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ORIGINAL CONTRIBUTION

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Infrared absorption spectra of Aerosol-OT homologous sodium dialkylsulfosuccinates and the effect of crystal polymorphism on the environment of the succinate segment

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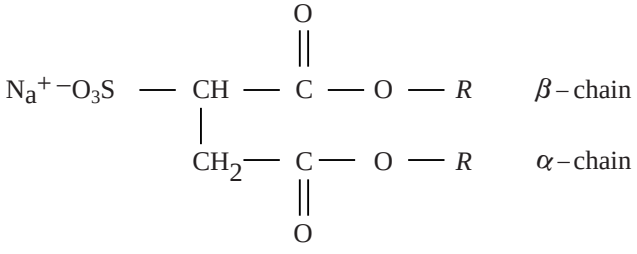
Abstract A homologous series of sodium dialkylsulfosuccinates, which are straight-chain analogues of Aerosol-OT with various chain lengths (dimethyl, diethyl, dibutyl, dihexyl, diheptyl, dioctyl, didecyl, diundecyl and didodecyl), has been synthesized. In order to assign the IR bands of the CH_2 scissoring and CH deformation modes of the succinate segment, sodium dideuteratedmethylsulfosuccinate and sodium dideuteratedethylsulfosuccinate have also been synthesized. IR absorption spectra have been recorded for these mono-, di- and multihydrate samples in the solid state. In particular, IR bands in the 1250–1500 cm^{-1} region have been compared for sodium dimethylsulfosuccinate, sodium diethylsulfosuccinate and sodium diheptylsulfosuccinate, whose crystal structures are known. The CH_2 scissoring and CH deformation modes of the succinate CH_2-CH portion strongly depend upon the environment of hydration and on the length of the hydrocarbon portions.

Key words Crystal polymorphism · Hydration · IR spectra · Sodium dialkylsulfosuccinates · Succinate segment

Introduction

Extensive studies have already been made in order to elucidate the microstructure of sodium 1,2-bis(2-ethylhexyl)sulfosuccinate (Aerosol-OT, AOT) normal and reverse micelles. In particular, it has been recognized that vibrational spectroscopic methods are powerful tools for investigating the chemical structure and the interaction between the counterion and the polar group in self-assembled systems [1–15]. However, in order to discuss quantitatively the behavior of AOT molecules at the interface formed by the aggregates, we need more fundamental vibrational spectroscopic data for very simple AOT homologous sodium di-*n*-alkylsulfosuccinates (SDAS).

The vibrational spectra of AOT consist mainly of the vibrational modes arising from the two 2-ethylhexyl chains, the CH-CH_2 segment of the succinate skeleton,



AOT:*R* = 2-ethylhexyl group

the hydrophilic sulfonate and the two ester portions. It is evident that these modes complicate the vibrational spectrum in the 500–700, 1000–1500 and 1700–1750 cm^{-1} regions. In particular, in spite of its importance in clarifying the microstructure of AOT micelles, the assignment of the characteristic vibrational bands

arising from the CH_2-CH segment of the succinate skeleton of an AOT molecule and its analogues still remains unresolved.

For a number of crystal structures it has been found that water molecules play an essential role in the formation of a three-dimensional network of hydrogen bonds [16, 17] and that the difference in the structure of such a hydrogen-bond network arising from the different hydration numbers brings about a variation in the environment of the hydrocarbon chains of the surfactant molecules [18–20].

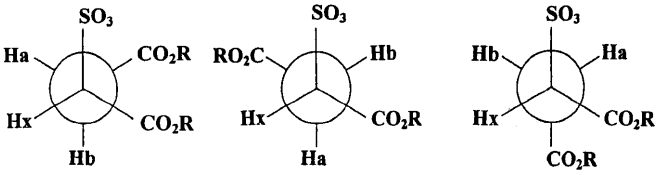
Lucassen and Drew [21] found evidence for crystal polymorphism after recrystallising sodium di-*n*-heptylsulfosuccinate from aqueous solution. Their X-ray powder analysis showed that there is a significant difference in the long spacing between the two (monohydrate and dihydrate) types of crystals. Furthermore, they confirmed the crystal structure of sodium diheptylsulfosuccinate dihydrate [SDHpS($2H_2O$)] by use of single-crystal X-ray diffraction analysis. A detailed vibrational spectroscopic study of such polymorphism should provide information on the nature of the environment of the two hydrophobic chains, the CH_2-CH portion of the succinate skeleton and the polar portions of an AOT molecule.

