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HEAT CAPACITY AND PHASE BEHAVIOR OF $\{C_6E_4$+WATER$\}$ SOLUTIONS BY DSC

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The specific heat capacities of {2-(hexyloxytriethoxy)ethanol (C_6E_4)+water} system have been measured from 280 to 333 K within the whole composition range by DSC. Changes of specific, apparent and partial molar heat capacities of investigated aqueous solution *vs*. composition and temperature, considered as an effect of structural transformations were analyzed in order to draw boundary between region where amphiphile molecules occur as monomers and small aggregates and the area in which the first micelles appear. For each solution, the temperature dependences of the differential heat flow were analyzed in order to find the curve of phase coexistence, i.e. the boundary between one- and two-phase areas for the examined system.

Keywords: *coexistence curve, DSC, heat capacity, 2-(hexyloxytriethoxy)ethanol, water*

Introduction

This work is a continuation of our research on a phase behavior of aqueous solutions of monoalkyl derivatives of polyoxyethylene glycol with a general formula $\{C_nH_{2n+1}(OCH_2CH_2)_mOH\}$ (abbreviated to C_nE_m), where *n* refers to the number of carbon atoms in the hydrocarbon chain of ‘tail’ and *m* refers to the number of oxyethylene ($–OCH_2CH_2–$) units in the ‘head’ of molecule. For these non-ionic short-chain amphiphiles the closed-loop immiscibility region in their mixtures with water is characteristic. It is also known that some of these compounds in the range of diluted aqueous solutions form a microheterogeneity region [1–3]; other papers report about the micellisation process, connected with the formation of isotropic micellar phase coexisting with monomers and small aggregates of the amphiphile [4–7] as well as about formation of globular micelles in dilute aqueous solutions [7–9]. In our earlier publications the thermochemical properties of the mixtures of C_6E_2 and C_6E_5 with water within a relatively wide temperature range were discussed [10, 11]. This paper is devoted to the results of examination of aqueous solutions of another compound from the same group, namely 2-(hexyloxytriethoxy)ethanol, $\{C_6H_{13}(OCH_2CH_2)_4OH\}$, abbr. ($C_6E_4$). The mean aggregation number of micelles in this system, *N*=56 was determined indirectly, by interpolation of data for similar systems (C_nE_4 with different *n*) [12]. The critical micellisation concentration (*cmc*) values at 298 K given in the literature are: 0.080 mol dm^{-3} [12]; 2 mass% [13] or mole fraction $1.97\cdot10^{-3}$ [14]. The *cmc* decreases slightly when the temperature increases [9, 13, 15]. What concerns the phase behavior of the C_6E_4 aqueous solutions there are only a few literature reports devoted to this subject. The experimentally determined values of lower critical solution temperature (LCST) are: 66.3 [12, 14], 60 [16] and 66.1°C [13]. The same quantity calculated from empirical correlation proposed by Kahlweit *et al*. [17] equals 65.1°C while the statistical associating fluid theory (SAFT) approach predict cloud curve with LCST=54°C [18]. The critical composition given in Schubert *et al*. paper equals 16.4 mass% [13]. However, neither the information about the effect of the temperature and the $\{C_6E_4$+water$\}$ mixture composition on the phase transition nor about the microphase formation in the system is given in these papers. Only in Schubert [13] and Strey’s paper [15] a small fragment of curve illustrating the immiscibility region in aqueous solutions of C_6E_4 is given, but due to the lack of numerical data the value of LCST≈65°C could be only estimated.

