

Study on phase behavior of compounded system of sodium lauryl glutamate and lauramide propyl hydroxysulfobetaine

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As a green and safe amino acid surfactant, sodium lauryl glutamate (SLG) has been favored by researchers. However, SLG has low solubility and its single system is not conducive to its application. Zwitterionic surfactants can increase its solubility by forming mixed micelles and possibly new phase states. In this paper, SLG was combined with lauramide propyl hydroxysulfobetaine (LHSB), and its different states were characterized. The type of phase states were determined and the pseudo-ternary phase diagrams were drawn. It was found that when the concentration of SLG was low and the proportion of SLG was not high, there were a large number of globular micelles in the solution, and rod-like micelles would appear as the concentration increased. As the concentration further increased, the SLG/LHSB system exhibited the characteristic of worm like micelles. With the further increase of concentration, the liquid crystal structures of hexagonal and layered phases were observed.

Keywords: Surfactant of amino acids; Wormlike micelles, Liquid crystal, Ternary phase diagram.

INTRODUCTION

As a green and safe new type of surfactant, amino acid surfactants have been favored by the majority of researchers and consumers, and their application in personal care products is becoming increasingly widespread. Amino acid surfactants are biomass-based surfactants, which are generally formed by the condensation of amino acids with long chain fatty acids, and their molecules have amino acid skeleton. In addition to having high surface activity^{1,2}, amino acid surfactant is also an environmentally friendly surfactant^{3,4}. It is safe and its degradation is rapid⁵. It can produce rich and delicate foam during cleaning process⁶, with low irritation to human organs⁷. At the same time, it also has certain bacteriostasis⁸ and hard water resistance⁹. Therefore, it is widely used in cosmetics and personal care products^{10–13}.

The application of amino acid surfactants in cosmetic products has the following limitations: (1) Amino acid surfactants are a mixture of acids and salts in weakly acidic environments. During storage, acids are prone to precipitate as crystals, which in turn affect the stability of the product system. (2) In most cases, the system viscosity of amino acid surfactants is low and it is difficult to regulate. Products with low viscosity often cannot meet the requirements of stability and performance.

Amino acid surfactants are anionic surfactants, which are combined with amphoteric surfactants to reduce the electrostatic repulsion between their hydrophilic groups and effectively regulate their micellar phase. At the same time, the synergistic effect of mixed micelles can effectively improve the solubilization of amino acids, solve the problems of stability and fluidity, and provide a theoretical basis for the design of actual formula systems.

Due to the low solubility of amino acid surfactants, precipitation may occur during use. So it is usually possible to choose some surfactants with good solubility, such as zwitterionic surfactants, to solubilize them by forming mixed micelles¹⁴. The phase behavior of surfactant systems is one of the most important research topics. The surfactant molecules in aqueous solution exhibit abundant self-assembly behavior with the change of concentration. By changing the content of different

components, the system will form different structures. At low concentrations, surfactant molecules tend to form spherical and rod-shaped micelles. As the concentration increases, worm like micelles gradually form, which can improve the viscoelasticity of the system to a certain extent. At higher concentrations, micelles continuously interweave to form dense spatial network structures, forming liquid crystal phases^{15,16}.

The structure of zwitterionic surfactants contains both anionic and cationic groups, exhibiting cationic properties in acidic solutions and anionic properties in alkaline solutions. Zwitterionic surfactants exhibit good surface activity over a wide pH range and are a class of high-performance surfactants¹⁷. The combination of zwitterionic surfactants and other types of surfactants exhibits good performance¹⁸.

In this paper, the phase behavior of the compound system of sodium lauroyl glutamate and zwitterionic surfactant was investigated by measuring the change of conductivity, viscosity, and polarization microscope observation, and pseudo-ternary phase diagram was drawn respectively. A new worm-like micelle phase, which has the characteristic of easy viscosity control, was found in the mixed sample formed with LHSB. The solubility and stability of SLG were improved by forming mixed micelles. The data obtained may be conducive to the application in the actual product development.

EXPERIMENTAL

Materials

Sodium lauryl glutamate (purity: > 95%) was purchased from Guangzhou Tinci Materials Technology Co., Ltd. Hydrochloric acid (content: 36–38%) was purchased from Sinopharm Chemical Reagent Co., Ltd. Sodium hydroxide (analytical grade) came from Shanghai Runjie Chemical Reagent Co., Ltd. Lauramide propyl hydroxysulfobetaine (content: 35%) came from Linyi Greensense Chemical Co., Ltd. The chemical structural formulas of SLG and LHSB are shown in Figure 1.