Recently, we reported the crystal structures of sodium dimethylsulfosuccinate monohydrate [SDMS($1H_2O$)] and sodium diethylsulfosuccinate trihydrate [SDES($3H_2O$)] [22], whose vibrational spectra mainly reflect the structure of the succinate skeleton.

Thus, detailed studies of the vibrational spectra of SDMS, SDES and SDHpS, whose crystal structures are known, should provide important data which will unequivocally define the vibrational spectroscopic data of AOT and its related surfactants.

In this study, the IR spectra of SDAS were mainly examined in the vicinity of the 1300–1400 cm^{-1} region to focus on the effect of hydration. In this range the IR spectra can be easily observed, although the Raman scattering intensity is very weak.

We briefly describe the skeletal structures of SDMS($1H_2O$) and SDES($3H_2O$) determined by single-crystal X-ray diffraction analysis [22] in order to aid the discussion of the relationship between the IR spectrum and the crystal structure. Although three rotational isomers (I, II, III) are possible for the conformations around the C(1′)CH_2-C(1)H single bond (Fig. 1), isomer III is stabilized for SDMS and SDES in the crystalline state and the α chain takes up the extended (trans) form for both compounds. For the conformations around the C(1)-C(2) single bond of SDMS and SDES, the β chain takes up neither the trans nor the gauche form, while around the C(1′)-C(2′) single bond the α chain is fully extended. For SDES there is a significant difference in the C=O bond distance between the α and β chains. Judging from the bond angles of the ester portions, the ester segment is approximately planar. The two ester segments of the α and β chains take up the trans form for both SDMS and SDES.



I II III

Fig. 1 Possible rotational isomers (*I*, *II* and *III*) about the CH_2-CH single bond of a succinate skeleton

Experimental

Materials

SDMS, sodium dideuteratedmethylsulfosuccinate (SDdMS), SDES and sodium dideuteratedethylsulfosuccinate (SDdES) were synthesized as follows. Reactions of maleic acid anhydride with the corresponding alcohol or deuterated alcohol were performed in dried benzene under reflux (328 K) for 4 h in the presence of concentrated H_2SO_4. The dimethyl and diethyl esters of maleic acid thus obtained were distilled at 3 mm Hg (b.p. 330.2–331.1 K for the dimethyl ester and 342.8–346.0 K for the diethyl ester). The purified maleic acid ester was sulfonated with an equimolecular amount of sodium hydrogen sulfite in H_2O at 373 K. The series of longer chain (SDAS) [dibutyl (SDBS), dihexyl (SDHS) and dioctyl (SDOS)], were prepared similarly, and were recrystallized in aqueous methanol. SDHpS, sodium didecylsulfosuccinate (SDDS), sodium diundecylsulfosuccinate (SDUS) and sodium didodecylsulfosuccinate (SDDoS) were prepared according to standard procedures [23, 24]. The amount of hydrated water in each crystalline sample was controlled by changing the growth rate of the crystals and was determined by Karl-Fisher titration (Kyoto Electric Co.). The samples of SDAS(nH_2O) (n = hydration number) were classified into three sample systems: A series, SDBS($1H_2O$), SDHS($1H_2O$), SDHpS($1H_2O$) and SDOS($1H_2O$); B series, SDBS($2H_2O$), SDHS($2H_2O$), SDHpS($2H_2O$) and SDOS($2H_2O$); C series, SDDS($1H_2O$), SDUS($1.3H_2O$) and SDDoS($1.5H_2O$). The samples of SDBS($3H_2O$), SDHS($3H_2O$) and SDHS($10H_2O$) were prepared by adding water to the monohydrate samples. When a sample with a finite hydration number was not obtained as a crystal, a frozen sample (213 K) of the aqueous solution containing a finite amount of water was used. Identification of these sample was confirmed by ^1H and ^{13}C NMR spectra and elemental analysis (C, H and S). All the reactants for preparation of SDMS, SDdMS and SDBS–SDOS were purchased from Wako Chemicals Co., and were purified before use. The reactants for the preparation of SDHpS, SDDS, SDUS and SDDoS were purchased from Aldrich or Avocado Chemicals and were used without further purification. AOT (pure grade) was purchased from Tokyo Kasei Kogyo Co., and was purified according to the method described in the literature [25].