In our work we intended to determine a phase diagram for $\{C_6E_4$+water$\}$ system. To this aim, similarly as in our earlier works [10, 11, 19], we have analyzed the partial molar heat capacity ($C_{p,2}$) of the amphiphile in solution. The mentioned function, as the third derivative of the excess Gibbs free energy *vs*. composition and temperature:

$$C_{p,2}=-T(\partial^3G/\partial n_2\partial T^2)_{p,n_1} \qquad (1)$$

is particularly sensitive for any structural changes in the system. Hence, the analysis of the $C_{p,2}$ *vs*. the mixture composition and the temperature makes it possible to distinguish regions in solution which are characterized by different molecular organizations, and especially between homogeneous and microheterogeneous

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areas. For this reason, we measured the specific heat capacity (c_p) of the $\{C_6E_4$+water$\}$ mixtures within the temperature range from 280 to 333 K within the whole composition range by differential scanning calorimetry which is an effective technique for the determination of thermal behavior of compounds [20, 21]. The measured c_p values were then used for calculations the apparent and partial molar heat capacities of C_6E_4 in the examined solutions at several temperatures. Additionally the temperature dependence of the differential heat flow (HF) was analyzed in order to find the curve of phase coexistence for the examined system and the boundary between the one-phase and two-phase area. In this way a complete set of information, we were interested in, was obtained.

Experimental

The 2-(hexyloxytriethoxy)ethanol (Bachem; >99%) was used as received without further purification. Deionised water was triply distilled in an argon atmosphere and degassed. The purity of water was checked by conductometry; the specific conductivity was less than $2\cdot10^{-6}$ S cm^{-1} at 298 K. All solutions were prepared by mass. A Sartorius RC 210D balance (with an accuracy of $2\cdot10^{-5}$ g) was used to determine the sample mass. The specific heat capacities at constant pressure of $\{C_6E_4$+water$\}$ mixtures were measured by means of a Micro DSC III (Setaram) high sensitivity differential scanning calorimeter based on the Tian–Calvet's principle. The 'continuous with reference' method (water as a reference) was applied. Within the examined temperature range 280–333 K the temperature was changed at a constant rate of 0.15 K min^{-1}. The measuring vessel was a standard 'batch' type cell with a volume of about 1.0 cm^3. The uncertainty of the c_p values obtained in this work did not exceed ±0.15%, excluding the effects of sample impurities. The details of apparatus and measuring procedure are described elsewhere [22].

In order to establish a relation between the composition of solution and the temperature, at which the second phase appears we determined additionally the course of temperature relationships of the differential HF for the mixtures containing from about 2 to 62 mass% ($0.001<x_2<0.09$) of the surfactant.

Results and discussion

Miscibility gap in C_6E_4/water system

Figure 1 presents the course of typical curve of the differential heat flow *vs.* temperature recorded between 337 and 358 K at a scanning rate of 0.1 K min^{-1}. The heat flow in the temperature range, where the single-phase solution is stable, shows a higher value than that within the range of two-phase system. The appearance of the second phase in solution brings about a stepwise decrease in HF that reflects a drop in the heat capacity of solution in this process. For each of the examined $\{C_6E_4$+water$\}$ mixtures we have determined the temperature of equilibrium transition (T) between the single-phase and two-phase solution, i.e., so-called extrapolated onset temperature. The onset point is an intersection point of tangent, drawn where the curve has the highest slope, and the extrapolated baseline. However, in the case of very diluted solutions, no characteristic leap in the curve HF=$f(T)$ was observed due to too small difference between the heat capacities of the single-phase and two-phase system, and that is why the analyzed range of the solutions composition was limited. The determined as above the dependence between the $\{C_6E_4$+water$\}$ mixture composition and the transition temperature between the single-phase and two-phase solution, $T=f(x_2)$ is presented in Fig. 2. Below the curve there is a homogenous and microheterogeneous region, but above the curve we have large region of liquid–liquid immiscibility between two isotropic micellar solutions. In the latter region two phases coexist, one rich in water and the other rich in surfactant. Obtained in this work value of the LCST=65.23°C is very close to these determined by Kahlweit *et al.* [17] and Strey [15], and the critical composition (16.1 mass%) is in a very good agreement with mentioned earlier one reported by Schubert *et al.* [13].

