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Microcalorimetric Study on the Critical Micelle Concentration and Thermodynamic Functions of Di(2-ethyl-hexyl) Phosphate Salts in Organic Solvent + *sec*-Octyl Alcohol Systems at 298.15 K

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The power−time curves of the micelle formation process were determined for three kinds of quasi-surfactants [lithium di(2-ethyl-hexyl) phosphate, sodium di(2-ethyl-hexyl) phosphate, and potassium di(2-ethyl-hexyl) phosphate] and five kinds of alkanes (octane, decane, dodecane, tetradecane, and hexadecane) with *sec*-octyl alcohol by titration microcalorimetry. The critical micelle concentration (CMC), aggregation number (n), formation constant of micelle (K), and thermodynamic functions ($\Delta_r H_m^\theta$, $\Delta_r G_m^\theta$, and $\Delta_r S_m^\theta$) were obtained. For different quasi-surfactants and homologue of the alkanes, the relationships between the carbon number (N) of the alkanes with the critical micelle concentration (CMC) and the thermodynamic properties of the quasi-surfactants are discussed. It can be concluded as follows: (1) The microcalorimetric method is a good method to study the process of micelle formation. (2) The CMC decreased with an increase of the carbon number (N) of the alkanes, and the relative order of the CMC of different quasi-surfactants was D2EHPA(K) > D2EHPA(Na) > D2EHPA(Li) at the same carbon number. (3) The aggregation numbers (n) and formation constant of micelle (K) increased with an increase of the carbon number (N) of the alkanes. The relative order of different quasi-surfactants was D2EHPA(K) > D2EHPA(Na) > D2EHPA(Li) at the same carbon number. (4) The $\Delta_r H_m^\theta$ and $\Delta_r S_m^\theta$ increased, while $\Delta_r G_m^\theta$ decreased with an increase of the carbon number (N) of the alkanes. The relative order of the different quasi-surfactants was D2EHPA(Li) > D2EHPA(Na) > D2EHPA(K) at the same carbon number.

1. Introduction

The physicochemical properties of surfactants in nonaqueous solutions are not similar to those in aqueous solution due to interactions between the nonaqueous solvent and the surfactant. The surfactant could form micelles in nonaqueous solution. The development of micelles depends on the action of the steric hindrance of dipole−dipole interactions. Therefore, the aggregation number (n) of the micelle in nonaqueous solution is small (usually under 10).[1] The critical micelle concentration (CMC) is one important character for surfactant solutions. The determintion of the critical micelle concentration (CMC) can be achieved using many methods. Among them, microcalorimetry is a sensitive, quick, and easy method to measure CMC.

In extraction chemistry, the structure of the extractant is similar to that of the surfactant. Therefore, the extractant cannot exist in the single molecular state but occurs in the micelle state[2−5] in nonaqueous solutions. A few reports about research of nonaqueous solvents have been published in recent years.[6−12] In this paper, the properties of several quasi-surfactants were studied under different circumstances because of their commercial and theoretical value.

In this paper, the power−time curves of the micelle formation process were determined using titration microcalorimetry. The properties of lithium di(2-ethyl-hexyl) phosphate, sodium di(2-ethyl-hexyl) phosphate, and potassium di(2-ethyl-hexyl) phosphate were studied in different alkanes. The CMC, aggregation number (n), formation constant of the micelle (K), and thermodynamic functions ($\Delta_r H_m^\theta$, $\Delta_r G_m^\theta$, $\Delta_r S_m^\theta$) were calculated according to the mass action model and thermodynamic theory. The relationship between the carbon number (N) of the homologue of the alkane and the properties of the quasi-surfactant is discussed. The microcalorimetric study of these aspects has not been reported before.

2. Theory

The concentration of nonionic surfactant in the titration solution was high. When this solution was titrated into water, the nonionic surfactant dispersed and dissociated into a simple molecular state. When the concentration of the nonionic surfactant exceeded a certain value, the surfactant existed partially in the micelle state. The ionic surfactant could form a micelle in the organic solvent. This process of dispersion and dissociation is an endothermic process. In this process, the mass action model was used to investigate the corresponding properties. If there exists an association−dissociation equilibrium of the micelle state and nonmicelle state of surfactant,[1] then

$$n\mathrm{S} \overset{K}{\rightleftharpoons} \mathrm{M} \tag{1}$$

where K is the formation constant of micelle; n is the aggregation number; [S] is the monomer of the surfactant; and M is the micelle. From reaction 1

$$K = \frac{[\mathrm{M}]}{[\mathrm{S}]^n} \text{ or } nK = \frac{n[\mathrm{M}]}{[\mathrm{S}]^n} \tag{2}$$

$$\lg(nK) = \lg\{n[\mathrm{M}]\} - n\lg[\mathrm{S}] \text{ or } \lg\{n[\mathrm{M}]\} = \lg(nK) + n\lg[\mathrm{S}] \tag{3}$$

