figure 5(d), the lamellar liquid crystals samples (BS2, BS3, BS4, BS6, BS7, BS8) show a different mechanism in the nonlinear viscoelastic response: *G*□ drops with increasing shear stress outside the linear viscoelastic region whereas *G*□□ reaches a maximum value before decreasing (Alfaro *et al.*, 2000).

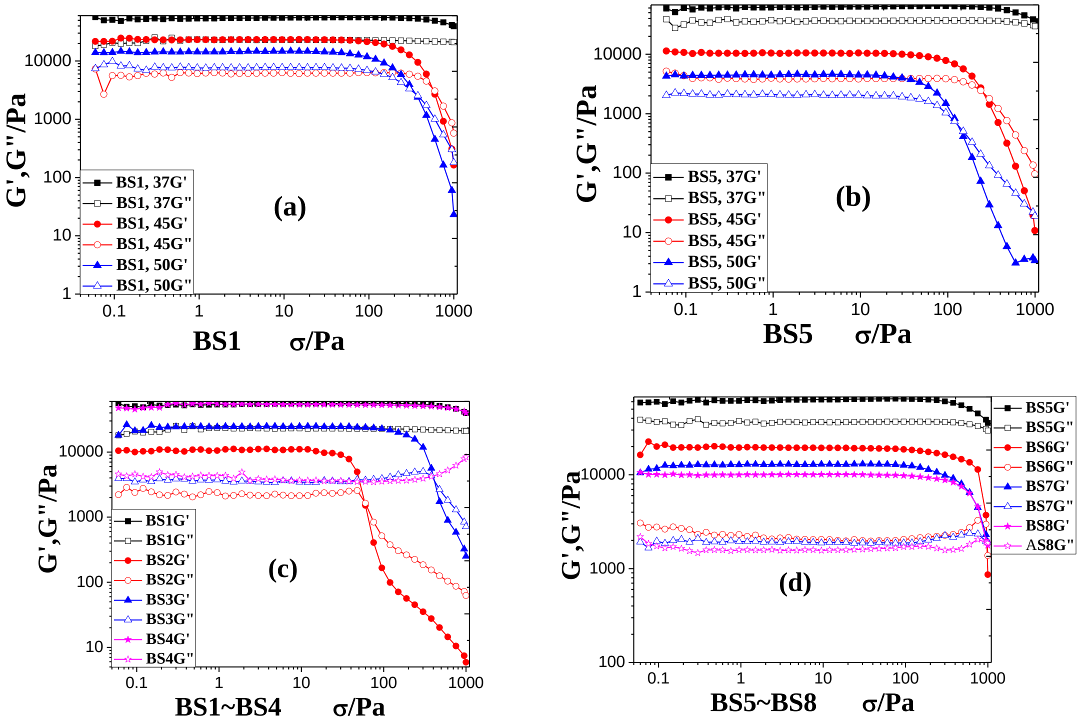

Figure 5 : Linear viscoelasticity domain of liquid crystals. (a) BS1 of the Brij98/S1570/water system at 37 °C, 45 °C, 50 °C. (b) BS5 of the Brij98/S1570/water system at 37 °C, 45 °C, 50 °C. (c)BS1~BS4 of the Brij98/S1570/IPM/water system at 37 °C.(d) BS5~BS8 of the Brij98/S1570/IPM/water system at 37 °C.

Table 2: *G*□, critical stress of *G*□and tan□ values for BS1~BS8 samples at 37°C

Sample	Phase	*G*□Pa	□$_c$/Pa	tan□
BS1	**H_1**	51322	688.8	0.41
BS2	**L□**	10705	16.0	0.21
BS3	**L□**	23853	252.2	0.15
BS4	**L□**	51322	688.8	0.07
BS5	**H_1**	63545	362.8	0.58
BS6	**L□**	19923	199.6	0.11
BS7	**L□**	12914	179.8	0.15
BS8	**L□**	9833	162.0	0.16

