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RESEARCH ARTICLE

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Studies on CMC and thermodynamic function of anionic surfactants in DMA/long-chain alcohol systems using microcalorimetric method

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Abstract The critical micelle concentration (CMC) and the thermodynamic function of the anionic surfactant, sodium laurate (SLA) and sodium dodecyl sulfate (SDS) in the *N,N*-dimethyl acetamide (DMA)/long-chain alcohol systems were studied using titration microcalorimetric method. The power-time curves of SLA and SDS in the presence of a long-chain alcohol (*n*-heptanol, *n*-octanol, *n*-nonanol, *n*-decanol) in the DMA medium were determined. Then, from the curves, the critical micelle concentration (CMC) and the thermodynamic standard formation functions (ΔH^{θ}_{m}, ΔG^{θ}_{m} and ΔS^{θ}_{m}) were obtained through thermodynamic theories. The relationships between temperature, alcohol's carbon number, concentration and thermodynamic properties were discussed. For SLA or SDS in a DMA solution, under the same concentration of alcohol, the values of CMC, ΔH^{θ}_{m} and ΔS^{θ}_{m} increase, while the values of ΔG^{θ}_{m} decrease with the increase of temperature. Under the same condition of identical temperature and alcohol concentration, the values of CMC, ΔH^{θ}_{m}, ΔG^{θ}_{m} and ΔS^{θ}_{m} decrease with the increase of the alcohol's carbon number. In the presence of the same kind of alcohol, the values of CMC and ΔG^{θ}_{m} increase, but the values of ΔH^{θ}_{m} and ΔS^{θ}_{m} decrease with the concentration increases in alcohol series at the same temperature.

Keywords microcalorimetric method, *N*, *N*-dimethyl acetamide, sodium laurate, sodium dodecyl sulphate, long-chain alcohol, critical micelle concentrations (CMC), thermodynamic function

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In comparison to the amount of work done on the properties of surfactant in water [1−4], the study of the physical-chemical properties of surfactant in non-aqueous solution is rather limited[5,6]. Recently, the practical applications of micelles and microemulsions in non-aqueous solutions has been increased considerably, especially, in the dispersal of the pesticide microemulsions. There are many pesticides which do not dissolve in xylene or in water. These are used as dusting or wettable powders. Meanwhile, the environment is polluted. Effective pesticides such as avermectins, acetamiprid, emamectin benzoate and thiamethoxam etc. can only be dissolved in a polar organic solvent. In order to further the research on microemulsions, the relationship between surfactant and polar solvent is investigated.

The non-aqueous solution system of a surfactant is complicated. The surfactants have many types, such as ionic, non-ionic and amphoteric surfactant. Non-aqueous solvents also have many types, such as solvents with a strong polarity which easily form hydrogen bonds, solvents with strong polarity but does not easily form hydrogen bonds, aromatic series solvents with low polarity and fatty solvents without polarity. To research the properties of surfactants in a water solution system, routine methods for measuring density, conductance, viscosity and surface tension can be used. As the micelle accumulation in surfactants in a non-aqueous solution is small (generally not more than 10 [7]), routine method can not determine the CMC. But heat effects can have visible changes around the CMC. Therefore, this heat change can be measured using microcalorimetry to determine CMC and the standard heat formation of micelles and to calculate ΔG^{θ}_{m} and ΔS^{θ}_{m} according to the thermodynamic theory.

1 Experimental

1.1 Instrument

The 2277 Thermal Activity Monitor (Sweden) is used in this experiment. This instrument can be used in the

thermostat range of 10−90°C. It is maintained at a given temperature constant within $\pm 2 \times 10^{-4}$°C. This system is very sensitive. The detection limit is 0.15 μW and the baseline stability (within a period of 24 h) is 0.2 μW. It holds up to four independent calorimetric units. A titration unit is inserted in the thermostats. A titration ampoule unit is equipped with a 4 mL stainless steel titration ampoule, an injector and a stirrer. We put one solution in an ampoule and the other solution in the injector. After preheating, we dropped the solution in the injector into the ampoule with a certain velocity and stirred. The instrument records the heat produced in this process so that the power-time curves can be obtained.