If the total concentration of surfactant is C_S

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$$[\mathrm{M}] = \frac{\{C_S - [\mathrm{S}]\}}{n} \qquad (4)$$

when eq 4 is substituted into eq 3, then

$$\lg\{C_S - [\mathrm{S}]\} = \lg(nK) + n\lg[\mathrm{S}] \qquad (5)$$

A curve was plotted of $\lg\{C_S-[S]\}$ against $\lg[S]$. According to this curve, the aggregation number (n) and formation constant of micelle (K) were calculated.

The values of C_S and [S] were calculated using the following method. The power−time curve is shown in Figure 1. The curve of P_{total} against V_t is shown in Figure 2, where $P_{\mathrm{total}} = P_1 + P_2 + P_3 + ... + P_i$, and P_i is the thermal power measured at the i time. $V_t = v \cdot t$, where v is the speed of the titrated solution and t is the titrated time. V_t is the titration volume. Two approximated lines can be made from Figure 2. From the data of Figure 2, a straight line was plotted between $P_{1,\mathrm{total}}$ and V_t together with the following equation.

$$(P_{1,\mathrm{total}} = a_1 V_t + b_1)$$

where a_1 was the slope of line 1 and b_1 was the intercept of line 1.

The values of a_2 and b_2 could also be obtained by a similar method according to the following beeline equation

$$(P_{2,\mathrm{total}} = a_2 V_t + b_2) \qquad (6)$$

where a_2 was the slope of line 2 and b_2 was the intercept of line 2.

The two lines have a crossover point. The equation of a certain experimental value behind this crossover point can be expressed as follows

$$P_{1,\mathrm{total}} = a_1 V^1 + b_1 \qquad (7)$$

where

$$P_{2,\mathrm{total}} = P_{1,\mathrm{total}} = P_{\mathrm{total}}$$

Then

$$V^1 = \frac{a_2}{a_1} V_t + \frac{b_2 - b_1}{a_1} \qquad (8)$$

V^1 is the corresponding titration volume of titrated solution.

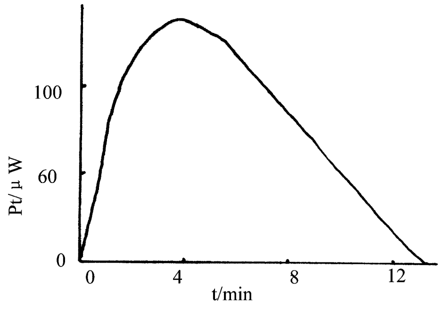

Figure 1. P_t−t curve of [D2EHPA(Na)] decane solution.

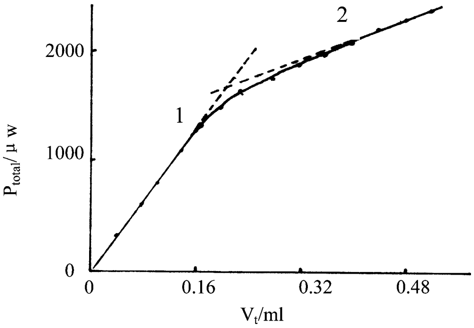

Figure 2. P_{total}−V_t curve of [D2EHPA(Na)] decane solution.

According to the following equimolar equation, eqs 9 and 10 could be obtained.

$$V_1 C_1 = V_2 C_2$$

where V was the volume of solution and C was the concentration of solution. Thus, $V^1 \cdot C$ (original solution concentration) = $(2 + V^1) \cdot [\mathrm{S}]$ (concentration of solution).

Finally, eq 9 was obtained.

$$[\mathrm{S}] = \frac{V^1}{2 + V^1} \cdot C \text{ (original solution concentration)} \qquad (9)$$

where V^1 was corresponding titrated volume of titrated solution; [S] was the concentration of solution of surfactant; and 2 was 2 mL of solvent.

