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Research Paper :

Density measurement of alkali and alkaline earth metal myristates in non-aqueous medium $(30^{\circ}-50^{\circ}C)$

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See end of the article for authors' affiliations	ABSTRACT The present investigation deals with density measurement of alkali (Li, Na, K) and alkaline earth
Correspondence to:	metal (Mg, Ca, Sr, Ba) myristates in 50% methanol-50% chloroform (V/V) solvent-mixture at 30°-
ANILKUMAR Department of Chemistry, D.A.V. (P.G) College, MUZAFFARNAGAR (U.P.) INDIA	S0°C. The data have been used to determine critical micelle concentration, cmc, and compute other significant parameters <i>viz</i> . apparent (ϕ_v) and partial (ϕ_v^0) molar volumes and partial molar expansibility, E° _{surfactant} . The decreasing cmc with the increase in temperature is in agreement with the result obtained from specific conductance measurements. The variation of cmc with alkali and alkaline earth metals also follows the same order as was found in the study on conductance measurements.

Key words : Critical micelle concentration (c.m.c.), Density, Molar volume and expansibility

ata on molar volume have proved to be a useful tool D to study the solute-solvent interactions (Bahadur, 1974). The apparent molar volume of glycine for inorganic salt solution of different concentrations was found to increase linearly with increasing concentration of the ions (Bhargava, 1976). Density data for aqueous solutions of 4-amino butyric acids at 25°C are also found to vary linearly with molarity (Blockhra and Verma, 1977). The apparent molar volume of ammonium acetate solutions was also determined from density data using Young's rule (Blokhra and Thakur, 1981). Dielectric constant of the medium was used to characterize tetraalkylamm-onium iodides in ethanol-water mixtures (Franks and Ives 1966). Dielectric constant however was found to play a dominating role in controlling the nature of the slope, Sv. Reference ('Franks and Luickenden, 1968; Franks et al., 1968; Hepler, 1969; Jalicoeur and Philip, 1975; Kashimato et al., 2006 and Key and Evans, 1966) on solute- solvent interactions indicate that density measurements proved useful to detect and explain the process of micellization. The density measurements were employed to study the micellar behaviour of the calcium soaps in water, methanol and water-methanol mixtures (Kim et al., 2006). Density data was also used to determine the c.m.c. of copper (Lafitte, 2005), iron (mason, 1947 and Mehrotra et al., 1970) and cobalt (Millero, 1968) soaps of lower fatty acids in non-aqueous medium.

Apparent molar volume and limiting apparent molar volumes for electrolytes (Milero, 1970) and nonelectrolytes (Millero, 1971) have found application to characterize solute-solute , solute-solvent and solvent – solvent interactions taking place in solutions. Hepler (Ram Gopal and Siddiqi, 1968) and Jolicoeur et al. (Ram Gopal et al., 1970) have however studied the effect of temperature on partial molar volume of hydrophopic solutes so as to obtain significant informations on solutesolvent interactions. Recently, however, research workers (Ram Gopal et al., 1973, Ram Gopal and Pathak, 1978 and root, 1933) have shown a keen interest for surface charge density of various amphipathetic substances. Kim et al., in the year, 2006 have thus carried out an investigation on polymerized rod like nanoparticles with controlled surface charge density. G. Lafitte and Co-Workers have utilized mixtures of mucin and oppositvely charged surfactant aggregates with varying charged density to probe their phase behaviour, association, and dynamics. Kashimoto et al. (Root, 1933) have also probed the surface density of surface-active substances through total reflection x-rays absorption fine structure measurement.

Present investigation however incorporates work in partial molar volume and expansibility so as to obtain useful information on micellization and solution behaviour *i.e.* solute-solvent and solute-solute interactions. Increasing surfactant concentration and increasing temperature increases the apparent molar volume which is consistent with the general expectation. Partial molar expansibility (E_{surf}^0) for these surfactant systems are also found to increase with increasing temperature.

MATERIALS AND METHODS

GR grade magnesium, calcium, strontium and barium

carbonates were used. Myristric acid obtained from Sigma Chemical Co. U.S.A. of stated high purity was used as received. The conductivity water used for experimental work was prepared by redistilling good quality distilled water over alkaline $KMnO_4$.