1.2 Materials

Surfactant: Sodium laurate, $CH_3(CH_2)_{10}COONa$ (SLA), analytical grade (Shanghai Chemical Reagents Plant); Sodium dodecyl sulfate, $CH_3(CH_2)_{11}SO_4Na$ (SDS), analytical grade (Shanghai Chemical Reagents Plant); Non-aqueous solvent: *N*, *N*-dimethyl acetamide (DMA), analytical grade (Tianjin Kermel Chemical Reagents Development Center); Solution: a certain amount of SLA or SDS and alcohol with DMA is dissolved, then, a fix volume is measured using a volumetric flask to prepare a titrating solution which contains 0.01 mol·L^{-1} SLA or SDS and alcohol (*n*-heptanol, *n*-octanol, *n*-nonanol, *n*-decanol) with various concentrations (0.1, 0.5, 1.0, 1.5 mol·L^{-1}).

1.3 Method

In this experiment, a microcalorimetric unit of 4 mL stainless steel titration ampoule was used. 2 mL DMA was poured into the ampoule. A titrating tube was used to titrate 0.5 mL titrate solution into an organic solvent in ampoule and preheated for 1 h. After the temperature stayed constant, the stirrer was turned on with the desired speed of 120 r·min^{-1}. The titrating solution was dropped into the ampoule at the rate of 0.02 mL·min^{-1} and we recorded the power-time curves. The experiment could be considered as finished when the curve returns to the baseline.

2 Results and discussion

2.1 Type of micelles

In order to determine the two surfactants' micelle type in a polar solvent (DMA), the conductance of the systems were measured. The data are summarized in Table 1.

Generally, the effect of a solvent on a surfactant can be divided into three types [8]: (1) the hydrophobic effect of surfactant is available, that is O/W type; (2) the surfactant forms reverse micelle, that is W/O type; (3) the surfactant can not form micelle.

Table 1 Specific conductance of SLA and SDS in DMA in the presence of 1.0 mol·L^{-1} octanol and in water at 303 K

Surfactant	κ/μS·cm^{-1}	
	H_2O	DMA/ octanol (1.0 mol·L^{-1})
0	2.06	0.88
0.01 mol·L^{-1} SLA	645	54.8
0.01 mol·L^{-1} SDS	669	239.0

If it belongs to (1), a hydrogen bond is formed between surfactant and solvent molecule like a water solution. As can be seen from Table 1, the surfactant (SDS or SLA) forms a micelle in DMA and the conductance data is similar to that in water. Hydrogen bonds are formed. Therefore these micelles in DMA solution belong to the O/W type. The conductance order of the two kinds of micelles in water is similar to that of other surfactants in water [9,10]. The conductance of micelles in polar solvent has not been reported.

2.2 Determination of CMC

The power-time curves of the micelle formation process were determined for anionic surfactants (0.01 mol·L^{-1} SLA and 0.01 mol·L^{-1} SDS) in DMA which contained various concentrations (0.1, 0.5, 1.0 and 1.5 mol·L^{-1}) of alcohols (*n*-heptanol, *n*-octanol, *n*-nonanol, *n*-decanol) at different temperatures. Every system determination was repeated thrice and the average values of the experimental curves were obtained. Partial curves are shown in Figs. 1 and 2.

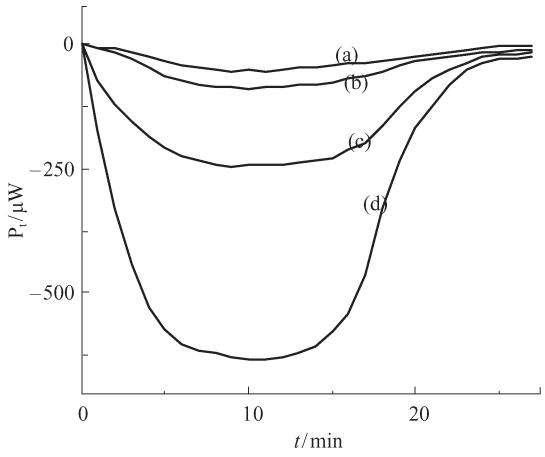

Fig. 1 The power-time curves of the 0.01 mol·L^{-1} SLA in DMA solution containing *n*-nonanol of different concentration at 308 K
(a) 0.1; (b) 0.5; (c) 1.0; (d) 1.5 mol·L^{-1}