Equation 10 could also be obtained using a similar method.

$$C_S = \frac{V_t}{2 + V_t} \cdot C \text{ (original solution concentration)} \qquad (10)$$

where V_t was the volume of the titrated solution; C_S was the total concentration of surfactant; and 2 was 2 mL of solvent.

From the power−time curve, the area (under the curve) represents the heat effect (Q). The concentration corresponding to the peak point of the curve is the critical micelle concentration (CMC). From the heat effect (Q) and the CMC, the standard enthalpy change ($\Delta H^{\theta}{}_{\mathrm{m}}$) of the micelle formation process was obtained.

According to the equation ($\Delta_r G_m^0 = -RT \ln K$ and $\Delta_r G_m^0 = \Delta_r H_m^0 - T\Delta_r S_m^0$), the thermodynamic function values ($\Delta_r G_m^0$, $\Delta_r S_m^0$) could be calculated.

3. Experimental Section

3.1. Instrument. A 2277 thermal activity monitor (Sweden) involves four independent calorimetric units. A monitored 23 L of isothermally thermostatic water was used. A 4 mL stainless steel titration ampoule unit was inserted into the thermostat. The titration ampoule unit was equipped with a stirrer and a stirrer motor, which rotated the stirrer shaft at the desired speed of (0 to 120) rpm. A Kelf turbine was used as a 4 mL ampoule to be filled with (2 to 3) mL of solution.

The working temperature range of the thermostat instrument was (10 to 80) °C. It was maintained at a given temperature constant to within $\pm 2 \cdot 10^{-4}$ °C. The system is very sensitive. The detection limit is 0.15 μW, and the baseline stability (over a period of 24 h) was 0.2 μW.

3.2. Materials. Solvent: homologue of the alkanes (normal octane, decane, dodecane, tetradecane, hexadecane), analytical grade (Tianjin Kermel Chemical Reagents Development Center).

Quasi-surfactant: lithium di(2-ethyl-hexyl) phosphate [D2EHPA(Li)], sodium di(2-ethyl-hexyl) phosphate [D2EHPA(Na)], and potassium di(2-ethyl-hexyl) phosphate [D2EHPA(K)] (synthesized in the laboratory, content 99.9 %).

Synthesis method: The pH of the solution, which included D2EHPAH and methyl alcohol, was adjusted to pH = 4.50 (5.20 or 5.40) by addition of LiOH (NaOH, KOH) solution. Then, the solution was flushed, filtered, purified, and dried. Finally, the solid salt of D2EHPA Li (Na or K) was obtained.

Table 1. Data of C_S and [S] for [D2EHPA(Li)] Dodecane Solution at 298.15 K

V_t / mL	V^1 / mL	$[S]\cdot 10^3$ / $mol\cdot L^{-1}$	$C_S\cdot 10^3$ / $mol\cdot L^{-1}$	$-lg[S]$	$-lg\{C_S-[S]\}$
0.32	0.273	1.239	1.423	2.907	3.735
0.34	0.281	1.271	1.499	2.896	3.642
0.36	0.288	1.299	1.574	2.886	3.561
0.38	0.295	1.327	1.648	2.877	3.495
0.40	0.302	1.354	1.720	2.868	3.437

Table 3. Data of C_S and [S] for [D2EHPA(K)] Dodecane Solution at 298.15 K

V_t / mL	V^1 / mL	$[S]\cdot 10^3$ / $mol\cdot L^{-1}$	$C_S\cdot 10^3$ / $mol\cdot L^{-1}$	$-lg[S]$	$-lg\{C_S-[S]\}$
0.30	0.276	1.251	1.346	2.903	4.022
0.32	0.286	1.291	1.423	2.889	43.879
0.34	0.295	1.327	1.499	2.877	3.765
0.36	0.304	1.362	1.574	2.866	3.674
0.38	0.314	1.400	1.648	2.854	3.606

Table 2. Data of C_S and [S] for [D2EHPA(Na)] Decane Solution at 298.15 K

V_t / mL	V^1 / mL	$[S]\cdot 10^3$ / $mol\cdot L^{-1}$	$C_S\cdot 10^3$ / $mol\cdot L^{-1}$	$-lg[S]$	$-lg\{C_S-[S]\}$
0.36	0.3191	1.4198	1.5742	2.8478	3.8119
0.38	0.3275	1.4519	1.6477	2.8380	3.7077
0.40	0.3359	1.4838	1.7200	2.8286	3.6267
0.42	0.3443	1.5155	1.7911	2.8194	3.5599
0.44	0.3527	1.5469	1.8610	2.8105	3.5029
0.46	0.3611	1.5781	1.9298	2.8019	3.4535

Cosurfactants: *sec*-octyl alcohol, analytical grade (Shanghai Chemical Reagents Plant).