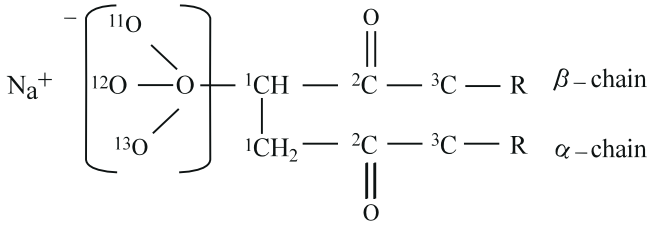
IR absorption and Raman scattering spectral measurements

IR spectra of the sample dispersed in KBr discs were recorded on a Nicolet Magna 750 Fourier-transform IR spectrometer

(4000–400 cm^{-1}) operating at a resolution of 2 cm^{-1} (25 °C). The reproducibility of the Raman and IR band frequencies was 0.01 cm^{-1}. The reported frequencies are accurate to ± 1 cm^{-1} for sharp bands and to ± 2–3 cm^{-1} for broad and weak bands.

Raman spectra below 4000 cm^{-1} were obtained with a Nicolet 950 Fourier-transform Raman spectrometer using the Nd: YAG laser (CVI) excitation wavelength of 1064 nm with a resolution of 4 cm^{-1} at room temperature. The Raman spectra of the solid samples were obtained from pressed solid samples in a capillary tube with a laser power of 400 mW.

Numbering scheme of succinate skeleton



Results and discussion

The IR spectra in the 1250–1500 cm^{-1} region for SDMS($1H_2O$) and SDdMS($1H_2O$) in the crystalline state are shown in Figs. 2a and 3a, respectively. For SDMS, six IR bands at 1348, 1376, 1416, 1439 and 1444 cm^{-1} are observed in this region, while for SDdMS the IR band corresponding to the 1439 and 1444 cm^{-1} bands of SDMS disappears and only three IR bands, at 1357, 1386 and 1418 cm^{-1}, are observed; therefore, the 1439 and 1444 cm^{-1} bands for SDMS obviously come from two CH_3 groups of the ester portions, and may be assigned to the deformation modes of the methyl groups. It is evident that the IR bands at 1348, 1376 and 1416 cm^{-1} for SDMS come from the CH_2-CH segment of the succinate skeleton, and may be assigned to the coupled modes between the CH_2 scissoring and the methyne (CH) deformation modes. The bands at 1357, 1386 and 1418 cm^{-1} for SDdMS correspond to those at 1348, 1376 and 1416 cm^{-1} for SDMS; however, the first two bands of SDdMS are shifted upwards by about 10 cm^{-1} compared with those of SDMS. This shift may be caused by the effect of deuteration of the methyl groups.

The IR spectra in the 1250–1500 cm^{-1} region for SDES($1H_2O$) and SDdES($1H_2O$) in the crystalline state are shown in Figs. 2b and 3b, respectively. For SDES, seven main IR bands at 1342, 1374, 1396, 1422, 1444, 1454 and 1476 cm^{-1} are observed in this region, while for SDdES only three bands at 1370, 1385 and 1421 cm^{-1} are observed, and these correspond to the bands at 1357, 1386 and 1418 cm^{-1} for SDdMS. Thus, the three bands at 1370, 1385 and 1421 cm^{-1} for SDdES may also be assigned to the coupled modes between the CH_2 scissoring and CH deformation of the succinate CH_2-CH segment. The bands at 1444, 1454 and

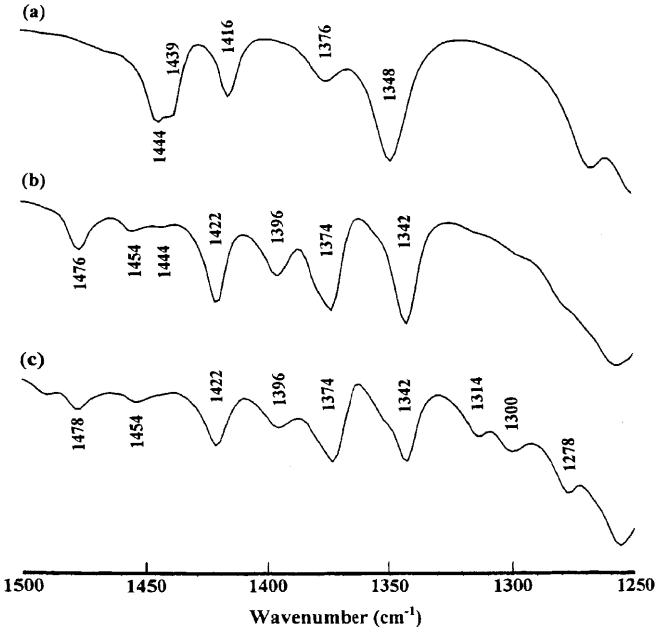


Fig. 2 IR spectra in the 1250–1500 cm^{-1} region of **a** sodium dimethylsulfosuccinate monohydrate, **b** sodium diethylsulfosuccinate monohydrate [*SDES*($1H_2O$)] and **c** SDES($3H_2O$)] in the crystalline state