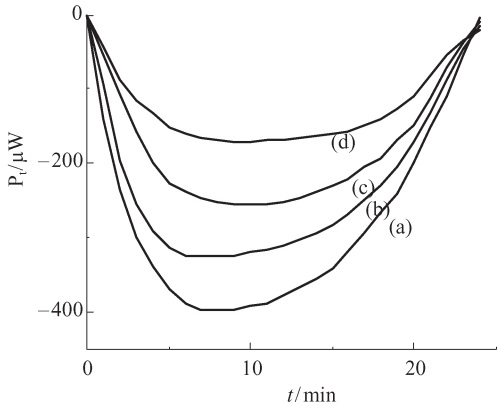

Fig. 2 The power-time curves of the 0.01 mol·L^{-1} SDS in DMA solution containing 1.00 mol·L^{-1} *n*-nonanol at different temperatures: (a) 298 K; (b) 303 K; (c) 308 K and (d) 313 K

The CMC can be obtained according to the corresponding concentration of the lowest point of the power-time curves [11]; The data are listed in Tables 2 and 3 respectively.

Plots of CMC-*T*, CMC-*n* (carbon number of alcohol), CMC-*c* curves for SLA and SDS are shown in Fig. 3.

(A) The CMC-*T* curves of 0.01 mol·L^{-1} SLA in DMA system in the presence of 1.00 mol·L^{-1} alcohols (a) *n*-heptanol; (b) *n*-octanol; (c) *n*-nonanol; (d) *n*-decanol; (B) The CMC-*n* curves of 0.01 mol·L^{-1} SLA in DMA system in the presence of 1.00 mol·L^{-1} alcohols (a) 298 K; (b) 303 K; (c) 308 K; (d) 313 K; (C) The CMC-*c* curves of 0.01 mol·L^{-1} SDS in DMA system in the presence of alcohols of different concentration at 308 K (a) *n*-heptanol; (b) *n*-octanol; (c) *n*-nonanol; (d) *n*-decanol; (D) The CMC-*n* curves of 0.01 mol·L^{-1} SDS in DMA system in the presence of alcohols of different concentration at 308 K (a) 1.5; (b) 1.0; (c) 0.5; (d) 0.1 mol·L^{-1}

It can be seen in Fig. 3 that the values of CMC increase with the increase in temperature under similar conditions of alcohol concentration and the same kind of alcohol. The values decrease with the increase of the alcohol's carbon number under similar conditions of identical temperature and alcohol concentration. The values increase with the increase in alcohol concentration with the same kind of alcohol and at the same temperature.

The next equation [12] can explain the above-mentioned constant.