Myristates of lithium, sodium and potassium were first prepared by reaction of warm aqueous solutions of alkali metal carbonates containing phenolphthalein as indicator with fused myristric acid. Stoichiometric amounts of both alkali metal carbonates and myristric acid were employed. The completion of the reaction is thus indicated by the decolourisation of the pink coloured solutions. The dried and recrystallised pure compounds were found to decompose between 190°-205°C. Now, alkaline earth metal myristates were prepared by metathesis method. Warm aqueous solutions of alkaline earth metal carbonates, containing phenolphthalein as indicator, were individually treated with the warm aqueous solutions of potassium myristate employing their stoichiometric amounts. The completion of the reaction is indicated as the pink colour gradually vanishes. The compounds formed were separated from their mother liquor by filtration, washed with benzene and dried. The white coloured compounds were recrystallised from 50% methanol +50% chloroform solvent-mixture and oven dried to obtain high quality products. The compounds are found to decompose between 190°-205°C.

Pyrex glass dilatometers having a 15 ml reservoir were used to determine density (r) of these solutions at different temperatures ($\pm 0.05^{\circ}$ C). The reproductibility of the data is found to be ± 0.002 gcm⁻³. The accuracy of the results was also checked by determining the molar volume of sodium chloride at 45°C. The experimental value (17.57 cm³ mol⁻¹) has been found to be consistent with the data (17.59 cm³ mole⁻¹) as obtained by Millero.

RESULTS AND DISCUSSION

The density, ρ (g cm⁻³) data of akali (Li, Na, K) and alkaline earth metal (Mg, Ca, Sr, Ba) myristates in solventmixture of 50% methanol and 50% chloroform at 30⁰– 50°C (Table 1, 2 and 3) are found to increase with increasing concentration, C (mol.dm⁻³) as is evident from ρ -c plots (Fig.1, 2). The critical micelle concentration (c.m.c.) as obtained from ρ -c plots is found to decrease with increasing metal size and increasing temperature. The graphical values for zero metal soap concentration (ρ - c plots extrapolated to zero concentration) are found to be consistent with experimental ρ_o data (Table 4), a fact indicating the accuracy of density data for these surfactant systems (1-3).

The equation $\rho = \rho_0 + AC - BC^{3/2}$, by W.C. Roots

E. D. C.	5 . DC 3 . DV C	E.E. O. 1.5	18 8 C. S.	5) 807.2°5	0. 2000)										
Core C×1.0'	3000	35°C	1.4 Wyriste 10°C	1500	20,05	2008	35%C	NE NYTÉRE	1500	2005	3000	32,60	< Nytsee	2.54	2050
2.0	093/20	. 093269	0932	01.092921	1.1.7.60	093/25	. 0932.73		092.922.		. 093/28	093278	093.19	09292.9	092125
37	093/38	.0932.86	093.76	092936	. 09273.	1.786	0932.92	093.2.9	092933	092.128	. 093/55	093303	. 093.7.	676260	\$ 11:660 .
6.0	. 093/75	.093302	. 7.860 .	676660";	\$ 11.260".	093/ 68	. 0933.	11.860	9767601	07/2601	. 093/8.	.0.93326	. 093.6	696760".	. 032/6.
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00.	. 093/91	. 09332.9		7.665.3	092:769	6032607 .	. 0933/5	1.093112	696760";	. 092.76	. 033530	. 093365	\$6.860.	666760".	092788
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31.	09352.3	.093353	. 093.89	. 092,993	. 092780	. 093539	. 093359	093.95	092988	097.780	. 093567	. 093397/	09322.	. 093023	. 0928.
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Table 2	: Density Dat	ta for Mg and	l Ca-myrista	tes $(30^{\circ} - 50^{\circ})$	C)					
Conc.		Ν	Ag -Myristate	:			(Ca - Myristate	e	
C×10 ⁴	$30^{\circ}C$	35 ⁰ C	40^{0} C	45 ⁰ C	50 ⁰ C	30 ⁰ C	35 ⁰ C	$40^{\circ}C$	45 ⁰ C	50°C
2.0	1.093422	1.093270	1.093112	1.092922	1.092718	1.093427	1.093275	1.092924	1.092924	1.092719
4.0	1.093443	1.093284	1.093127	1.092935	1.092780	1.093451	1.093297	1.092940	1.092940	1.092735
6.0	1.093463	1.093301	1.093141	1.092950	1.092742	1.093475	1.093318	1.092956	1.092956	1.092750
8.0	1.093482	1.093316	1.093156	1.092961	1.092754	1.093498	1.093338	1.092971	1.092971	1.092764
10.0	1.093490	1.093330	1.093170	1.092973	1.092763	1.093521	1.093358	1.092984	1.092984	1.092777
12.0	1.093517	1.093342	1.093181	1.092983	1.092772	1.093542	1.093372	1.092996	1.092996	1.092786
14.0	1.093532	1.093354	1.093192	1.092993	1.092781	1.093555	1.093388	1.093009	1.093009	1.092797
16.0	1.093544	1.093365	1.093201	1.093000	1.092787	1.093573	1.093398	1.093019	1.093019	1.092807
18.0	1.093557	1.093374	1.093211	1.093008	1.092795	1.093590	1.093411	1.093039	1.093039	1.092817
20.0	1.093564	1.093383	1.093217	1.093015	1.092805	1.093598	1.093418	1.093040	1.093040	1.092826