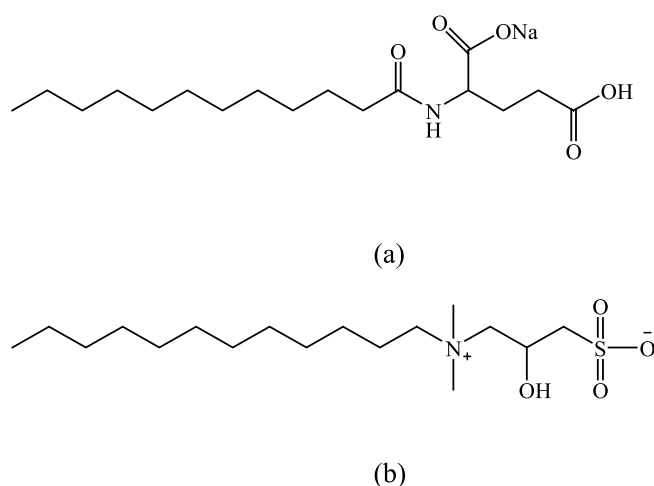


Figure 1. The chemical structural formulas of SLG (a) and LHSB (b)

Methods

Sample preparation

A certain amount of surfactants was weighed separately in a beaker using an electronic balance (PL602E, Mettler Toledo International Trade Co., Ltd). The surfactants were mixed well and then distilled water was added. The mixture was heated in a digital constant temperature water bath (BHS-2, Jiangyin Baoli Scientific Research Apparatuses Co., Ltd) until it was dissolved. The sample bottle was placed in the water-jacket constant temperature incubator (PCE-3000, Shanghai Yiheng Technology Co., LTD) at 25 °C for 24 h to obtain the samples required for the experiment.

Determination of electrical conductivity

A composite solution of 25 g SLG and amphoteric surfactant was prepared (mass ratio 1:9, 2:8, 3:7..., to 9:1). At 25 °C, the mass concentration of the composite solution was diluted by continuously adding distilled water. The conductivity of the solution at different concentrations was recorded in real time using a conductivity meter (DS-307A, Shanghai INESA Scientific Instrument Co., Ltd.). The tendency of conductivity change was observed to obtain the aggregation state of the surfactant solution.

Polarizing microscope observation

A small amount of sample was taken on a glass slide. A cover glass was covered promptly to prevent water evaporation. The microscope slide was placed on the heating table to ensure a constant temperature and was observed with a biological microscope (BX53, Olympus Corporation). Under polarized light source, the liquid crystal structure of high concentration surfactants can be observed, and the aggregation state of surfactants can be determined by its polarization characteristics.

Determination of rheological property

The rheological properties were measured using a rotary rheometer (AR-G2, Shanghai Benang Scientific Instrument Co., Ltd.). About 2 mL of sample was added, and the sample should not have bubbles. The cone-and-plate sensor Ti-60 (specification: Standard ETC steel, radius: 40 mm, cone Angle: 2 °C, Gap: 0.105 mm) was selected. During the testing process, the AWC100 cooling circulator (JULABO Technology (Beijing) Co., Ltd.) is used for temperature control in a circulating water bath. Each sample was balanced on the lower plate for 5 min before testing.

The drawing of pseudo ternary phase diagrams

Based on the changes in phase states under different conditions, the boundary points for the transitions between different phase states were identified. These points were drawn and connected. The regions of different phase states were marked, and finally, the complete pseudo ternary phase diagram of the system was drawn.

RESULTS AND DISCUSSION

The appearance of composite systems with different proportions

The mixtures of SLG and LHSB with different concentrations and ratios were prepared and placed in a 25 °C incubator for 24 h to observe the dissolution/precipitation. After being kept in 25 °C incubator for 24 h, the appearance of the composite solution of SLG and LHSB with different concentrations and proportions was shown in Table 1.

Amino acid surfactants are mixtures of acids and salts. Betaine and amino acid salts can form mixed micelles, which have a certain solubilizing effect on amino acids. The solubilization ability decreases with the decrease of betaine content. When the total surfactant concentration in the system increased, the amino acid content also increased, which enhanced the possibility of system precipitation.