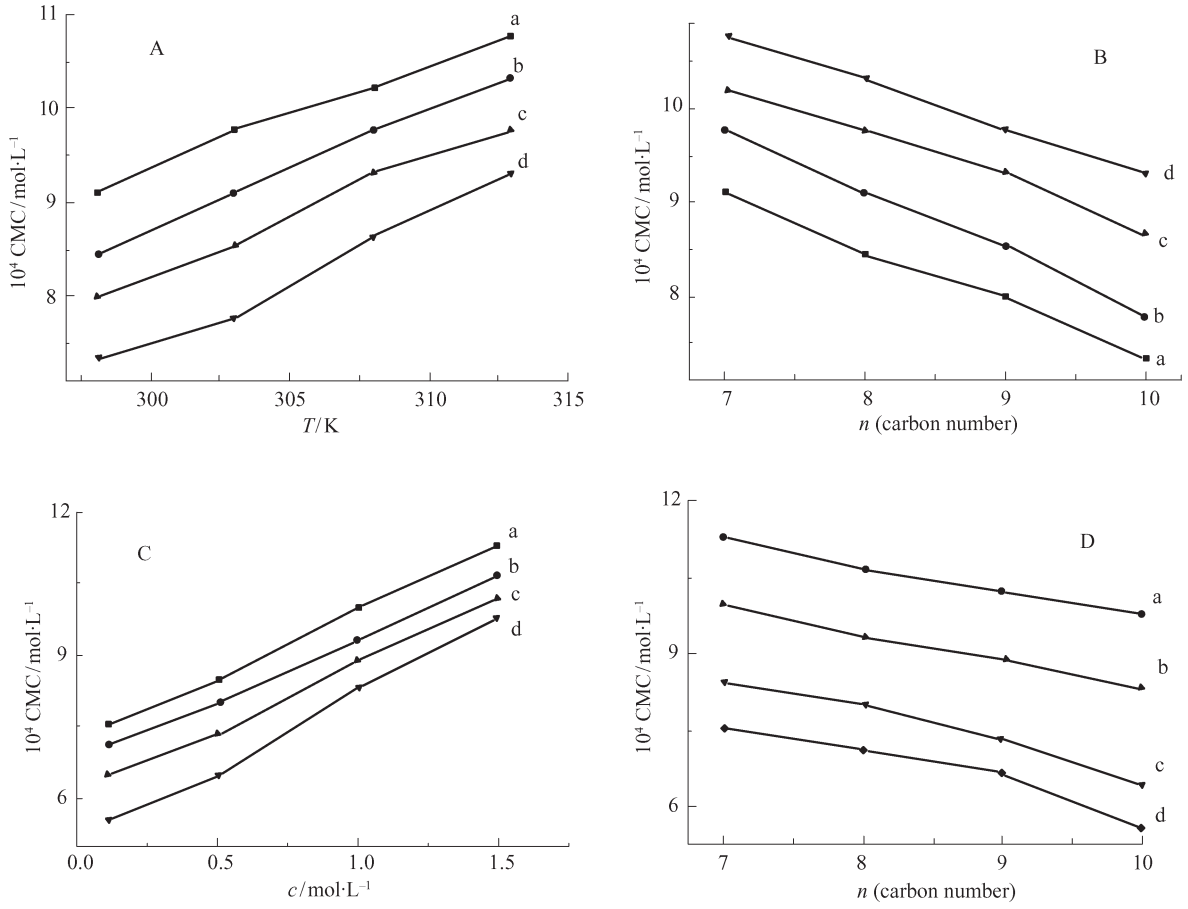

Fig. 3 The CMC-*T*, CMC-*n* and CMC-*c* curves of 0.01 mol·L^{-1} SLA or SDS in DMA in the presence of different alcohols concentrations or at different temperatures

Table 2 The CMC (10^4 mol·L^{-1}) and the thermodynamic functions (ΔG^{θ}_m, ΔH^{θ}_m/kJ·mol^{-1}; ΔS^{θ}_m/J·K^{-1}·mol^{-1}) of 0.01 mol·L^{-1} SLA and 0.01 mol·L^{-1} SDS in DMA system in the presence of 1.00 mol·L^{-1} alcohols at different temperature

surfactant	temperature	*n*-heptanol				*n*-octanol			
	T/K	CMC	ΔG^{θ}_m	ΔH^{θ}_m	ΔS^{θ}_m	CMC	ΔG^{θ}_m	ΔH^{θ}_m	ΔS^{θ}_m
SLA	298	9.10	−23.25	−19.96	−11.03	8.44	−23.43	−39.21	−52.95
	303	9.77	−23.46	−16.21	23.92	9.10	−23.64	−23.57	0.218
	308	10.21	−23.73	−14.10	31.27	9.77	−23.84	−18.78	16.44
	313	10.77	−23.98	−10.24	43.89	10.32	−24.09	−10.82	29.61
SDS	298	8.44	−23.43	−34.15	−35.97	7.99	−23.57	−42.39	−63.16
	303	9.32	−23.58	−27.66	−13.48	8.88	−23.70	−30.57	−22.68
	308	9.99	−23.79	−16.21	24.60	9.32	−23.97	−20.65	10.76
	313	10.66	−24.00	−12.18	37.78	10.11	−24.14	−15.07	28.98
SLA	298	7.99	−23.57	−90.96	−226.2	7.33	−23.78	−160.6	−459.3
	303	8.54	−23.76	−51.47	−69.73	7.77	−24.03	−84.50	−171.7
	308	9.32	−23.97	−36.93	−53.23	8.64	−24.09	−56.34	−104.7
	313	9.77	−24.23	−23.50	2.335	9.32	−24.35	−39.56	−48.58
SDS	298	7.10	−23.86	−92.38	−229.9	6.66	24.02	−158.4	−451.0
	303	7.77	−24.03	−51.83	−91.74	6.88	−24.34	−96.37	−237.7
	308	8.88	−24.09	−40.89	−54.55	8.44	−24.22	−72.03	−155.2
	313	9.55	−24.29	−38.10	−44.12	8.98	−24.45	−43.65	−61.34