Solution (1): $1.085\cdot 10^{-2}$ mol·L^{-1} D2EHPA(Li) (containing 10 % *sec*-octyl alcohol) homologue of the alkane solution.

Solution (2): $1.032\cdot 10^{-2}$ mol·L^{-1} D2EHPA(Na) (containing 10 % *sec*-octyl alcohol) homologue of the alkane solution.

Solution (3): $1.046\cdot 10^{-2}$ mol·L^{-1} D2EHPA(K) (containing 10 % *sec*-octyl alcohol) homologue of the alkane solution.

3.3. Method. A 4 mL stainless steel titration ampoule microcalorimetric unit was used in this experiment. A 2 mL organic solvent was added to the ampoule. A titration tube was used to titrate 0.5 mL of titrated solution (1), (2) or (3). This solution was pumped into the organic solvent in an ampoule by a micropepex pump. Since the speed of the titration addition was 0.02 mL·min^{-1}, the stirrer shaft was rotated at the desired speed of 120 rpm. Every system was determined three times at 298.15 K, and the average values of the power−time curves were obtained with a reported relative error less than 1.5 %.

4. Results

4.1. Determination of the Critical Micelle Concentration (CMC), Aggregation Numbers (n), and Formation Constant of Micelle (K). The power−time curves of the micelle formation process were determined at 298.15 K for different systems. Partial curves are shown in Figure 1. The critical micelle concentrations (CMC) were determined according to the corresponding concentration at the turn point of the power−time curve. Using data given in Figure 2, the C_S and [S] were calculated and are partial values shown in Tables 1 to 3. Plots of $-lg\{C_S-[S]\}$ against $-lg[S]$, the aggregation numbers (n) and formation constant of micelle (K) can be obtained, and the values are shown in Table 4.

4.2. Measurement of Thermodynamic Functions ($\Delta H^{\theta}{}_{m}$, $\Delta_r G^0_m$, $\Delta_r S^0_m$). From the power−time curve, the area (under the curve), which represented the heat effect (Q), could be obtained. Parallel control experiments were conducted to achieve the thermal effect caused by dilution of alcohol in the alkane solvent. From the area (Q) and the CMC, the standard enthalpy change ($\Delta H^{\theta}{}_{m}$) of the micelle formation process was calculated.

According to the formula ($\Delta_r G_m{}^0 = -RT \ln K$), $\Delta_r G_m{}^0$ was calculated. On the basis of the equation ($\Delta_r G^0_m = \Delta_r H^0_m - T\Delta_r S^0_m$), $\Delta_r S^0_m$ was also calculated. The values of these thermodynamic functions ($\Delta_r H_m{}^{\theta}$, $\Delta_r G_m{}^{\theta}$, $\Delta_r S_m{}^{\theta}$) are shown in Table 4.

5. Discussion

5.1. Relationship between the CMC and the Carbon Number (N) in the Homologue of the Alkane System. Plots of the CMC of quasi-surfactants [D2EHPA(Li), D2EHPA(Na), D2EHPA(K)] against the carbon number (N) which contained 10 % *sec*-octyl alcohol in different alkane systems are given in Figure 3 at 298.15 K. As can be seen from the figure, the CMC of quasi-surfactants decreased with an increase of the carbon number (N) in a homologue of the alkanes. The relative order of the CMC of different quasi-surfactants was D2EHPA(K) > D2EHPA(Na) > D2EHPA(Li) at the same carbon number.

5.2. Relationship between the Aggregation Number (n) and the Carbon Number (N) in the Homologue of the Alkane Systems. Plots of the aggregation number (n) of quasi-surfactants [D2EHPA(Li), D2EHPA(Na), D2EHPA(K)] against carbon number (N) in homologue of the alkanes are shown in