As can be seen from Table 1, when the concentration of the system was 10% and the proportion of SLG was 0.8 or above, white solid began to precipitate in the system. The mixed systems under other ratios are all colorless and transparent solutions. When the concentration of the system was 20% and the proportion of SLG was 0.6, white solid precipitates appeared in the system. When the proportion of SLG in the system was less than 0.6, the mixed system was a colorless and transparent solution. When the concentration of the system was 30% and the proportion of SLG was 0.5, white solid precipitates appeared in the system. When the proportion of SLG in the system was less than 0.5, the mixed system was a colorless and transparent solution. When the concentration of the system was 40% and the proportion of

Table 1. The appearance of SLG/LHSB compound system with different concentrations and ratios

Concentration/Ratio	1:9	2:8	3:7	4:6	5:5	6:4	7:3	8:2	9:1
10%	D	D	D	D	D	D	D	P	P
20%	D	D	D	D	D	P	P	P	P
30%	D	D	D	D	P	P	P	P	P
40%	D	D	D	P	P	P	P	P	P

D: Dissolution, P: Precipitation

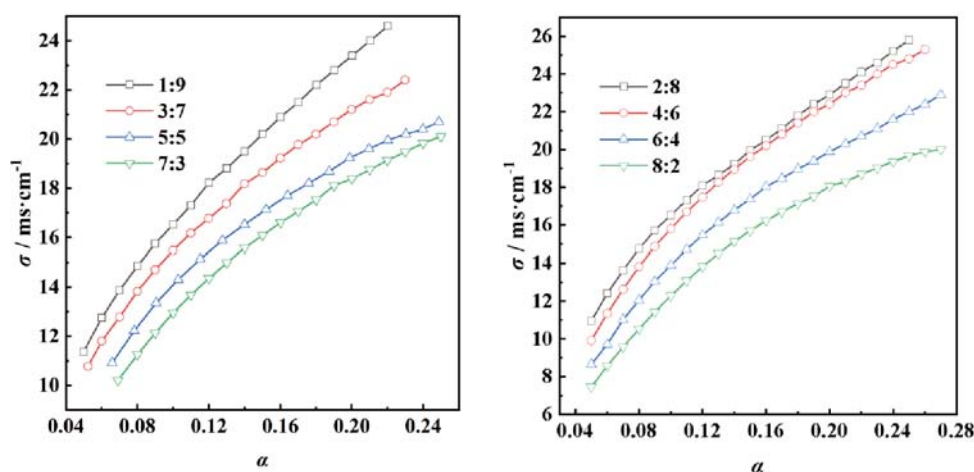


Figure 2. Variation of conductivity with concentration for different ratios of SLG/LHSB complex system

SLG was 0.4, white solid precipitates appeared in the system. When the proportion of SLG in the system was less than 0.4, the mixed system was a colorless and transparent solution.

Micelle morphology of composite systems with different proportions

The mixed solutions (SLG/LHSB) of sodium lauroyl glutamate and lauramide propyl hydroxysulfobetaine in different ratios were prepared. The mass concentration of these solutions was 30%. By continuously diluting with water, the conductivity of the solution at different concentrations was recorded in real-time using a DDS-307A conductivity meter. The curves of the conductivity of SLG/LHSB composite systems with concentrations for different ratios of SLG/LHSB are shown in Figure 2.

As can be seen from Figure 2, the complex systems of SLG and LHSB with different ratios show the same change trend. With the increase of system concentration, the conductivity of the system also gradually increases, and the increase rate gradually decreases, and the curve gradually becomes flat. At the same concentration, the higher the proportion of SLG in the mixed system, the lower the conductivity. This is due to the increasing aggregation number of small micelles in the system. Larger high-charge micelles can be further formed (there may be the transformation of spherical micelles into rod-like micelles). Therefore, the ion atmosphere retardation formed by counter-ions is greatly increased. After forming micelles, some small ions with opposite charges are attracted around the micelles, which cancel out the positive and negative charges and reduce the conductivity of the micelles. With the change of the concentration of surfactant solution, the aggregation number of micelles in the system is also constantly changing. When the concentration of the system reaches a certain value, the morphology of micelles will also change to a certain extent. The transformation of spherical micelles to rod-like micelles can be inferred from the rate of change in conductivity. By drawing auxiliary lines on the curves of the conductivity of SLG/LHSB composite systems, it

can be seen that the inflection point position where the upward trend of the curve decreases. Table 2 shows the concentration of conductivity inflection points for compound systems of SLG and LHSB with different ratios.