Table 3 The CMC (10^4 mol·L^{-1}) and the thermodynamic functions (ΔG^{θ}_m, ΔH^{θ}_m/kJ·mol^{-1}; ΔS^{θ}_m/J·K^{-1}·mol^{-1}) of 0.01 mol·L^{-1} SLA and 0.01 mol·L^{-1} SDS in DMA system in the presence of different concentration's alcohol at 308 K

Surfactant	concentration of alcohol	*n*-heptanol				*n*-octanol			
	(mol·L^{-1})	CMC	ΔG^{θ}_m	ΔH^{θ}_m	ΔS^{θ}_m	CMC	ΔG^{θ}_m	ΔH^{θ}_m	ΔS^{θ}_m
SLA	0.1	7.10	−24.66	−2.45	72.11	6.66	−24.83	−3.26	70.02
	0.5	8.88	−24.09	−4.23	64.48	8.44	−24.22	−6.56	57.33
	1.0	10.21	−23.73	−14.10	31.27	9.77	−23.84	−18.78	16.44
	1.5	11.32	−23.47	−29.26	−18.82	10.88	−23.57	−40.46	−54.84
SDS	0.1	7.55	−24.50	−0.86	76.76	7.10	−24.66	−1.24	76.04
	0.5	8.44	−24.22	−4.70	63.35	7.99	−24.36	−9.12	49.48
	1.0	9.99	−23.79	−16.21	24.60	9.32	−23.97	−20.65	10.76
	1.5	11.32	−23.47	−36.16	−41.21	10.66	−23.62	−47.65	−47.46
SLA	0.1	6.22	−25.00	−7.40	57.14	5.55	−25.29	−8.21	55.46
	0.5	7.99	−24.36	−16.96	24.02	7..33	−24.58	−26.97	−7.760
	1.0	9.32	−23.97	−40.36	−53.23	8.88	−24.09	−56.34	−104.7
	1.5	10.43	−23.68	−90.64	−217.4	9.99	−23.79	−136.97	−367.5
SDS	0.1	6.46	−24.83	−3.18	70.28	5.55	−25.29	−4.88	66.27
	0.5	7.33	−24.59	−14.59	32.44	6.44	−24.91	−18.17	21.89
	1.0	8.88	−24.09	−40.89	−54.55	8.30	−24.22	−72.03	−155.2
	1.5	10.21	−23.73	−90.57	−247.6	9.77	−23.84	−135.5	−362.4

$$\ln\mathrm{CMC} = \ln\frac{1000}{N_0 V} - 1 - \frac{n_i w}{RT} - \frac{E_{el}}{RT} = A_1 - \frac{n_i w}{RT} + \frac{e\Psi}{RT} \quad (1)$$

Where N_0 is the Avogadro constant, V is volume of one molecule, is carbon number of a straight-chain alkyl hydrocarbon,is the cohesion energy of one $-CH_2-$, E_{el} is the electric energy of a one-price ion, e is the charge constant, Ψ is the ion electrical force and $e\Psi$ is the energy when a one-price surfactant ion moves from solution to micelle state.