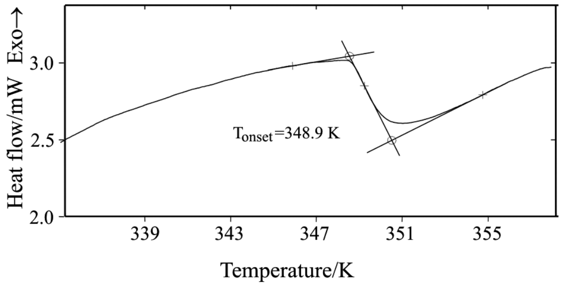

Fig. 1 A DSC trace of aqueous 2-(hexyloxytriethoxy)ethanol (the mole fraction of amphiphile x_2=0.06896)

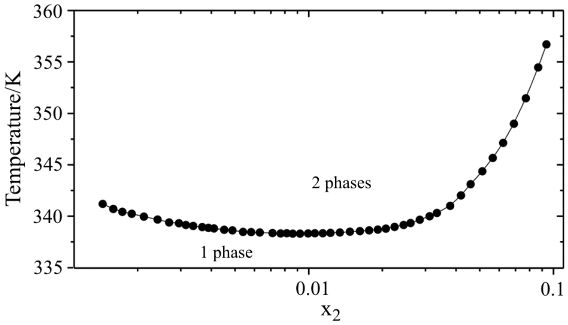

Fig. 2 Coexistence curve for $\{C_6E_4$+water$\}$ mixtures

Heat capacity

The determined specific heat capacities of mixtures within the examined temperature range were described by the polynomial:

$$c_p(T)=\sum_{i=-1}^{3}A_i T^i \qquad (2)$$

where $c_p(T)$ – specific heat capacity of the examined solution at temperature T; A_i – constant.

The selection of the polynomial degree (i) depends of the shape of the experimental curve $c_p=f(T)$ obtained for the mixture with precisely defined composition. The interpolated values of c_p at seven selected temperatures are given in Table 1. The dependence of the specific heat capacity of the {C_6E_4+water} system on the composition at several temperatures is presented in Fig. 3. For better clarity, in Fig. 3a only two curves for extreme temperatures are shown. The curves $c_p=f(x_2)$ for $x_2>0.01$ have similar character, the monotonous decrease of the specific heat capacity when the amphiphile content in solution increases. Significant differences in the shape of $c_p=f(x_2)$ function can be observed only in very dilute solutions (Fig. 3b). At each of the examined temperatures for $0<x_2<0.002$ the specific heat capacity slightly decreases when the concentration of the 2-(hexyloxytriethoxy)ethanol growths. The further growth of the amphiphile content in the system ($0.002\leq x_2\leq 0.0043$) brings about a significant increase of the c_p up to its maximal value. The increase of the temperature makes the maximum of the $c_p=f(x_2)$ functions lower and shifted to smaller content of the C_6E_4 in solution. At the highest of the examined temperatures (333 K) there is no maximum on the $c_p=f(x_2)$ curve. Instead a significant change in the slope of the function is observed. It can be supposed that the growth of the temperature promote the dehydration process of C_6E_4 molecules, which facilitate formation of different type associates. Their presence in solution can be a reason of the observed different shape of the solution specific heat capacity curves at different temperatures.

On the base of experimentally determined specific heat capacity data, we have calculated the reduced excess molar heat capacities (C_p^E/x_1x_2) of solutions as well as apparent ($C_{\phi,2}$) and partial molar heat capacities ($C_{p,2}$) of C_6E_4 in aqueous solutions, using general thermodynamic relationships:

$$C_p^E/x_1x_2=(C_{p,S}-C_{p,1}^*x_1-C_{p,2}^*x_2)/x_1x_2 \qquad (3)$$

$$C_{\phi,2}=M_2c_p+1000(c_p-c_{p,1}^*)/m_2 \qquad (4)$$

$$C_{p,2}=C_{\phi,2}+m_2(\partial C_{\phi,2}/\partial m_2)_{T,p} \qquad (5)$$

where M_2 (g mol^{-1}) is the amphiphile molecular mass; m_2 (mol kg^{-1}) is the solution molality; x_1 and x_2 are the molar fractions; $C_{p,S}$ is the molar heat capacity of solution; $C_{p,1}^*$ and $C_{p,2}^*$ is the molar heat capacity of pure water and amphiphile; c_p and $c_{p,1}^*$ (J g^{-1} K^{-1}) is the specific heat capacity of solution and pure water, respectively.