From Table 2, it can be seen that with the increasing proportion of SLG in the complex system, the inflection point concentration of the system conductivity shows an increasing trend. This inflection point can be used as the dividing point between spherical micelles and rod-like micelles, that is, the higher the proportion of SLG, the higher the concentration of the inflection point at which spherical micelles transform into rod-like micelles. By determining the concentration range at the inflection point, the concentration at which rod-shaped micelles appear in the mixed system can be preliminarily determined. It will pave the way for the subsequent drawing of ternary phase diagram of complex system.

According to the measured conductivity inflection point and Table 1, it can be seen that when the proportion of SLG in the system is greater than 0.1 but less than 0.8, the inflection point concentration of the system conductivity is all greater than 10%. When the proportion of SLG in the system is 0.1, the inflection point concentration of the system conductivity is less than 10%. Therefore, it can be inferred that when the concentration of the composite solution is 10% and the proportion of SLG in the system is 0.1, there are rod-like micelles in the mixed system. When the proportion of SLG in the system is less than 0.6, the inflection point concentration of the system conductivity is all less than 20%. Therefore, it can be inferred that when the concentration of the composite solution is 20% and the proportion of SLG in the system is less than 0.6, there are rod-like micelles in the mixed system. When the proportion of SLG in the system is less than 0.5, the inflection point concentration of the system conductivity is all less than 30%. Therefore, it can be inferred that when the concentration of the composite solution is 30% and the proportion of SLG in the system is less than 0.5, there are rod-like micelles in the mixed system. When the proportion of SLG in the system is less than 0.4, the inflection point

Table 2. Concentration of inflection points of conductivity for different ratios of SLG/LHSB compound system

SLG:LHSB	1:9	2:8	3:7	4:6	5:5	6:4	7:3	8:2	9:1
Concentration/%	9.8	10.1	10.8	11.1	11.2	13.7	14.5	16.2	16.3

concentration of the system conductivity is all less than 40%. Therefore, it can be inferred that when the concentration of the composite solution is 40% and the proportion of SLG in the system is less than 0.4, there are rod-shaped micelles in the mixed system.

Polarizing microscope observation of compound systems with different ratios and concentrations

When the concentration of SLG/LHSB composite system reaches 50%, the viscosity of the system significantly increases and the fluidity decreases, indicating the possibility of liquid crystal structure appearing in the system at this time. The liquid crystal phase of the SLG/LHSB composite system can be observed by placing different ratios and concentrations of the composite solution under a polarizing microscope. The polarizing microscope photos of the SLG/LHSB composite system under different concentrations and ratios are shown in Figure 3.

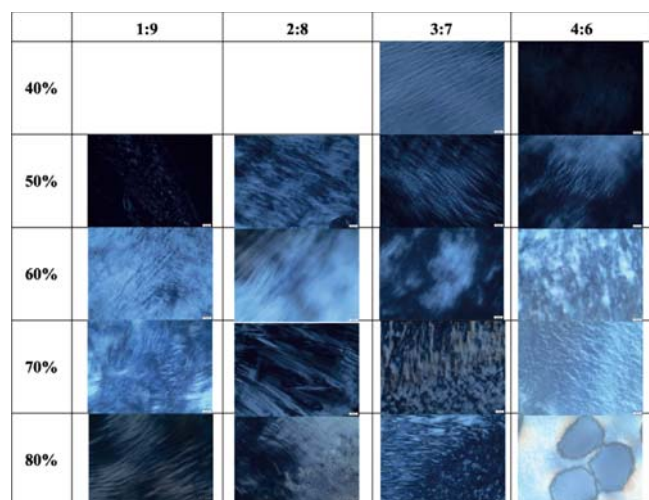


Figure 3. Polarizing microscope photos of the SLG/LHSB composite system under different concentrations and ratios

From Figure 3, it can be seen that when the concentration of the compound system is 40% and the SLG ratio exceeds 0.3, the characteristics of hexagonal phase can be observed under the microscope²⁰. With the continuous increase of system concentration, when the concentration of the compound system reaches 50% to 60%, the characteristics of the hexagonal phase become more obvious. When the system concentration is 70% and the proportion of SLG exceeds 0.2, in addition to the hexagonal filamentous structure observed under a polarizing microscope, crystallization is also observed, indicating that the surfactant has begun to precipitate at this time.