Equation (1) suggests that the effect of temperature on the CMC has two opposite facts: On the one hand, the increase of the temperature will weaken the hydration for hydrophilic group of surfactant. Then, the micelle formed easily and as a result, the CMC decreases. On the other hand, the increase in the temperature will cause the breakage of the iceberg's structure around it and the formation of the micelle will be difficult and thus, the CMC will increase. According to the experimental result, the influence of temperature on the latter is greater than on the former, so the CMC values increase with the increase in temperature. The CMC values decrease while the hydrophobic action of the system increases ($n_i w$

values increase) with the increase of the carbon number of the long-chain alcohol. For this system, when the temperature and long-chain alcohol are fixed, the reasons that the CMC values increase with the increase in the concentration of alcohol may be as follows: First, because the polarity of the DMA is greater than the long-chain alcohol, the polarity of the system decreases with the increase in the concentration of alcohol. The electrical repellent force of the ion group strengthens and thus, the formation of the micelle is difficult and results in the increase in CMC values. Next, as shown in the study of the solubilization site of benzoic alcohol in SDC micelle solution [13], benzoic alcohol solubilizes in the palisade layer of the micelle between the hydrophilic groups and the inner core of the micelle. It distributes mainly around the site where α $-CH_2-$ joins five methylene chain of SDC hydrocarbon chain of the surfactant. When the mole fraction concentration of benzoic alcohol is 0.77, it may enter the inner core of micelle. Therefore, because of the solubilization of alcohol, the micelle dimension increases with the increase of concentration of alcohol. Then, a CMC value increase is the inevitable outcome.

2.3 Calculation of the ΔH^{θ}_{m}, ΔG^{θ}_{m} and ΔS^{θ}_{m}

The power-time curves of the micelle formation process were recorded for anionic surfactants (0.01 mol·L^{-1} SLA and 0.01 mol·L^{-1} SDS) in DMA, which contained various concentrations (0.1, 0.5, 1.0 and 1.5 mol·L^{-1}) of alcohols (*n*-heptanol, *n*-octanol, *n*-nonanol, *n*-decanol) at different temperatures. Based on the area of the power-time curves, the heat effect could be obtained. Every system was determined thrice and the average values of the experimental curves were obtained. From the heat effect and CMC, ΔH^{θ}_{m} was calculated. According to thermodynamic theory, from Eq. (2) [14]:

$$\Delta G^{\theta}_{m} = RT \ln X_{CMC} \quad (2)$$

ΔG^{θ}_{m} can be calculated.

$$\Delta G^{\theta}_{m} = \Delta H^{\theta}_{m} - T\Delta S^{\theta}_{m} \quad (3)$$

ΔS^{θ}_{m} can be calculated from Eq. (3). The data are given in Table 2, 3.

2.4 The regularity of thermodynamic standard formation function of micelle

A micelle's thermodynamic formation function is mainly determined by the hydrophobic action of surfactant in DMA. When temperature increases, an increase in the molecule's movement can decrease the hydrophobic action. Then, the absolute value of ΔH^{θ}_{m} decreases. This is comparable with Table 2. Under similar conditions in temperature, an increase in the concentration or the carbon number of an alcohol can cause an increase in hydrophobic action because the hydrophobic action of a long-chain alcohol is larger than that of DMA. With the increase of the concentration or the carbon number of alcohol, the absolute value of ΔH^{θ}_{m} of these systems also increases. This is a comparable with Table 3.

For SLA and SDS systems, plots of CMC-*T*, CMC-*n* and CMC-*c* curves are shown in Fig. 4−7, respectively.

It can be seen in Figs. 4−7, ΔH^{θ}_{m} increases with the increase in the temperature under the same alcohol concentration and same kind of alcohol. It decreases with the increase of the alcohol's carbon number under identical temperatures and alcohol concentration. It decreases with the increase in alcohol concentration in the same kind of alcohol and the same temperature.