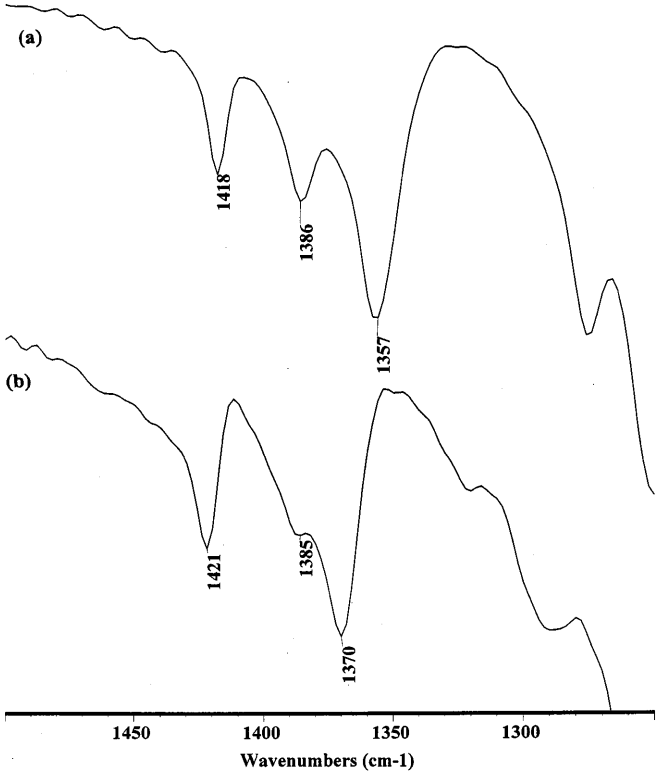


Fig. 3 IR spectra in the 1300–1500 cm^{-1} region of **a** sodium dideuteratedmethylsulfosuccinate monohydrate and **b** sodium dideuteratedethylsulfosuccinate monohydrate in the crystalline state

1476 cm^{-1} may be assigned to the CH_3 and CH_2 deformation modes of the ethyl groups.

In order to elucidate the assignment of vibrational bands coming from the succinate skeleton, we recently carried out the normal coordinate analysis of an SDMS molecule, based on its skeletal structure determined by single-crystal X-ray diffraction analysis [22]. As a consequence of the normal coordinate calculations for the three rotational isomers (I, II and III) (Fig. 1), it was found that the band frequencies of the CH_2 scissoring and CH deformation modes strongly depend upon the conformations about the CH_2-CH single bond of the succinate segment and are located in the 1340–1430 cm^{-1} region. We summarize the assignments of the observed Raman and IR bands of SDMS in the 1300–1500 cm^{-1} region in Table 1 together with the calculated frequencies of isomer III. Excellent agreement between the observed and calculated band frequencies was obtained. It is obvious that the IR band at 1416 cm^{-1} mainly comes from the scissoring mode of the CH_2 group of the succinate segment and the two IR bands at 1348 and 1376 cm^{-1} may be assigned to the deformation modes of the methyne group (>CH-).

The band at 1396 cm^{-1} for SDES may be assigned to the scissoring mode characteristic of the methylene groups (O-CH_2) adjacent to the ester oxygen atoms, since the 1396 cm^{-1} band disappears in the IR spectrum of solid SDMS. Moreover, it closely corresponds to the 1396–1400 cm^{-1} bands observed in common for the series of SDAS monohydrates and dihydrates.

Table 1 Observed IR and Raman bands (cm^{-1}) of crystalline sodium dimethylsulfosuccinate monohydrate [*SDMS*($1H_2O$)] in the 1300–1500 cm^{-1} region and their assignment on the basis of the normal coordinate analysis for isomer III of SDMS

Observed (cm^{-1})		Calculated (cm^{-1})[a]	Assignment[b]
Raman	IR	isomer III	
1466 w	1465 vw	1466	
		1466	$\nu_{as}(CH_3)$
		1465	
		1465	
1444 w	1444 m	1445	$\nu_s(CH_3)$
	1439 w	1445	
1417 w	1416 w	1418	CH_2 sci.
1379 vw	1376 w	1381	C-H def.
1350 vw	1348 m	1351	

[a] The normal coordinate calculations were made using a modified Urey–Bradley force field. The structural parameters of an SDMS($3H_2O$) molecule determined by single-crystal X-ray diffraction analysis were used for this calculation. The force constants used for the calculation have been omitted in this paper, since details of the calculations using isomers I, II and III will be reported separately [26]

[b] ν_{as} and ν_s: asymmetric and symmetric stretching modes, sci: scissoring, def: deformation

The IR spectrum in the 1250–1500 cm^{-1} region for SDES($3H_2O$) in the crystalline state is shown in Fig. 2c. It is now seen that bands at 1278, 1300 and 1314 cm^{-1} appear upon further hydration. Therefore, these IR bands may be regarded as bands sensitive to the environment of hydration. We have also examined the vibrational spectra of SDMS($6H_2O$) (frozen sample); however, they were very similar to those of SDMS($1H_2O$) (spectra not shown).