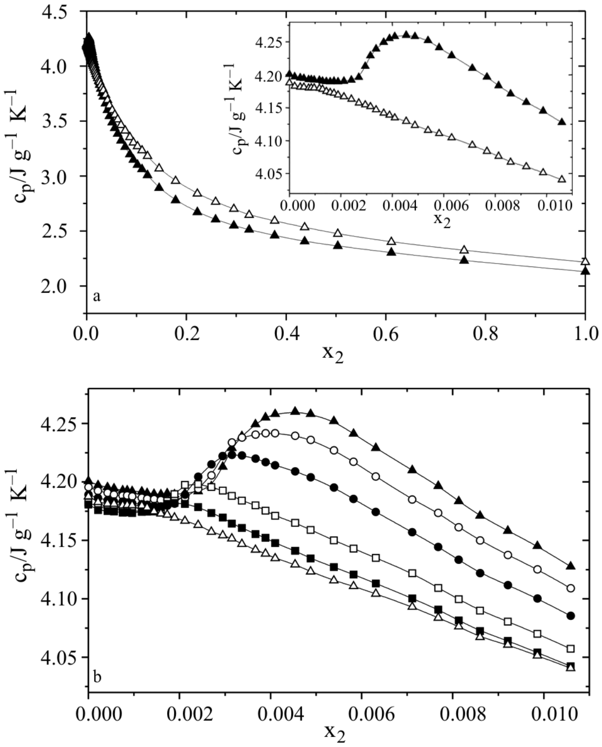

Fig. 3 The specific heat capacities (c_p) of aqueous solutions of C_6E_4 *vs.* the mole fraction of amphiphile at various temperatures: a – ▲ – 280 K, △ – 333 K, b – ▲ – 280 K ○ – 283 K, ● – 288 K, □ – 298 K, ■ – 303 K and △ – 333 K

As it can be seen in Fig. 4 the relationships $C_p^E=f(x_2)$ pass through a broad and symmetrical maximum, whose shape and position are independent of temperature, although its height grows with temperature. The observed positive values of the excess of molar heat capacity (C_p^E) within a whole range of the examined mixture composition and at each temperature suggest an association process leading to formation of a mixture of different type aggregates having relatively high thermal stability. The pronounced differences in the shape of the C_p^E function at different temperatures are observed only in the range of highly diluted solutions, $x_2<0.003$. They are even better visible when we replace the excess molar heat capacity by, so-called, reduced excess molar heat capacity (C_p^E/x_1x_2), a function which more emphasizes the existing differences (Fig. 4). The $C_p^E/x_1x_2=f(x_2)$ curves exhibit sharp maximum, that decreases and its position shifts towards lower amphiphile content when the temperature growths. It is noteworthy, that the reduced excess molar heat capacity for solution with $x_2\approx 0.025$ is temperature independent, i.e. the deviations from the ideality of the solution having this composition are almost the same at each examined temperature.

Table 1 Specific heat capacity (c_p) of {$C_6E_4(x_2,m_2)$+water}system from 280 to 333 K