Table 2 is the values of the storage modulus (*G*□), the threshold shear stress (σ_c) of *G*□, and the tangent phase angle (tan□) that is defined as *G*□□/ *G*□ for all samples. From Table 2, we can find the facts: (i) The values of tan δ for hexagonal liquid crystal samples are larger than lamellar liquid crystal samples. In other words, lamellar liquid crystal samples have the predominant elastic property than hexagonal liquid crystal samples. That fact because of the rodlike micelles of hexagonal liquid crystal more close packing and the viscosity property more obvious. (ii) The *G*□and critical stress of *G*□ values all drop down when the isopropyl myristate concentration continue rise (BS2<BS3<BS4). This behavior is attributed to the isopropyl myristate molecule mainly solubilized into the lamellar liquid crystal palisade layer which can increase the lamellar liquid crystal elastic property and the storage modulus. The storage modulus increase more quickly than the loss modulus, and the tan□ will decrease with this change (Table 2). (iii) Oppsite to BS2, BS3 and BS4, the *G*□and critical stress of *G*□□$_c$ values gradually decrease when the isopropyl myristate concentration continue rise (BS6<BS7<BS8). This behavior is attributed to the isopropyl myristate molecule hardly solubilized into palisade layer and mainly incorporated into the lamellar liquid crystal hydrophobic core. So the lamellar liquid crystal change swelling and loose, and the storage modulus change smaller. The decrease in storage modulus is more sharply than that of loss modulus, and the tan□ gets increased.

Solubilization Position of Oil Phase in Different Surfactant Composition

The different solubilization position of IPM in the lamellar phase formed by the two systems can be schemed in Figure 6. It has been analysed that the polyoxyethylene has a certain hydrophobic property (Wang *et al.*, 1994), which causes a longer effective hydrophobic chain of the Brij98 than that of S1570. Usually, when the surfactant hydrophobic chain length is longer than that of oil molecule, the oil component tends to solubilize in the palisade layer. Therefore, in the system of S1570/Brij98/IPM/Water with the mass ratio of Brij98/S1570 at 5/5, oil inducing the critical stress and platform modulus increase of the lamellar liquid crystal. Opposite, when the mass ratio of Brij98/S1570 is 5/5, oil mainly solubilize in hydrophobic core, inducing decreases in the critical stress and platform modulus of the lamellar liquid crystal.

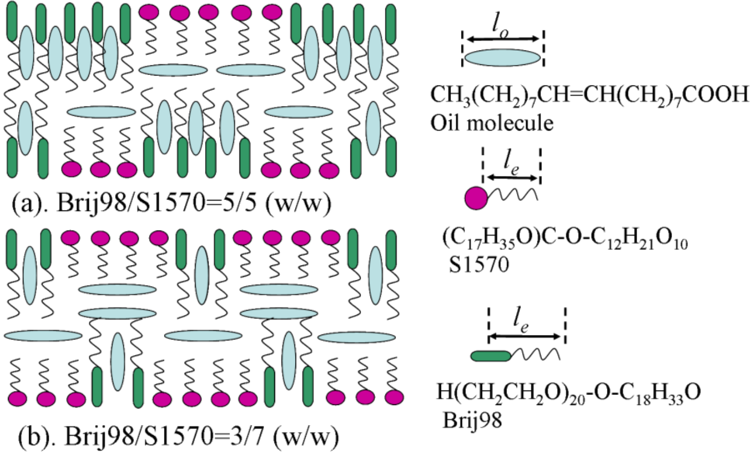

Figure 6 : Solubilization position of oil phase in different surfactant concentrations. (a). the system of S1570/Brij98/IPM/Water with the mass ratio of Brij98/S1570 is 5/5. (b). the system of S1570/Brij98/IPM/Water with the mass ratio of Brij98/S1570 is 3/7.

Conclusions

The main conclusions of above experments are that:

(i). The second surfactant polyoxyethylene 20 input the S1570/water systems decrease the lower limit temperature needed for the liquid crystal phase formation, and increase the liquid crystal phase area.

(ii). Isopropyl myristate into the Brij98/S1570/water systems, results in a phase transition from hexagonal liquid crystal to lamellar liquid crystal, which may reflecting an increase in the value of surfactant packing parameter ($v_L/a_S l_c$).

(iii). At the Brij98/S1570 mass ratio of 5/5 for Brij98/S1570/IPM/water system, shear stress and platform modulus of the lamellar liquid crystal get increased as the IPM concentration increases. Whereas at the Brij98/S1570 mass ration of 3/7, these values decrease with this change. This implies that the induced oil component solubilizes in different position of liquid crystals formed by Brij98/S1570 surfactant mixtures with different compositions.

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