The IR spectra of longer SDAS monohydrates (A series) in this region are shown in Fig. 4. It is found that the bands at 1344–1346, 1396–1400 and 1418–1420 cm^{-1} are observed in common for this SDAS series and correspond well to the 1342, 1396 and 1422 cm^{-1} bands of SDES($1H_2O$). This observation indicates that the environment of the CH_2-CH segment of the succinate skeleton for the A series is very similar to that for SDES($1H_2O$).

However, it should be noted that the spectral features of the SDHS, SDHpS and SDOS dihydrates (B series) (Fig. 5) in this region differ markedly from those of

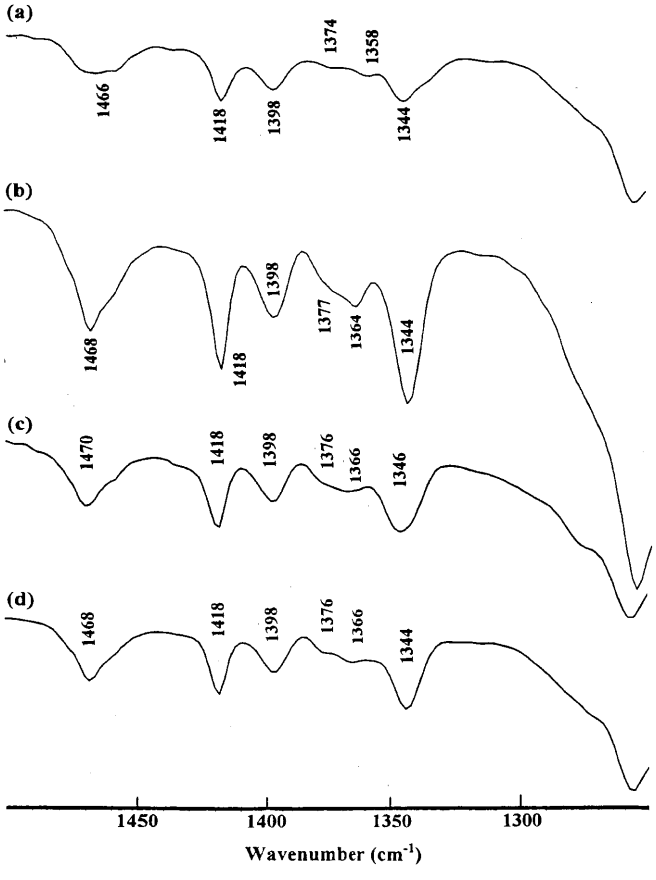


Fig. 4 IR spectra in the 1250–1500 cm^{-1} region of **a** sodium dibutylsulfosuccinate monohydrate [*SDBS*($1H_2O$)], **b** sodium dihexylsufosuccinate monohydrate [*SDHS*($1H_2O$)], **c** sodium diheptylsulfosuccinate monohydrate [*SDHpS*($1H_2O$)] and **d** sodium dioctylsulfosuccinate monohydrate [*SDOS*($1H_2O$)] in the crystalline state