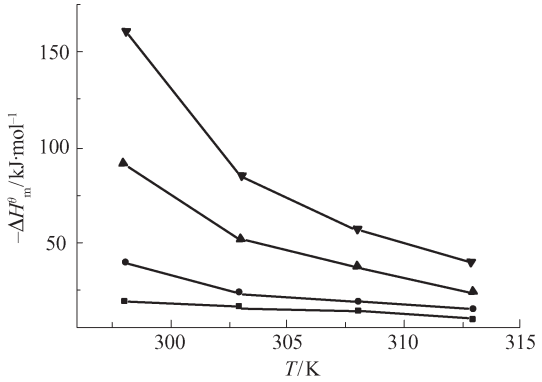

Fig. 4 The ΔH^{θ}_{m}-T curves of 0.01 mol·L^{-1} SLA in DMA system in the presence of 1.00 mol·L^{-1} alcohols at different temperatures

■ *n*-heptanol; ● *n*-octanol; ▲ *n*-nonanol; ▼ *n*-decanol

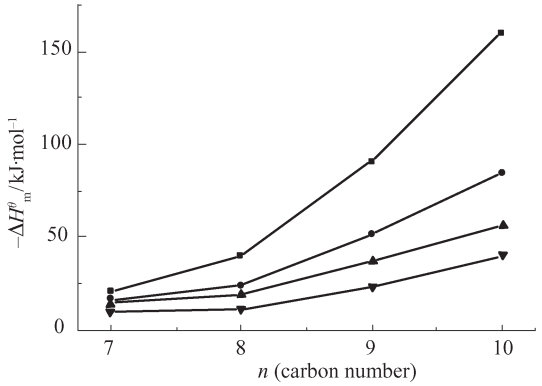

Fig. 5 The ΔH^{θ}_{m}-n curves of 0.01 mol·L^{-1} SLA in DMA system in the presence of 1.00 mol·L^{-1} alcohols at different temperatures

■ 298 K; ● 303 K; ▲ 308 K; ▼ 313 K

From Tables 2 and 3, ΔG^{θ}_{m} decreases with the increase in temperature under the same concentration and the same kind of alcohol. It decreases with the increase of the

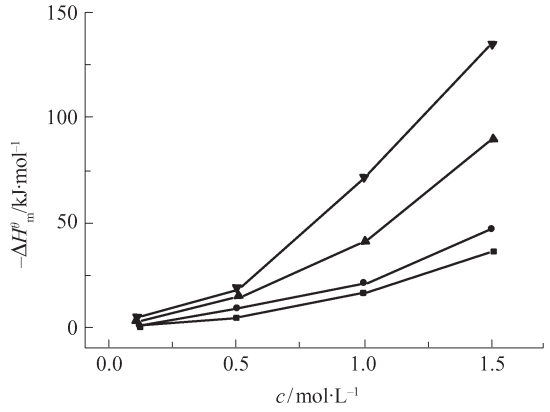

Fig. 6 The ΔH^{θ}_{m}-c curves of 0.01 mol·L^{-1} SDS in DMA system in the presence of different concentration's alcohol at 308 K

■ *n*-heptanol; ● *n*-octanol; ▲ *n*-nonanol; ▼ *n*-decanol

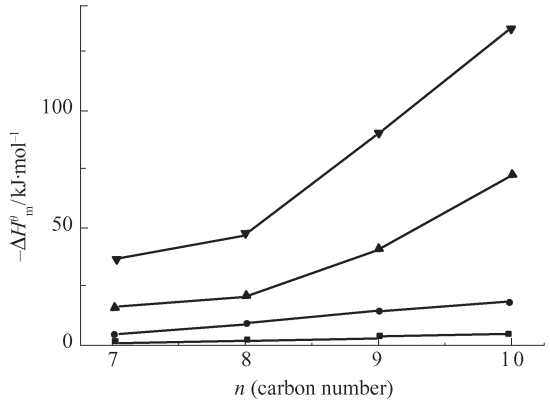

Fig. 7 The ΔH^{θ}_{m}-n curves of 0.01 mol·L^{-1} SDS in DMA system in the presence of different alcohol concentrations at 308 K

■ 0.1; ● 0.5; ▲ 1.0; ▼ 1.5 mol·L^{-1}

alcohol's carbon number under identical temperatures and alcohol concentration. It increases with the increase of alcohol concentration in the same kind of alcohol and the same temperature; ΔS^{θ}_{m} increases with the increase in temperature under the same alcohol concentration and the same kind of alcohol. It decreases with the increase of the alcohol's carbon number under identical temperatures and alcohol concentration. It decreases with the increase of alcohol concentration in the same kind of alcohol and the same temperature.