The change of viscosity with pH of compound systems with different proportions

With the wide application of surfactants in daily chemical products, the viscosity of surfactant system has become an important research direction. Appropriate viscosity can make the surfactant play a better role in the product. The viscosity of the system can be changed by changing the pH of the system. Therefore, the influence of pH change on the system viscosity of the SLG/LHSB complex solution with a concentration of 10%

and different proportions was explored. The results of viscosity changes with pH of SLG/LHSB complex solutions at 10% and different ratios are shown in Figure 4.

From Figure 4, it can be seen that the peak viscosity of compound systems with different ratios decreases with the increase of SLG ratio in the system, and the pH values corresponding to the peak viscosity of compound systems with different ratios also change. As the proportion of SLG in the system increases, the pH value corresponding to the peak viscosity of the system also increases.

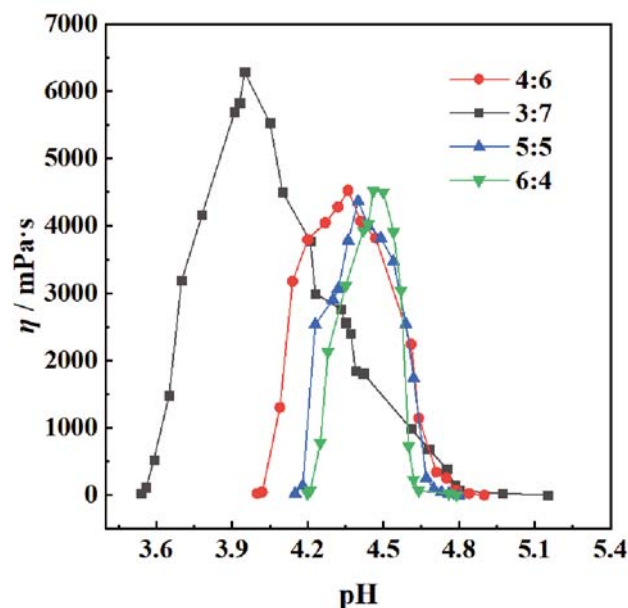


Figure 4. Variation of viscosity with pH of SLG/LHSB compound solution at 10% and different ratios

When the proportion of SLG in the system is 0.3 and the pH is about 3.91, the viscosity of the system reaches its peak. However, when the proportion of SLG in the system is 0.6, the peak viscosity of the system appears around pH of 4.50. When pH is in the range of 3.40 to 4.85, the viscosity of the system has an obvious trend of first increasing and then decreasing. When the pH of the system is less than 3.40 or greater than 4.85, the viscosity of the system is very low. Only when the pH is within the range of 3.40 to 4.85 does there be a significant change in viscosity. Perhaps due to the increase in pH, the number of positive charges at the polarity end of the two surfactants decreased and the number of negative charges increased. It leads to an increasing electrostatic effect when they form micelles in the compound system. When pH reaches a certain value, the electrostatic effect reaches the maximum, and the surfactant molecules are more likely to flocculate, thus the viscosity of the system reaches the maximum.

Rheological performance test

When the concentration of SLG/LHSB composite system exceeds 30% and the proportion of SLG exceeds 0.2, the composite system exhibits a significant viscous state and exhibits a “filamentous shape” as shown in Figure 5. In the complex system in the state shown in Figure 4, no liquid crystal phase can be observed under polarized light sources.



Figure 5. Photo of SLG/LHSB composite system

The rheological properties of the composite system with SLG:LHSB=4:6 and a concentration of 30% were tested, and the results are shown in Figure 6.

According to Figure 6a, the viscosity of the SLG/LHSB composite system can reach up to 10^4 mPa·s or above. When the shear rate is lower than 1 s^{-1} , the viscosity of the SLG/LHSB composite system remains almost unchanged with the change of shear rate. When the shear rate is higher than 1 s^{-1} , the viscosity of the SLG/LHSB composite system decreases sharply, and the lowest viscosity can be reduced to $10 \text{ mPa} \cdot \text{s}$. This phenomenon of shear thinning indicates that the system is a pseudoplastic fluid, which is also a characteristic of worm-like micelles¹⁹. The system was tested by dynamic rheology, and the results are shown in Figure 6b. The elastic modulus (G') and viscous modulus (G'') of the sample have an intersection with the shear frequency. The shear frequency corresponding to this intersection point is ω_c . When $\omega < \omega_c$, $G'' > G'$, SLG/LHSB solution exhibits viscous properties. When $\omega > \omega_c$, $G' > G''$, SLG/LHSB solution exhibits elastic properties. Cole-Cole