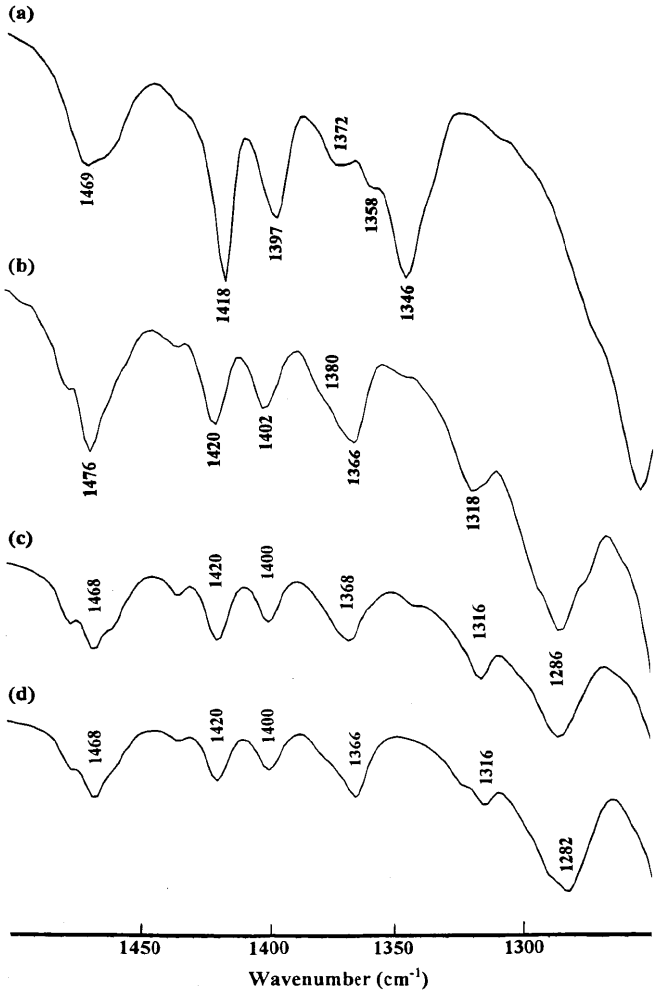


Fig. 5 IR spectra in the 1250–1500 cm^{-1} region of **a** $SDBS(2H_2O)$, **b** $SDHS(2H_2O)$, **c** $SDHpS(2H_2O)$ and **d** $SDOS(2H_2O)$ in the crystalline state

the monohydrates, although the spectral feature of $SDBS(2H_2O)$ is similar to that of the monohydrate. In the IR bands of the B series samples, the bands at 1344–1346 cm^{-1}, observed in common for the monohydrates, disappear or decrease in intensity. Furthermore, the bands at 1282–1286 and 1316–1318 cm^{-1}, which were extremely weak or disappeared in the spectra of the A series, increased in intensity or appeared for the B series; therefore, the 1282–1286, 1316–1318 and 1366–1368 cm^{-1} bands may be regarded as bands characteristic of the dihydrates. This assignment is in keeping with the polymorphic transition between the crystal types arising from the difference in the extent of hydration, and may be accounted for by the difference in the crystal structure between the mono- and dihydrates.

The reason for the similarity in the spectral features of the SDBS mono- and dihydrates may be that the length of the *n*-butyl chain is too short to allow for the crystal polymorphism induced by an increase in the extent of hydration.

Lucassen and Drew [21] have shown that there is a marked difference in the crystal structure of $SDHpS(1H_2O)$ and the dihydrate $SDHpS(2H_2O)$. Their results show that, for crystals of the monohydrate, molecular arrangements which include a tilt angle (38°) between the hydrocarbon chains and the normal to the monolayer planes are possible, while in the dihydrate crystals the hydrocarbon chains have a tilt angle of zero with respect to the monolayer plane normal. They also examined X-ray powder long spacings for various SDAS and showed that in the crystals of $SDHS(1H_2O)$ and $SDOS(1H_2O)$, as well as those of $SDHpS(1H_2O)$, the tilted structure of the hydrocarbon chains may be present. Molecular arrangements with a similar tilt structure have been found for crystals of lysophosphatidylcholine [27] and of cerebroside [28].

Such a difference in the crystal structures of the SDAS monohydrate and dihydrate should bring about a variation in the configuration of the succinate skeleton which induces the difference in the structure of the intermolecular hydrogen-bond network in the hydrophilic layers.

We may use the torsion angles of the succinate skeleton for crystalline $SDMS(1H_2O)$, $SDES(3H_2O)$ and $SDHpS(2H_2O)$, which were calculated by single-crystal X-ray diffraction analysis [21, 22], in order to discuss the origin of the IR spectral variation of the succinate CH_2-CH segment for the SDAS mono- and dihydrates in the CH_2 and CH deformation region. The torsion angles (θ) of the $^{2}C\text{-}^{1}C\text{-}^{1'}C\text{-}^{2'}C$, $^{1'}C\text{-}^{1}C\text{-}^{2}C\text{-}^{3}O$ and $^{1}C\text{-}^{1'}C\text{-}^{2'}C\text{-}^{3'}O$ segments for the R form of each molecule are listed in Table 2.