x_2	m_2/mol kg^{-1}	c_p/J g^{-1} K^{-1}						
		280 K	283 K	288 K	298 K	303 K	313 K	333 K
0	0	4.2007	4.1956	4.1891	4.1823	4.1807	4.1822	4.1875
0.00021	0.01167	4.1971	4.1924	4.1863	4.1789	4.1760	4.1773	4.1827
0.00042	0.02319	4.1954	4.1906	4.1845	4.1771	4.1751	4.1761	4.1814
0.00056	0.03138	4.1944	4.1892	4.1832	4.1764	4.1747	4.1756	4.1812
0.00075	0.04155	4.1927	4.1883	4.1831	4.1760	4.1738	4.1746	4.1799
0.00085	0.04743	4.1927	4.1885	4.1823	4.1757	4.1739	4.1751	4.1802
0.00096	0.05355	4.1921	4.1876	4.1818	4.1752	4.1735	4.1760	4.1800
0.00117	0.06484	4.1917	4.1864	4.1810	4.1750	4.1745	4.1774	4.1796
0.00129	0.07190	4.1904	4.1857	4.1805	4.1746	4.1751	4.1773	4.1780
0.00144	0.08028	4.1894	4.1853	4.1794	4.1747	4.1771	4.1767	4.1747
0.00159	0.08856	4.1893	4.1844	4.1790	4.1763	4.1785	4.1760	4.1730
0.00173	0.09635	4.1902	4.1844	4.1797	4.1837	4.1811	4.1752	4.1720
0.00189	0.10504	4.1893	4.1850	4.1814	4.1896	4.1821	4.1734	4.1694
0.00212	0.11807	4.1902	4.1864	4.1892	4.1975	4.1812	4.1708	4.1667
0.00241	0.13438	4.1919	4.1940	4.2045	4.1981	4.1784	4.1666	4.1631
0.00270	0.15008	4.1962	4.2057	4.2151	4.1956	4.1734	4.1614	4.1573
0.00295	0.16420	4.2129	4.2221	4.2222	4.1926	4.1695	4.1579	4.1543
0.00315	0.17532	4.2289	4.2337	4.2232	4.1881	4.1644	4.1545	4.1516
0.00337	0.18756	4.2392	4.2379	4.2226	4.1840	4.1607	4.1499	4.1469
0.00367	0.20450	4.2493	4.2405	4.2200	4.1800	4.1555	4.1446	4.1419
0.00389	0.21696	4.2551	4.2416	4.2170	4.1754	4.1522	4.1418	4.1386
0.00410	0.22857	4.2580	4.2417	4.2143	4.1710	4.1477	4.1375	4.1347
0.00454	0.25297	4.2600	4.2395	4.2091	4.1650	4.1411	4.1322	4.1294
0.00488	0.27210	4.2580	4.2360	4.2040	4.1590	4.1345	4.1248	4.1233
0.00539	0.30007	4.2520	4.2270	4.1952	4.1500	4.1271	4.1173	4.1157
0.00582	0.32473	4.2412	4.2173	4.1854	4.1430	4.1208	4.1120	4.1108
0.00631	0.35234	4.2290	4.2047	4.1743	4.1350	4.1132	4.1058	4.1043
0.00711	0.39763	4.2100	4.1850	4.1570	4.1220	4.1004	4.0920	4.0931
0.00768	0.42984	4.1965	4.1736	4.1444	4.1094	4.0908	4.0829	4.0838
0.00813	0.45475	4.1835	4.1613	4.1334	4.0997	4.0816	4.0736	4.0763
0.00860	0.48159	4.1710	4.1500	4.1220	4.0900	4.0725	4.0640	4.0673
0.00921	0.51598	4.1582	4.1375	4.1117	4.0806	4.0641	4.0570	4.0606
0.00986	0.55265	4.1451	4.1253	4.1003	4.0701	4.0541	4.0474	4.0514
0.01059	0.59438	4.1276	4.1090	4.0855	4.0573	4.0424	4.0363	4.0407
0.01141	0.64043	4.1050	4.0880	4.0660	4.0430	4.0246	4.0196	4.0255
0.01226	0.68879	4.0854	4.0703	4.0506	4.0260	4.0138	4.0098	4.0158
0.01341	0.75434	4.0606	4.0455	4.0275	4.0101	4.0038	3.9980	4.0019
0.01472	0.82949	4.0372	4.0253	4.0101	3.9906	3.9811	3.9786	3.9863
0.01618	0.91276	4.0086	3.9983	3.9850	3.9687	3.9605	3.9584	3.9671
0.01765	0.99743	3.9811	3.9728	3.9620	3.9486	3.9422	3.9412	3.9507
0.01915	1.0839	3.9502	3.9434	3.9347	3.9244	3.9194	3.9187	3.9286
0.02067	1.1716	3.9257	3.9206	3.9140	3.9059	3.9021	3.9021	3.9107
0.02242	1.2728	3.8955	3.8916	3.8874	3.8831	3.8805	3.8803	3.8906
0.02440	1.3880	3.8624	3.8608	3.8586	3.8562	3.8558	3.8576	3.8692

