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Complexation in the heptanoic acid–heptylamine system

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Abstract The complexation between heptylamine and heptanoic acid has been elucidated at 298.15 K using spectroscopic methods and also by measuring macroscopic quantities such as viscosity, conductivity and surface tension. Fourier transform IR and ^{13}C NMR measurements point towards the existence of a compound consisting of one amine molecule and one acid molecule in an equimolecular mixing ratio. These suggestions are supported by viscosity and surface tension measurements. This compound is further able to interact with excess acid, but similar behaviour is not observed with excess amine. The equimolecular compound behaves like a catanionic surfactant; this is seen in the phase diagram for the heptylamine–heptanoic acid–water system at 298.15 K, where the dominating phase is the lamellar liquid-crystalline phase. This phase is in equilibrium with almost pure water. At low water content a solution phase extending from the binary heptylamine–heptanoic acid axis and covering all mixing ratios between the amine and the acid is also present.

Key words Alkanoic acid · Alkylamine · Catanionic surfactant · Ternary phase diagram

Introduction

Long-chain alkylamines form lamellar liquid crystals in contact with water [1–4]. This formation is induced by the ordering forces of water on amine [2]. Short-chain amines, such as hexylamine, form a lamellar liquid-crystalline phase, D, with water but this phase melts at approximately 291 K and an isotropic solution phase, L, forms [5]. For longer alkylamines the melting point of the lamellar liquid-crystalline phase is higher than 298.15 K [1, 2, 4]. Although amphiphilic in nature, alkylamines do not aggregate in hydrophobic solvents. Self-diffusion measurements in amine–xylene–water solutions gave no evidence for discrete inverse micelles or for separation into polar and apolar regions [5]. In water–octylamine solutions, on the other hand, experimental results point towards the existence of aggregates [4, 6] but the actual structure of the solution is still uncertain. Self-diffusion data seem to support an open structure, continuous both in water and amine [6].

Amines have also been used as cosurfactants in microemulsions [7] and as solubilizates in micellar solutions [8–12]. In micellar solutions of cationic or anionic surfactants, amines induce the growth of micelles.

Interactions between alkylamines and alkanoic acids have been studied extensively by several groups [13–19]. It is generally accepted that alkanoic acids form cyclic dimers (A_2) in pure form or in hydrocarbon solvents [13, 15, 18]. On addition of an amine, proton transfer from the acid to the amine will occur with the subsequent formation of a complex in an equimolecular ratio of acid and amine (A_1B_1). This complex is highly polar and can interact with acid dimers. A new complex consisting of three acid molecules and one amine molecule (A_3B_1) might form [13–16, 18]. In systems containing ternary amines the A_3B_1 complex is the predominating one and as a consequence the viscosity for the binary mixture has a maximum in these systems at an acid mole fraction around 0.75 [13–15]. An exception is trifluoroacetic acid, where the viscosity maximum is shifted towards an equimolecular ratio [15]. This is also the case when the ternary amine is replaced by a secondary or primary one. Saeten et al. [18] found similar behaviour in fatty acid–amine mixtures studied by time domain spectroscopy. They concluded that when small amounts of amine are present in the mixture, the A_3B_1 complex is formed, but on addition of more amine the equilibrium is shifted towards an equimolecular ratio of acid and amine. It must be emphasized that the formation of other complexes in these systems has also been suggested [14, 18].

Ternary systems with alkylamines, alkanoic acids and a third component, i.e. water [2–4], formamide [17] or xylene [20], have also been investigated. The phase diagrams at 298.15 K with water all show similarities on the binary axes. Provided the alkyl chains of the acid and the amine are long enough, a crystalline salt is formed in the binary acid–amine system [2, 4]. The same precipitation is also observed in a system where the carbon atom balance between the acid and the amine has been altered, for example, acetic acid–dodecylamine [3]. A lamellar liquid-crystalline phase emerges from the binary water–amine axis at 298.15 K, except for hexylamine, as mentioned earlier. However, the phase behaviour in the ternary systems depends on the added alkanoic acid. For a practically water insoluble acid, the water content in the lamellar phase is reduced and no other liquid-crystalline phases are present in the system [2, 4]. On the other hand, when a water-soluble alkanoic acid is used, the phase diagram (although not completely determined) looks different and contains both a lamellar and a hexagonal liquid-crystalline phase [3]. In the phase diagrams where water has been replaced with formamide or xylene no liquid-crystalline phases are present. Instead a solution phase connecting the three corners of the phase diagram dominates the system [17, 20].