It has been found that the type III conformation about the $^{1}C\text{-}^{1'}C$ single bond (Fig. 1c) was stabilized for SDHpS as well as for SDMS and SDES. For the $^{1}C\text{-}^{1'}C\text{-}^{2'}C\text{-}^{3'}O$ segment (α chain), the torsion angle for SDHpS is 167.9°, indicating that the segment in the α chain is approximately in the extended form. In particular, it should be noted that the torsion angle of the $^{1'}C\text{-}^{1}C\text{-}^{2}C\text{-}^{3}O$ segment for SDHpS is 17.5°, implying that there is a marked difference in the $^{1'}C\text{-}^{1}C\text{-}^{2}C\text{-}^{3}O$ configuration between the simple SDAS and the longer-chain SDAS dihydrate. We may assume that this configuration (i.e., $\theta = 17.5°$) might be induced by

Table 2 Torsion angles (θ°) of the succinate skeleton for the R form of SDMS, sodium diethylsulfosuccinate (*SDES*) and sodium diheptylsulfosucinate (*SDHpS*)

Segment	SDMS	SDES	SDHpS
$^{2}C\text{-}^{1}C\text{-}^{1'}C\text{-}^{2'}C$	−60.8	−64.7	−62.6
$^{1'}C\text{-}^{1}C\text{-}^{2}C\text{-}^{3}O$	140.4	141.2	17.5
$^{1}C\text{-}^{1'}C\text{-}^{2'}C\text{-}^{3'}O$	−177.0	−168.3	167.9

stacking of the monolayers with close mutual contact between the hydrocarbon and polar groups in adjacent layers. In fact, in this 17.5° torsion angle of the $^{1'}C$-^{1}C-^{2}C-^{3}O segment, it seems that the $\beta C{=}O$ groups are more readily hydrated compared with those in a configuration (about 140°) similar to that seen for SDES. Accordingly, the torsion angle of the $^{1'}C$-^{1}C-^{2}C-^{3}O segment may be changed by a variation in the extent of the hydration of the $\beta C{=}O$ group, leading to the variation in the >CH deformation mode.

Thus, the origin of the 1366–1368 cm^{-1} bands, observed in common for longer SDAS dihydrates, may be due to the deformation modes of the methyne group characteristic of the configuration of the $^{1'}C$-^{1}C-^{2}C-^{3}O segment taking up the 17.5° torsion angle. The bands at 1282–1286 and 1316–1318 cm^{-1} probably come from the CH_2 wagging and twisting modes coupled with the CH deformation mode of the segment taking up such a 17.5° torsion angle.

The IR spectra of SDBS($3H_2O$), SDHS($3H_2O$) and SDHS($10H_2O$) in the same region are shown in Fig. 6. It is found that the IR bands (1282–1286, 1316 and 1366–1368 cm^{-1}) characteristic of the SDAS dihydrates become very weak and broaden with an increase in hydration number, probably due to the torsion angle of the $^{1'}C$-^{1}C-^{2}C-^{3}O segment which may vary as a consequence of an increase in the extent of hydration around $\beta C{=}O$.

We have examined the IR spectra of the C series in the 1250–1500 cm^{-1} region, as shown in Fig. 7a–c. It should be noted that the bands at 1282–1288, 1316 and 1361 cm^{-1} (marked with an asterisk) closely correspond to those at 1282–1286, 1316 and 1366–1368 cm^{-1} for the SDHpS and SDOS dihydrates. This result shows that the appearance of these bands for the C series is likely to be due to the effect of close packing of long hydrocarbon chains, rather than to the effect of hydration, since the samples of the C series are not dihydrates and their hydration numbers are in the range 1–1.5. That is, for SDHpS and SDOS of intermediate chain length, the molecular arrangement of the 17.5° configuration in the monolayer may be induced by the increased hydration number; however, for the C series having long *n*-alkyl