Table 1 Continued

x_2	m_2/mol kg^{-1}	c_p/J g^{-1} K^{-1}						
		280 K	283 K	288 K	298 K	303 K	313 K	333 K
0.02606	1.4852	3.8290	3.8400	3.8390	3.8384	3.8396	3.8422	3.8546
0.02846	1.6263	3.7940	3.8030	3.8066	3.8091	3.8118	3.8155	3.8279
0.03120	1.7879	3.7540	3.7650	3.7725	3.7781	3.7834	3.7883	3.8026
0.03343	1.9201	3.7230	3.7290	3.7444	3.7532	3.7560	3.7654	3.7796
0.03789	2.1860	3.6600	3.6670	3.6910	3.7000	3.7090	3.7214	3.7363
0.04189	2.4272	3.6000	3.6050	3.6118	3.6490	3.6540	3.6583	3.6900
0.04585	2.6672	3.5450	3.5600	3.5706	3.5942	3.6112	3.6241	3.6408
0.05122	2.9969	3.4870	3.5000	3.5181	3.5448	3.5649	3.5808	3.5983
0.05653	3.3262	3.4365	3.4503	3.4697	3.4990	3.5212	3.5387	3.5577
0.06230	3.6877	3.3844	3.3992	3.4202	3.4520	3.4762	3.4951	3.5150
0.06896	4.1117	3.3229	3.3357	3.3556	3.3895	3.4171	3.4393	3.4632
0.07709	4.6367	3.2651	3.2808	3.3034	3.3388	3.3665	3.3887	3.4130
0.08666	5.2665	3.1974	3.2128	3.2357	3.2727	3.3024	3.3265	3.3532
0.09364	5.7347	3.1480	3.1588	3.1916	3.2341	3.2658	3.2879	3.3050
0.10205	6.3086	3.1017	3.1131	3.1306	3.1638	3.2080	3.2277	3.2705
0.11094	6.9269	3.0622	3.0660	3.1003	3.1392	3.1719	3.1997	3.2331
0.12204	7.7158	3.0072	3.0150	3.0441	3.0827	3.1159	3.1449	3.1807
0.14537	9.4416	2.8906	2.9039	2.9248	2.9640	2.9991	3.0299	3.0676
0.17837	12.051	2.7798	2.7917	2.8107	2.8478	2.8825	2.9142	2.9554
0.22202	15.841	2.6714	2.6815	2.6980	2.7321	2.7654	2.7967	2.8394
0.25855	19.356	2.6045	2.6136	2.6287	2.6602	2.6916	2.7220	2.7649
0.29525	23.255	2.5491	2.5573	2.5708	2.5995	2.6290	2.6580	2.7003
0.32582	26.826	2.5120	2.5198	2.5333	2.5643	2.5980	2.6334	2.6450
0.37691	33.578	2.4592	2.4652	2.4761	2.5007	2.5265	2.5525	2.5915
0.43727	43.134	2.4050	2.4110	2.4210	2.4400	2.4640	2.4870	2.5330
0.50349	56.289	2.3636	2.3683	2.3760	2.3953	2.4169	2.4394	2.4737
0.61112	87.232	2.3024	2.3058	2.3121	2.3286	2.3476	2.3679	2.3997
0.75698	172.90	2.2299	2.2329	2.2387	2.2542	2.2723	2.2917	2.3225
1		2.1299	2.1325	2.1379	2.1524	2.1695	2.1878	2.2171