The effect of temperature, the kind of alcohol and the concentration of alcohol on ΔG^{θ}_{m} and ΔS^{θ}_{m} can be explained from the influence on CMC. This will not be repeated anymore.

According to the data, it can also be discussed as follows:

(1) For DMA/*n*-octanol system, is the micelle formation process driven by entropy or enthalpy? When the ionic surfactant is in a water solution under standard conditions, ΔG^{θ}_{m} is around -26 kJ·mol^{-1}, ΔH^{θ}_{m} is about 2.0$-$6.0 kJ·mol^{-1} and $T\Delta S^{\theta}_{m}$ is about 29$-$49 kJ·mol^{-1} [15]. Therefore, the micellization process is governed by entropy. For the DMA/long-chain alcohol system, ΔG^{θ}_{m} is around -49 kJ·mol^{-1}, ΔH^{θ}_{m} is about $-79-300$ kJ·mol^{-1} and $T\Delta S^{\theta}_{m}$ is about 4.8$-$7.9 kJ·mol^{-1}. It shows that enthalpy is the driving force in the micellization process for DMA/long-chain alcohol system.

(2) Calculation of the contribution of methylene (CH_2) in the micellization process. Micellization undergoes electrical repulsive forces of the ionic head and the hydrocarbon's hydrophobic interaction of surfactant, thus $\Delta G^{\theta}_{m} = F_{he} + F_{el}$. In this formula, the former is hydrophobic interaction energy, the latter is electric repulsive energy. Generally, the occupancy of the latter is only 3%$-$5%, which indicates that hydrophobic interaction is major factor, $\Delta G^{\theta}_{m}(CH_2)$ is 2.72 kJ·mol^{-1}[16]. For the system of 1 mol·L^{-1} *n*-octanol in DMA solvent, $\Delta G^{\theta}_{m}(CH_2)$ is about 4.1 kJ·mol^{-1}. It shows that in an organic solvent whose polarity is smaller than that of water, although the electrical repulsive force increases, the hydrophobic interaction of methylene (CH_2) increases more greatly, Therefore, the micellization process is driven by enthalpy.

3 Conclusions

(1) These power-time curves of anionic surfactants (SLA and SDS) in the DMA/long-chain alcohol system are studied using titration microcalorimetry. From the curves, CMC and ΔH^{θ}_{m} are obtained. According to thermodynamic theory, ΔG^{θ}_{m} and ΔS^{θ}_{m} can be calculated. It is a new method to obtain CMC and thermodynamic function.

(2) For the system in the presence of alcohol of identical concentration, values of CMC, ΔH^{θ}_{m} and ΔS^{θ}_{m} increase while the ΔG^{θ}_{m} decrease with the increase of the temperature.

(3) For the system with identical temperature and concentration of alcohol, values of CMC, ΔH^{θ}_{m}, ΔG^{θ}_{m} and ΔS^{θ}_{m} decrease with the increase in the carbon number of alcohol.

(4) For the system with identical temperature and alcohol, the CMC and ΔG^{θ}_{m} increase, while the ΔH^{θ}_{m} and ΔS^{θ}_{m} decrease with the increase in the concentration of alcohol.

(5) The micelle formed by the two kinds of anionic surfactants (SLA and SDS) in mixed alcohol and DMA solvent belong to the O/W type and the system is driven by enthalpy.

In a word, the power-time curves of micelle formation process in non-aqueous solution which measured by titration microcalorimetry contains a lot of information. It is this information from which the CMC and thermodynamic function (ΔH^{θ}_{m}, ΔG^{θ}_{m} and ΔS^{θ}_{m}) can be obtained. These parameters are significant for theory

and practical application study of a surfactant's properties in a non-aqueous solution.

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