When an alkanoic acid and an alkylamine are mixed in an equimolecular ratio, the salt formed is called a catanionic surfactant. Catanionic surfactants are compounds consisting of oppositely charged surfactants in an equimolecular ratio where all inorganic counterions have been removed. The term was first introduced by Jokela et al. [21]. Catanionic surfactants show interesting phase behaviour differing from that of the parent surfactants. Much work has been done in the area of catanionic surfactants in the last decade and an extensive review has been written by Khan and Marques [22].

In the catanionic surfactant–water binary system only one liquid-crystalline phase, i.e. the lamellar phase, is present above the Krafft temperature [21]. This phase is in equilibrium with almost pure water over a wide two-phase region [21]. For catanionic surfactants it was found that the Krafft temperature decreased with increasing size of the polar head group while the maximum amount of water increased in the D phase. Emphasis was put on developing a thermodynamic model explaining the stability of the lamellar phase. Since the lamellae in the phase are uncharged the stability was explained with a hydration force type of interaction. The hydration force is a repulsive, exponentially decreasing force with a decay length of about 0.2–0.3 nm [21].

When ionic [23] or nonionic [24] amphiphilic substances are added to the binary catanionic surfactant–water system there is an increase in the water uptake by the lamellar D phase. In the case of an ionic surfactant small amounts will lead to a huge swelling of the lamellar phase while in the case of an alcohol the swelling is moderate. The alcohol is also able to induce the formation of both an inverse hexagonal liquid-crystalline phase, F, as well as an inverse micellar solution, L_2. Ternary systems containing a catanionic surfactant, water and an alkane have also been studied [3, 25, 26].

The study of the heptylamine–heptanoic acid–water ternary system is of fundamental importance since the behaviour of the hexylamine–hexanoic acid–water and octylamine–octanoic acid–water systems at 298.15 K are different. The phase diagram for heptylamine–heptanoic acid–water at 298.15 K has been determined. The heptylamine–heptanoic acid binary system has been studied spectroscopically in order to thoroughly determine the complexation between the acid and the amine. The results of these studies are compared with viscosity, conductivity and surface tension measurements.

Experimental

Materials

Heptylamine (purity above 98%) and heptanoic acid (purity above 99%) were obtained from Fluka (Buchs, Switzerland). They were used without further purification. Water was distilled and deionized immediately before use.

Phase diagram

Samples were prepared by weighing the components into glass vials with screw caps. The samples in the solution regions were mixed by shaking and the samples in the liquid-crystalline regions were mixed by heating, shaking and centrifugation. The samples were allowed to equilibrate in a water bath at 298.15 K for at least 1 week. They were then examined with and without crossed polarizers in order to determine the phase boundary between isotropic solution phases and liquid-crystalline phases.

Viscosity, conductivity and surface tension measurements

The viscosities were measured with Ostwald or Übbelohde viscometers. The conductivities were determined using a Wayne–Kerr bridge with automatic recording of the resistance. The surface tension measurements were performed with a home-made tensiometer, using a du Noüy ring. All measurements were made at 298.15 K.

Fourier transform IR and ^{13}C NMR measurements

The Fourier transform (FT) IR spectra were recorded with a Bruker IFS 66 spectrometer equipped with a DTGS detector. The resolution of the experiments was 4 cm^{-1}. A mountable solvent cell equipped with a spacer was used. The spacer length was 0.06 mm. The ^{13}C NMR measurements were made using a Jeol JNM-LA400 FT NMR apparatus. The measurements were carried out at room temperature.

Care was taken to avoid contamination by atmospheric carbon dioxide in all experiments.

Results and discussion

Phase behaviour

The ternary phase diagram at 298.15 K for heptylamine–heptanoic acid–water (C_7 system) is seen in Fig. 1. It is similar to both the hexylamine–hexanoic acid–water (C_6 system) and octylamine–octanoic acid–water (C_8 system) systems at 298.15 K [3, 4]. As in the C_6 system the acid and the amine are completely miscible and an isotropic solution phase, L_2, extending from the binary acid–amine axis exists. For the C_8 system an increase in temperature to 318.15 K leads to the disappearance of the miscibility gap, demonstrating the correlation between an increase in temperature and a decrease in the number of carbon atoms in these systems [27]. For the C_7 system the amount of water at the phase border in the L_2 phase varies with the ratio of acid to amine. The maximum amount of water is found for pure amine, $w_{water} = 0.33$. This corresponds to about three water molecules per amine molecule, a number coinciding with the hydration number of the amine polar group [28]. Almost the same amount of water, $w_{water} = 0.28$, is found for an acid-to-amine mass ratio of 6:4. This ratio corresponds roughly to four acid molecules to three amine molecules, i.e. a molecular excess of acid. The minimum amount of water is again found for pure acid, $w_{water} = 0.03$. Also for an equimolecular ratio of acid to amine ($w_{amine} = 0.47$ on the binary axis) a relatively small amount of water is found, $w_{water} = 0.07$.