Essential information on the behavior of examined system can be obtained from the analysis of the apparent and partial molar heat capacity of 2-(hexyloxytriethoxy)ethanol in water as a function of the solution composition and the effect of temperature changes on the course of these functions (Figs 5 and 6). The both analyzed functions, i.e. $C_{\phi,2}=f(x_2)$ and $C_{p,2}=f(x_2)$ have a similar shape with a maximum in the range of dilute solutions which shifts towards smaller amphiphile content when the temperature grows. The intensity of the maximum is the highest at 280 K and it becomes smaller at higher temperatures. The observed changes in the shape of the $C_{\phi,2}=f(x_2)$ and $C_{p,2}=f(x_2)$ functions can be attributed to formation of metastable micellar or pseudomicellar aggregates in solution, similarly as in the case of examined earlier aqueous solutions of 2-butoxyethanol (C_4E_1) [19], 2-(hexyloxyethoxy)ethanol (C_6E_2) [10], and 2-(hexyloxytetraethoxy)ethanol (C_6E_5) [11]. Therefore, a curve which illustrates the temperature dependence of the solution composition corresponding to the $C_{p,2}=f(x_2)$ maximum, determined on the base of our experimental data (Fig. 7) presents a boundary between a homogenic region in which monomers and small molecular aggregates dominate and a microheterogenic area. It must be also borne in mind that the micellar solutions behave differently from the simple binary mixtures as the micelles are polydispersional and they can change their size and shapes when we move from one to another point on the temperature–mixture composition plot. Therefore, as we suggested earlier, this process is not of the sharp jump nature but the gradual restructuring of the mixture structure occurs in the some vicinity of the critical concentration [10, 19].

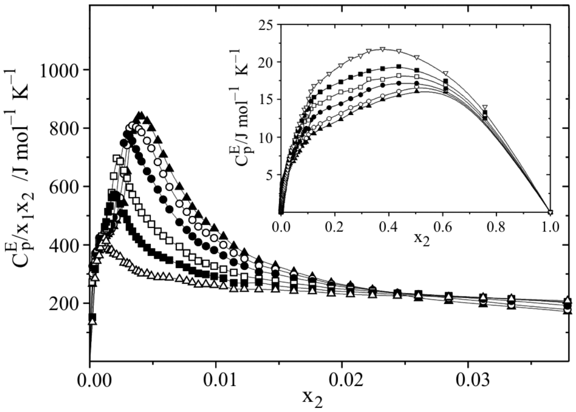

Fig. 4 The reduced excess molar heat capacities (C_p^E/x_1x_2) and excess molar heat capacities (C_p^E) of {C_6E_4+water} system *vs*. the mole fraction of amphiphile as a function of temperatures: ▲ – 280 K, ○ – 283 K, ● – 288 K, □ – 298 K, ■ – 303 K and △ – 333 K

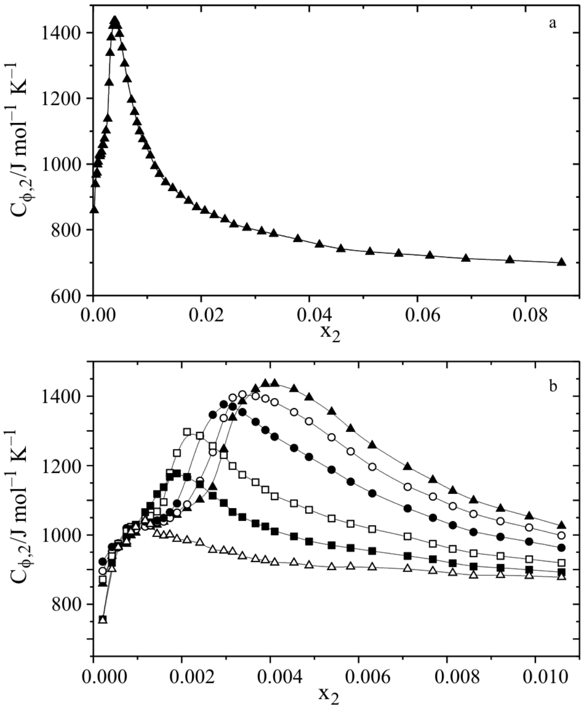

Fig. 5 Apparent molar heat capacities ($C_{\phi,2}$) of C_6E_4 in aqueous solutions at various temperatures: a – at 280 K; b – at different temperatures: ▲ – 280 K, ○ – 283 K, ● – 288 K, □ – 298 K, ■ – 303 K and △ – 333 K