Table 3	: Density Da	ata for Sr an	d Ba-myrista	ntes (30°–50°	C)					
Conc.			Sr-Myristate	e				Ba-Myristate	e	
$C \times 10^4$	30^{0} C	35 ⁰ C	40^{0} C	45 [°] C	$50^{0}C$	$30^{0}C$	35°C	40^{0} C	$45^{0}C$	50^{0} C
2.0	1.093413	1.093263	1.093104	1.092914	1.092710	1.093418	1.093269	1.093111	1.092921	1.092717
4.0	1.093425	1.093273	1.093112	1.092922	1.092716	1.093434	1.093284	1.093126	1.092934	1.092730
6.0	1.093436	1.093283	1.093121	1.092929	1.092723	1.093449	1.093299	1.093140	1.092948	1.092742
8.0	1.093447	1.093293	1.093129	1.092936	1.092729	1.093464	1.093313	1.093153	1.092959	1.092750
10.0	1.093458	1.093301	1.093135	1.092942	1.092734	1.093478	1.093325	1.093164	1.092969	1.092762
12.0	1.093468	1.093308	1.093141	1.092948	1.092740	1.093489	1.093336	1.093175	1.092978	1.092771
14.0	1.093478	1.093315	1.093148	1.092954	1.092745	1.093500	1.093348	1.093186	1.092988	1.092780
16.0	1.093485	1.093321	1.093153	1.092960	1.092750	1.093509	1.093358	1.093195	1.092995	1.092787
18.0	1.093492	1.093328	1.093159	1.092965	1.092754	1.093520	1.093368	1.093203	1.093004	1.092795
20.0	1.093500	1.093331	1.093169	1.092970	1.092759	1.093530	1.093376	1.093215	1.093010	1.092800

Table 4 : Extra from	apolated value Alkali and Al	es of density f kaline earth n	or 50% metha netal myristate	anol + 50% chlo es	oroform solve	nt, ρ _o (g cm-3) as obtained	from p –C plots
Temp.	Lithium Myristate	Sodiam Myristate	Potassium Myristate	Magnesium Myristate	Calcium Myristate	Strontium Myristate	Barium Myristate	Experimental data for ρ _o
30 ⁰ C	1.093408	1.093407	1.093406	1.093398	1.093400	1.093403	1.093399	1.093400
35°C	1.093255	1.093254	1.093255	1.093253	1.093255	1.093250	1.093251	1.093252
40^{0} C	1.093102	1.093095	1.093097	1.093097	1.093099	1.093095	1.093096	1.093095
45°C	1.092909	1.092912	1.092908	1.092907	1.092909	1.092908	1.092908	1.092906
50 ⁰ C	1.092704	1.092703	1.092705	1.092703	1.092706	1.092708	1.092704	1.092703