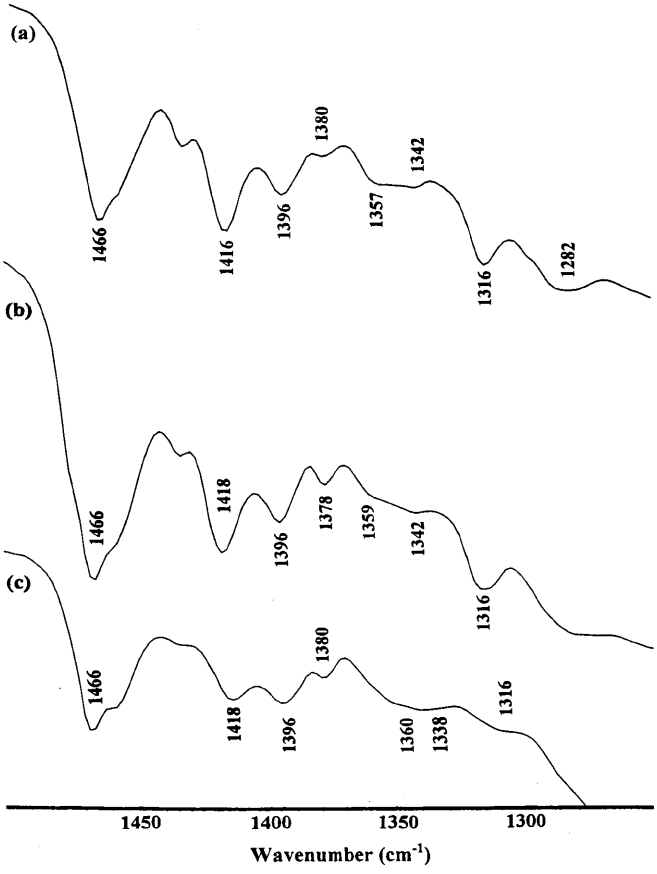


Fig. 6 IR spectra in the 1250–1500 cm^{-1} region of **a** SDBS($3H_2O$), **b** SDHS($3H_2O$) and **c** SDHS($10H_2O$) in the crystalline state

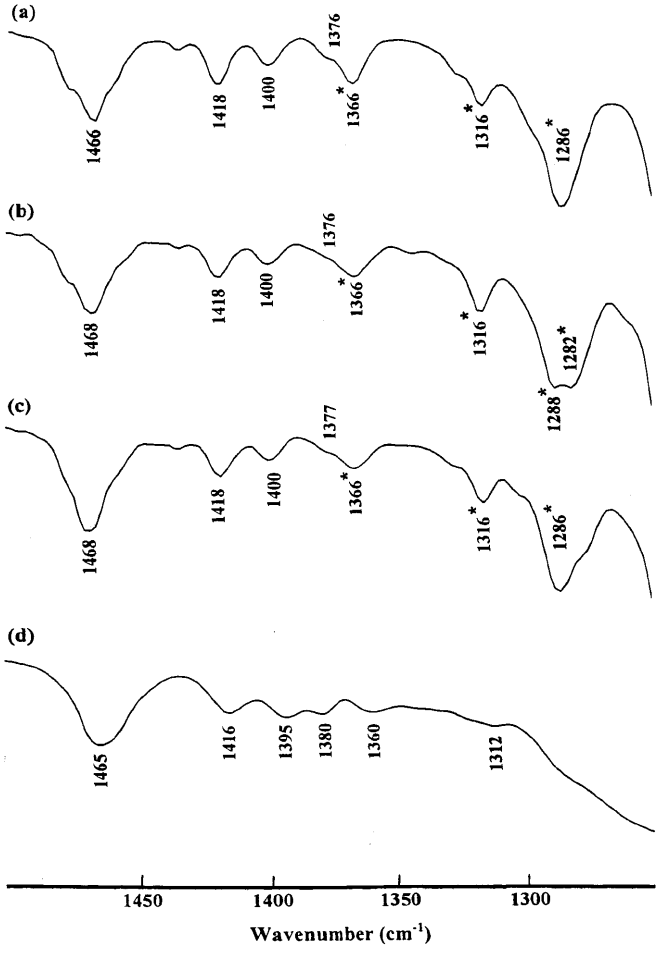


Fig. 7 IR spectra in the 1250–1500 cm^{-1} region of the C series **a** sodium didecylsulfosuccinate monohydrate, **b** sodium diundecylsulfosuccinate 1.3 hydrate and **c** sodium didodecylsulfosuccinate 1.5 hydrate and **d** sodium 1,2-bis(2-ethylhexyl)sulfosuccinate

chains, the close packing of long hydrocarbon portions may induce the molecular arrangement with the 17.5° torsion angle. In order to confirm this idea, we also measured the X-ray powder patterns of these samples, and, as a consequence, it has been confirmed that the untilted structure of the hydrocarbon chains exists in the crystalline state, in spite of the 1–1.5 range of the hydration number [29].

The IR spectra of solid AOT in the 1300–1500 cm^{-1} region is shown in Fig. 7d. Bands at 1360, 1381, 1394 and 1416 cm^{-1} are found, and correspond well to the bands at 1366–1368, 1379, 1400 and 1420–1422 cm^{-1} for the SDAS dihydrate samples, indicating that these bands for AOT come from the CH_2-CH segment of the succinate skeleton. In particular, it should be emphasized that the presence of the 1360 cm^{-1} band implies that the environment of hydration for the succinate CH_2-CH segment of AOT is very similar to that of the SDAS dihydrates.

Conclusion

It was confirmed that the IR bands observed in common for the SDAS series in the 1300–1430 cm^{-1} range come from the CH_2 scissoring and >CH- deformation modes of the CH_2-CH segment for the succinate skeleton. In particular, it has been shown that the bands arising from the CH deformation mode are sensitive to the extent of hydration.

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