Table 4. CMC and Thermodynamic Property of D2EHPA(Li), D2EHPA(Na), and D2EHPA(K) in Different Alkane Systems at 298.15 K

quasi-surfactant	property	octane	decane	dodecane	tetradecane	hexadecane
D2EHPA(Li)	10^4CMC/mol·L^{-1}	6.51 ± 0.10	6.39 ± 0.09	6.14 ± 0.09	5.96 ± 0.08	5.75 ± 0.08
	n	7.3 ± 0.1	7.5 ± 0.1	7.7 ± 0.1	8.2 ± 0.1	8.9 ± 0.1
	K	$4.99\cdot 10^{16}$	$1.01\cdot 10^{17}$	$5.41\cdot 10^{17}$	$1.28\cdot 10^{19}$	$7.71\cdot 10^{20}$
	$\Delta_r H_m{}^{\theta}$/kJ·mol^{-1}	149 ± 2	180 ± 3	227 ± 4	274 ± 4	332 ± 5
	$\Delta_r G_m{}^{\theta}$/kJ·mol^{-1}	−95 ± 1	−97 ± 1	−101 ± 1	−109 ± 1	−119 ± 1
	$\Delta_r S_m{}^{\theta}$/J·K^{-1}·mol^{-1}	818 ± 12	929 ± 12	1100 ± 15	1285 ± 18	1513 ± 19
D2EHPA(Na)	10^4CMC/mol·L^{-1}	6.94 ± 0.10	6.75 ± 0.09	6.58 ± 0.09	6.41 ± 0.08	6.21 ± 0.08
	n	7.4 ± 0.1	7.8 ± 0.1	8.1 ± 0.1	8.6 ± 0.1	9.4 ± 0.1
	K	$6.73\cdot 10^{16}$	$2.47\cdot 10^{17}$	$5.70\cdot 10^{18}$	$4.83\cdot 10^{20}$	$6.11\cdot 10^{22}$
	$\Delta_r H_m{}^{\theta}$/kJ·mol^{-1}	131 ± 2	169 ± 3	206 ± 3	249 ± 4	307 ± 4
	$\Delta_r G_m{}^{\theta}$/kJ·mol^{-1}	−96 ± 1	−99 ± 1	−107 ± 1	−118 ± 2	−130 ± 2
	$\Delta_r S_m{}^{\theta}$/J·K^{-1}·mol^{-1}	771 ± 12	899 ± 12	1050 ± 14	1231 ± 16	1466 ± 20
D2EHPA(K)	10^4CMC/mol·L^{-1}	7.18 ± 0.11	7.04 ± 0.10	6.87 ± 0.10	6.70 ± 0.09	6.56 ± 0.08
	n	7.7 ± 0.1	8.0 ± 0.1	8.5 ± 0.1	9.1 ± 0.1	10.0 ± 0.1
	K	$1.51\cdot 10^{17}$	$1.13\cdot 10^{18}$	$6.52\cdot 10^{19}$	$2.74\cdot 10^{22}$	$5.20\cdot 10^{24}$
	$\Delta_r H_m{}^{\theta}$/kJ·mol^{-1}	115 ± 1	154 ± 2	180 ± 3	228 ± 3	275 ± 4
	$\Delta_r G_m{}^{\theta}$/kJ·mol^{-1}	−98 ± 1	−103 ± 1	−113 ± 1	−128 ± 1	−141 ± 2
	$\Delta_r S_m{}^{\theta}$/J·K^{-1}·mol^{-1}	714 ± 10	862 ± 11	983 ± 14	1194 ± 16	1395 ± 18

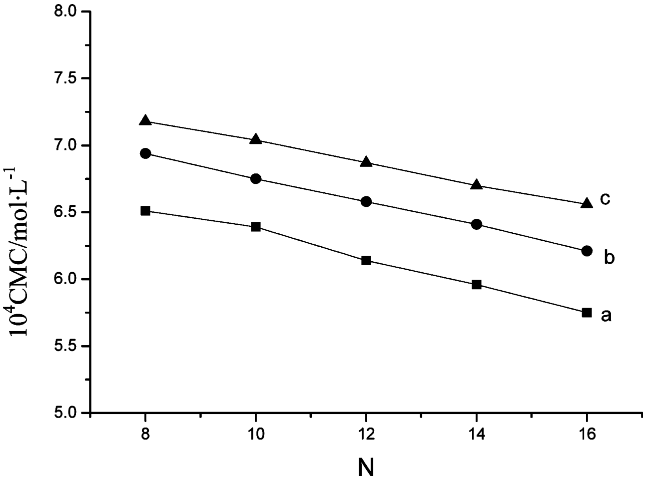

Figure 3. CMCs vs carbon number (N) curves in the homologue of the alkane at 298.15 K (a) D2EHPA(Li). (b) D2EHPA(Na). (c) D2EHPA(K).