Table 5:	: Roots c	onstant	s, A and	B as obt	tained f	rom (p –	ρ _o)/c Vs	C ^{1/2} plo	ots.							
emp		Mg- M	Iyristate			Ca- M	yristate			Sr- My	ristate			Ba- M	yristate	
$({}^{0}C)$	A ₁	A_2	$-B_1$	$-B_2$	A ₁	A_2	$-B_1$	$-B_2$	A ₁	A_2	$-B_1$	$-B_2$	A ₁	A_2	$-B_1$	$-B_2$
30 ⁰ C	0.128	0.156	10.201	9.804	0.139	0.181	0.400	0.913	0.072	0.081	0.101	0.260	0.098	0.109	0.550	0.901
35°C	0.098	0.113	8.200	8.001	0.124	0.167	0.390	0.891	0.060	0.073	0.200	0.281	0.092	0.101	0.540	0.801
40 ⁰ C	0.092	0.112	6.02	5.701	0.104	0.122	0.381	1.101	0.049	0.056	0.210	0.370	0.087	0.099	0.420	0.841
45°C	0.087	0.099	5.700	8.101	0.095	0.107	0.500	1.301	0.043	0.047	0.208	0.341	0.080	0.095	0.370	0.911
50 ⁰ C	0.079	0.095	5.401	8.903	0.087	0.099	0.601	1.800	0.037	0.041	0.204	0.336	0.078	0.087	0.401	0.750

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Table 6: Root	s constant	s, A and I	3 as obtair	ned from ((ρ-ρ₀)/C \	vs C ^{1/2} plo	ots					
Temp $\binom{0}{C}$		Li - M	yristate			Na- M	yristate			K- M	lyristate	
Temp. (C)	A ₁	A_2	-B ₁	-B ₂	A_1	A_2	-B ₁	- B ₂	A_1	A_2	$-B_1$	-B ₂
30 ⁰ C	0.105	0.126	0.400	1.051	0.135	0.175	0.701	1.602	0.147	0.195	6.012	12.013
35 ⁰ C	0.096	0.109	0.552	1.000	0.111	0.158	0.300	1.051	0.138	0.186	6.202	9.013
40^{0} C	0.089	0.107	0.500	1.101	0.098	0.113	0.600	1.152	0.132	0.161	8.504	10.500
45°C	0.082	0.104	0.350	1.051	0.073	0.087	0.250	0.700	0.132	0.147	9.501	9.902
50 ⁰ C	0.079	0.097	0.452	1.101	0.070	0.075	0.300	0.451	0.128	0.143	10.510	15.501



(Millero, 1968) has been successfully applied to these solutions to evaluate constants A_1 and B_1 (below the c.m.c.) and A_2 , B_2 (above the c.m.c.) as recorded in Table (5, 6). It is observed that $A_1 > B_1$ and $A_2 > B_2$. The above facts suggested that solute-solvent interactions in the premicellar region are predominant whereas in the post micellar region, solute-sulute (ion-ion) interactions are predominant. It is therefore concluded that micellization just begin at a particular concentration termed as c.m.c.

The data for apparent molar volume (ϕv) for these solutions have been evaluated employing the equation (Shukla, 1982)

$$\varphi_{v} = \frac{M}{\rho_{0}} - \frac{(\rho - \rho_{0})}{C\rho_{0}} \times 10^{3}$$
(1)

where, M, ρ , ρ_0 Signify for molecular weight, density



of solutions, density of solvent, the solution concentration, respectively. Tables (7, 8 and 9) clearly show that apparent volume date increase with increasing surfactant concentration. ϕ_v vs. C^{1/2} plots are also found to intersect in the vicinity of c.m.c. A number of factors *viz.*, hydration of amphiphilic solutes, electrostriction of solvent molecules by charged moieties, nature of the ionic head group and the length of non-polar portion of amphiphilic molecules etc. contribute to apparent molar volume and may thus affect its value to a different extent. Frenks et at. (visser *et al.*, 1977) have however observed a decrease in apparent molar volume, ϕ_v of NaDS beyond the c.m.c.

Root's equation (16) facilitates plots of $(\rho - \rho_0)/c$ vs. C^{1/2} (Fig. 3 showing intersection in the vicinity of critical micelle concentration. The data on limiting apparent molar