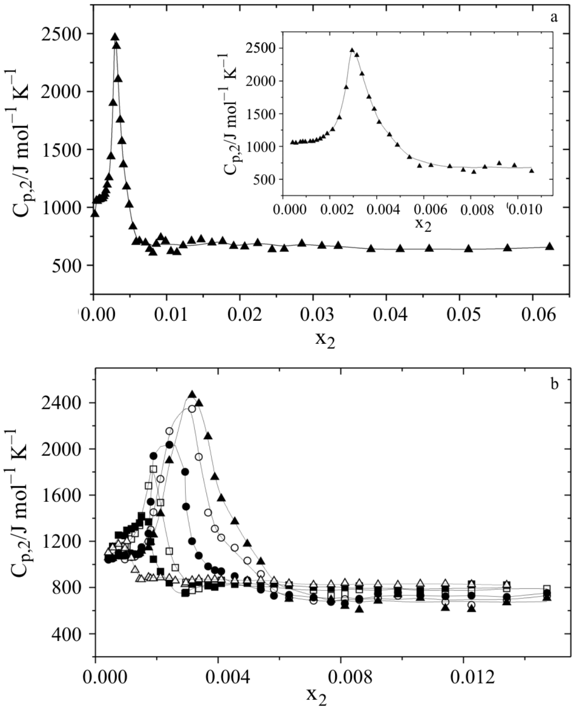

Fig. 6 Partial molar heat capacities ($C_{p,2}$) of C_6E_4 in the amphiphile/water mixtures at various temperatures: a – at 280 K; b – at different temperatures: ▲ – 280 K, ○ – 283 K, ● – 288 K, □ – 298 K, ■ – 303 K and △ – 333 K

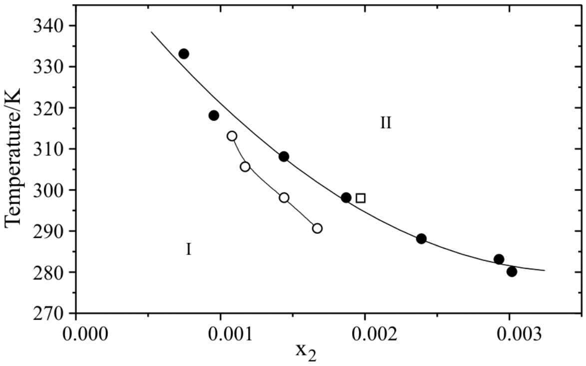

Fig. 7 The mixing scheme boundary separating regions I and II in aqueous solution C_6E_4 : I – domination of monomers and small associates; II – isotropic micellar phase; ● – the maximum of partial molar heat capacity [this paper]; ○ – *cmc* value [12]; □ – *cmc* value [8]

The borderline between the homo- and microheterogenic areas (Fig. 7), which defines simultaneously the micellisation process conditions (temperature–mixture composition), obtained in this work, is very close to the *cmc* curve determined for the same system on the base of literature data [12]. At the temperature of 298 K the maximum of partial molar heat capacity ($C_{p,2}$) of the amphiphile appears in the solution containing 0.10399 mole of C_6E_4 in 1 kg of water (x_2=0.00187). This value is in a good agreement with the previously cited literature data concerning the critical concentration of micellisation (x_2=0.00197 [8]). Also the value of *cmc* at the temperature of 313 K, obtained by Telgmann and Kaatze [12], amounting to 60 mM (x_2≈0.00108), is well located on the curve shown in Fig. 7. Hence, the mixture composition for which the $C_{p,2}$=*f*(x_2) function reach its maximum approximately corresponds to *cmc*.

Conclusions

The performed analysis of the apparent and partial molar heat capacity of C_6E_4 in aqueous solution as a function of composition and temperature suggests that in the examined system the phase transition from regular to micelle-like solution takes place in the range of highly diluted solutions.

The examination of the temperature relationship of the differential heat flow obtained by DSC is useful for determination of miscibility diagrams in the liquid systems where the phase separation occurs.

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