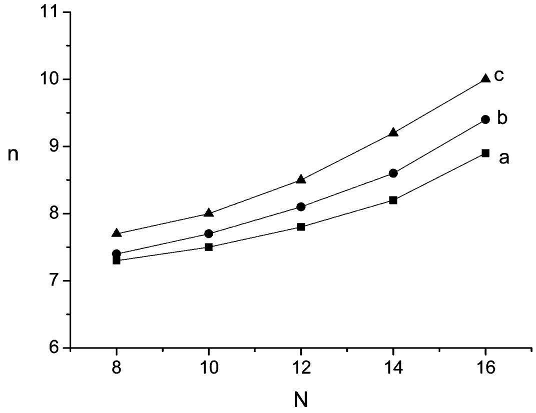

Figure 4. Aggregation number (n) vs carbon number (N) curves in homologue of the alkane at 298.15 K. (a) D2EHPA(Li). (b) D2EHPA(Na). (c) D2EHPA(K).

Figure 4. As shown in the figure, the aggregation number (n) increased with an increase of the carbon number (N) in the homologue of the alkanes. The relative order of different quasi-surfactants was D2EHPA(K) > D2EHPA(Na) > D2EHPA(Li) at the same carbon number.

5.3. Relationship between $\Delta_rH_m^0$ and the Carbon Number (N) in the Homologue of the Alkane System. The curves of $\Delta_rH_m^0-N$ are shown in Figure 5. As can be seen from the figure, the $\Delta_rH_m^0$ of the quasi-surfactants increased with an increase in the carbon number (N) of the homologue of the alkanes. The relative order of $\Delta_rH_m^0$ of different quasi-surfactants was D2EHPA(Li) > D2EHPA(Na) > D2EHPA(K) at the same carbon number.

5.4. Relationship between $\Delta_rG_m^0$ and the Carbon Number (N) in the Homologue of the Alkane System. From the values of Table 4, the $\Delta_rG_m^0$ of quasi-surfactants decreased with the increment of the carbon number (N) in the homologue of the alkanes. The relative order of different quasi-surfactants was D2EHPA(Li) > D2EHPA(Na) > D2EHPA(K) at the same carbon number.

5.5. Relationship between $\Delta_rS_m^0$ and the Carbon Number (N) in the Homologue of the Alkane System. From the values ($\Delta_rG_m^0$, ΔH^θ_m) in Table 4, the $\Delta_rS_m^0$ values were calculated. The $\Delta_rS_m^0$ of quasi-surfactants increased with the increment of the carbon number (N) in the homologue of alkane. The relative order of different quasi-surfactants was D2EHPA(Li) > D2EHPA(Na) > D2EHPA(K) at the same carbon number.

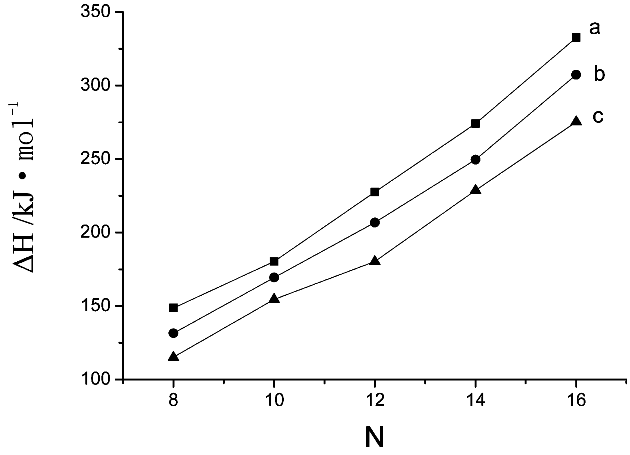

Figure 5. $\Delta_rH_m^0$ vs carbon number (N) curves in the homologue of alkane at 298.15 K. (a) D2EHPA(Li). (b) D2EHPA(Na). (c) D2EHPA(K).

According to the experimental results of the thermodynamic functions (ΔH^θ_m, $\Delta_rG^0_m$, ΔS^0_m), it can be concluded that it was consistent with the results of the thermodynamic theory.

In a word, the state of existence and thermodynamic properties of quasi-surfactants [di(2-ethyl-hexyl) phosphate salts] in an organic solvent were studied. The results are significant for the theory and application of micelle formation within extraction processes.

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