Table 7:	ø° v data	for Li, N	la and K	myrista	ntes at di	ifferent t	emperat	tures (30	$0^{0}-50^{0}C)$						
Conc.		L	i-Myrista	ite			N	a-Myrista	ate			K	- Myrista	ate	
(mol	$30^{\circ}C$	35 ⁰ C	40^{0} C	45 ⁰ C	$50^{\circ}C$	$30^{0}C$	35 ⁰ C	40^{0} C	$45^{\circ}C$	$50^{0}C$	$30^{0}C$	$35^{\circ}C$	40^{0} C	$45^{\circ}C$	$50^{\circ}C$
dcm^{-3}															
C×10 ⁴															
2.0	123.47	137.21	137.23	146.40	150.99	115.24	133.55	147.29	156.46	170.22	116.15	125.32	134.48	139.08	143.68
4.0	128.05	137.21	144.09	146.40	150.99	122.10	138.12	151.86	167.90	172.51	118.44	127.57	139.05	145.94	148.26
6.0	129.34	138.73	144.85	149.45	154.05	125.91	139.64	154.91	168.66	173.27	120.72	131.38	143.64	148.23	155.88
8.0	130.33	141.78	146.38	149.83	155.57	126.67	140.41	155.29	170.42	174.80	123.01	132.18	147.60	152.80	160.84
10.0	131.70	144.53	149.12	152.80	158.65	129.87	144.52	159.18	172.02	176.63	125.29	140.87	152.78	159.21	166.56
12.0	132.62	146.36	150.95	155.55	162.44	135.82	153.36	161.77	174.00	178.61	132.15	145.90	158.88	164.24	171.14
14.0	134.58	148.97	155.56	158.81	164.73	138.76	159.68	164.27	176.07	179.38	135.09	149.49	161.92	167.83	173.75
16.0	137.19	150.93	156.10	160.69	167.01	144.96	163.27	167.87	177.50	181.09	141.87	155.05	167.64	171.67	176.86
18.0	139.22	152.96	157.56	164.70	169.30	150.30	168.61	171.17	179.34	181.41	145.11	158.86	171.58	175.17	181.26
20.0	141.76	155.50	159.64	166.07	172.51	154.11	168.76	172.44	182.21	182.58	150.90	164.65	177.02	179.79	185.78

Table 8: ¢° _v data fo	or Mg and (Ca myristat	es at differe	ent tempera	tures (30 ⁰ -	-50^{0} C)				
Conc. (moldm ⁻³)		Μ	g-Myristate				(Ca-Myristat	e	
$C \times 10^4$	30^{0} C	35 ⁰ C	$40^{\circ}C$	45 ⁰ C	50^{0} C	30^{0} C	35 ⁰ C	40^{0} C	45 ⁰ C	50°C
2.0	310.05	328.37	332.99	337.63	342.27	328.33	342.37	360.08	368.65	378.88
4.0	312.33	335.24	337.57	342.49	349.14	335.19	348.96	367.31	374.24	378.89
6.0	314.62	336.00	340.62	343.73	351.42	336.48	351.24	368.07	375.76	380.40
8.0	316.91	337.52	341.00	347.92	352.57	339.76	353.52	369.60	377.67	382.30
10.0	319.19	339.35	342.15	349.53	356.00	341.14	354.90	373.25	380.64	384.37
12.0	321.48	342.10	345.20	352.12	358.92	343.57	360.38	376.46	383.39	388.79
14.0	324.42	344.06	347.37	353.97	359.92	350.54	363.0	378.74	384.69	390.64
16.0	328.34	348.09	350.15	357.07	362.84	352.91	368.39	381.60	387.39	392.60
18.0	330.87	348.69	351.80	358.98	364.14	355.61	371.06	384.34	388.98	394.13
20.0	335.65	350.78	354.95	360.96	365.15	361.25	375.78	385.70	390.80	396.26

Table 9: ø° , data	a for Sr and	Ba myrista	ates at differ	rent temper	atures (30 ⁰ -	-50 ⁰ C)				
Conc.			Sr-Myristate					Ba-Myristat	e	
(moldm^{-3})	30^{0} C	35 ⁰ C	40^{0} C	$45^{\circ}C$	50^{0} C	$30^{0}C$	$35^{0}C$	40^{0} C	45 ⁰ C	50 ⁰ C
C×10*										
2.0	435.90	445.11	454.32	459.18	463.63	458.52	463.15	467.79	472.45	477.11
4.0	438.19	447.40	455.61	459.18	465.91	463.09	467.70	470.09	477.02	479.40
6.0	440.48	448.16	455.85	460.71	465.15	466.14	469.25	472.36	477.02	481.68
8.0	441.62	448.54	456.61	461.47	465.91	467.66	471.15	474.65	480.45	485.12
10.0	442.30	450.60	458.90	462.84	466.46	469.49	474.13	477.86	483.43	486.18
12.0	443.52	452.73	461.18	463.76	467.44	472.99	476.87	479.99	486.17	489.31
14.0	444.39	454.26	461.86	464.41	468.21	475.50	478.18	481.15	487.48	490.84
16.0	446.76	455.97	462.33	464.90	468.78	478.52	480.30	483.80	490.19	493.12
18.0	448.60	456.80	462.96	465.90	469.73	479.86	481.95	486.08	491.25	494.40
20.0	454.19	459.28	463.47	466.50	470.04	481.38	484.19	486.09	493.49	496.78