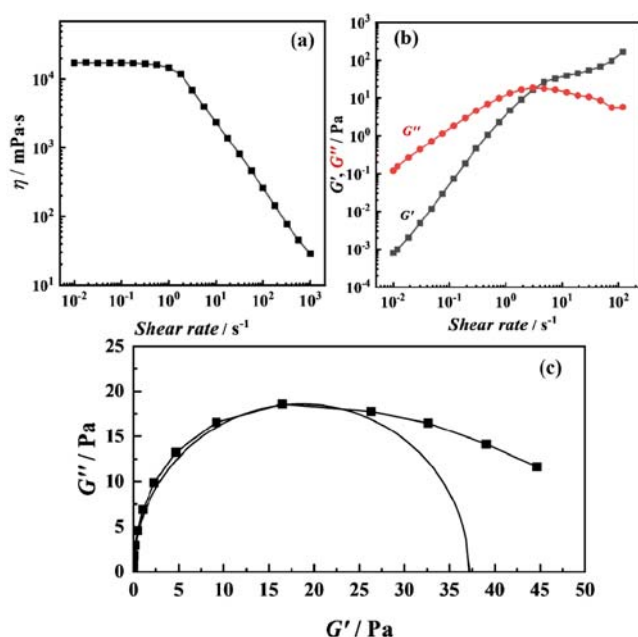


Figure 6. Rheological property test of SLG/LHSB composite system (a: viscosity changes with shear rate; b: elastic modulus and viscosity modulus changes with shear rate; c: Cole-Cole diagram)

diagram was made with its elastic modulus and viscous modulus as horizontal and vertical coordinates respectively (Figure 6c). The curve exhibits a semicircular characteristic, which also indicates the formation of worm-like micelles in the system²¹.

Pseudo ternary phase diagram of SLG/LHSB/H₂O complex system

Based on the measured data and images, the ternary phase diagram of the SLG/LHSB complex system can be initially drawn, and the resulting pseudo-ternary phase diagram of the SLG/LHSB/H₂O complex system was shown in Figure 7.

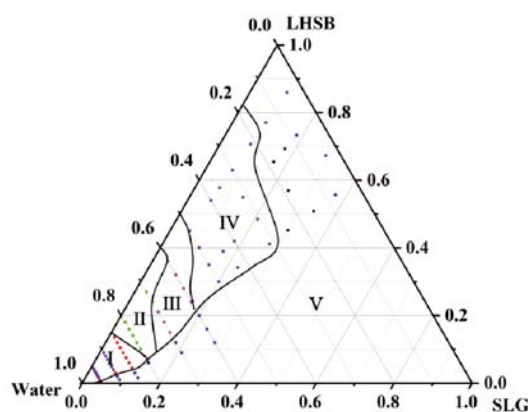


Figure 7. Pseudo-ternary phase diagram of SLG/LHSB/H₂O complex system (I: homogeneous solution, II: rod-like micelles, III: worm-like micelles, IV: hexagonal phase, V: undissolved surfactants)

From Figure 7, it can be seen that when the concentration of the system is lower than 5% and the proportion of SLG is lower than 0.8, the system will present a homogeneous solution (I), otherwise there will be solid surfactant precipitation from the system (V). As the concentration increases, there will be a transition towards rod-like micelles (II) and worm-like micelles (III). When the concentration of the system exceeds 50% and the proportion of SLG does not exceed 0.4, the system transforms into hexagonal phase (IV). When the concentration of the system exceeds 70% and the proportion of SLG exceeds 0.3, crystallization (V) is observed under a polarizing microscope. When the concentration of the system exceeds 90% and SLG exceeds 0.1, the system is in a complete precipitation state (V).

CONCLUSIONS

In this paper, sodium lauryl glutamate was mixed with zwitterionic surfactant. The phases of the complex system were characterized by different methods, and the pseudo-ternary phase diagram of the complex system was drawn. The inflection point of the transformation from spherical micelle to rod-like micelle can be determined by the change in the conductivity of the composite system with different ratios and concentrations. The characterization of the phase state at higher concentrations was obtained through observation under a polarizing microscope. By observing the complex systems with

different concentrations and ratios under a polarizing light source, combined with macroscopic performance, the type of liquid crystal in the system can be determined. Compared with the phase behavior of single SLG, a new phase, worm-like micelle phase, appeared in the mixed sample formed with LHSB. This phase has the characteristic of easy viscosity control. It is conducive to the application in the actual product development.

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