volume (ϕ_v) are obtained by extrapolating the linear plots of ϕ_v vs. $C^{1/2}$ in premicellar region (dilute solutions) in accordance with the equation by masson,

 $\phi_{v} = \phi_{v}^{0} + S_{v}^{1/2}$

The value for limiting apparent molar volume (ϕ_v°) and experimental limiting slope (S_v) , as recorded in

Table 10, are a measure of solute-solvent and ion-ion interactions, respectively. The ϕ_v^0 and S_v data for these solutions are found to increase with increasing temperature and vary with the metal. The ϕ_v^0 data for alkali metal myristates vary as, Li > K > Na, and for alkaline earth metal myristates as, Ba > Sr > Ca > Mg.

Table 10 : ϕ_v^0 and S_v dat	a for alkali	and alkalin	e earth me	tal myrista	tes at diffe	ent tempe	ratures (30	$0^{0}-50^{0}C$		
Compounds			ϕ^0_v					Sv×10 ⁻³		
Compounds	30°C	35 ⁰ C	40^{0} C	45 ⁰ C	50 ⁰ C	30 ⁰ C	35°C	40^{0} C	45°C	50 ⁰ C
Li- Myristate	302.8	320.0	326.0	329.0	333.5	4.0	5.1	5.4	5.6	5.8
Na-Myristate	319.5	331.5	348.0	365.0	373.5	5.1	5.2	5.3	5.8	6.0
K-Myristate	431.5	442.5	447.0	457.2	458.0	5.9	6.0	6.3	6.4	6.5
Mg-Myristate	452.0	455.0	459.0	465.0	471.0	4.6	4.9	5.2	5.8	6.5
Ca-Myristate	119.5	126.0	132.5	137.2	142.5	5.5	5.8	6.0	6.1	6.2
Sr-Myristate	110.8	127.5	142.5	157.6	165.2	3.0	3.4	3.7	3.8	4.0
Ba-Myristate	170.0	114.5	123.5	132.8	143.0	5.5	5.6	5.7	5.9	6.0

Table 11 : E° _{surf} myrist at diff	data for alk tates in 50% erent temper	ali and all methanol atures (30 ⁰	kaline eart + 50% chl –50 ⁰ C)	th metal oroform
Surfactant		E^0_{su}	rf	
Li- Myristate	1.00	1.20	1.20	1.30
Na-Myristate	2.93	3.00	3.02	3.10
K-Myristate	1.50	1.80	1.86	2.04
Mg-Myristate	1.84	1.90	2.00	2.10
Ca-Myristate	2.30	2.90	3.10	3.50
Sr-Myristate	1.40	1.50	1.70	1.76
Ba-Myristate	0.60	0.80	1.20	1.20



The order of S_v values however varies as: for alkali metal soaps K > Na > Li, and as Ca > Sr > Mg > Ba for alkaline

earth metal soaps. The molar expansibility, E_{surf}^0 (Table 11) is found to increase with increase in electrostriction as well as loosening of solvent structure at higher temperatures. The plots of molar expansibilities and partial molar volume as a function of temperature confirmed that all these compounds appear to be a structure breaker above 35°C which may be due to the fact that at higher temperatures increased thermal agitation does not allow structure formation to an extent detectable by the present technique.

In conclusion, it may therefore be stated that micellar aggregates are formed in the present surfactant systems. The partial molar expansibility data, E^0_{surf} has been evaluated using the temperature dependence of partial molar volume ϕ^0_{v} . It is observed that the increase in E^0_{surf} with increasing temperature may by attributed to a decrease in electrostriction as well as loosening of water structure at higher temperatures.

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