As mentioned, alkylamines are able to form lamellar liquid-crystalline D phases with water at 298.15 K provided the alkyl chain is long enough [1–4]. Heptylamine is the amine with the shortest alkyl chain which still forms a lamellar phase at this temperature. In the heptylamine–water binary system the lamellar phase appears between $w_{water} = 0.38$ and 0.51. On addition of acid the maximum amount of water is continuously reduced. For an excess of acid the D phase is destabilized and the L_2 phase is formed. Besides these two one-phase regions there is an isotropic solution phase on the water–amine binary axis. This phase destabilizes at low amounts of acid.

IR spectra

The most interesting spectral regions in alkanoic acid–alkylamine mixtures are for wave-numbers (σ) between 1800 and 1500 cm^{-1} and between 1000 and 850 cm^{-1} [2, 17, 20]. The broad peak at σ around 935 cm^{-1} caused by the OH out-of plane deformation vibration in the acid is shown in Fig. 2 for different amine contents. This peak is characteristic for the dimeric form and an absence of this peak is fairly good proof for the absence of acid dimers [29]. Also broad, multiple absorption bands from the wagging and twisting vibrations of the

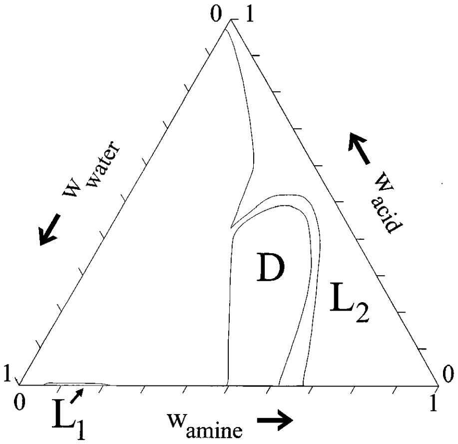

Fig. 1 Ternary phase diagram at 298.15 K for the heptylamine–heptanoic acid–water system

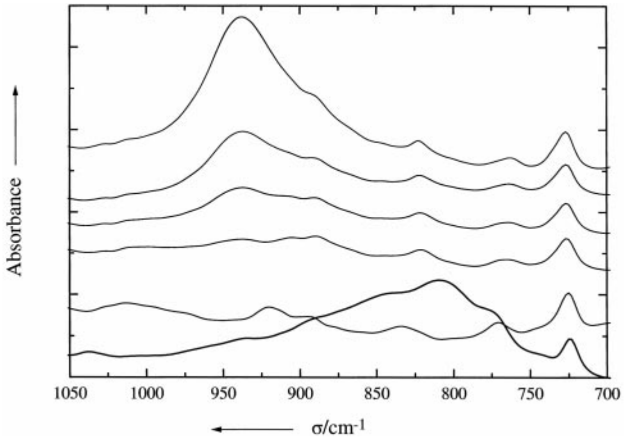

Fig. 2 Fourier transform (*FT*) IR spectra at room temperature for σ between 1050 and 700 cm^{-1} with the mass fraction of amine as a parameter in the heptylamine–heptanoic acid system. The mass fraction of amine is (*top* to *bottom*) 0, 0.05, 0.1, 0.15, 0.47 and 1

NH_2 group in the amine are present in this region for σ between 850 and 750 cm^{-1} [29]. As can be seen qualitatively from Fig. 2 there is a decrease in the dimeric peak at σ around 935 cm^{-1} with increasing amount of amine. This can be understood as a complexation between the acid and the amine, leading to a decrease in the amount of the acid dimer.

A curve-fitting procedure using a least-squares method was done in order to evaluate the absorbance of the dimeric peak. In Fig. 3 the resultant dimeric peak is shown for $w_{\mathrm{amine}} = 0$ and 0.1, together with the experimental spectra. The absorbance of the peak at σ around 935 cm^{-1} as a function of the mole fraction of amine, x_{amine}, is seen in Fig. 4. The absorbance drops quite drastically at small amounts of amine but decreases after that almost linearly and reaches zero at an amine mole fraction of about 0.225. According to Friberg et al. [17] and Saeten et al. [18] a complex A_3B_1 would be predominant at low amine content. This would indicate the absence of acid dimers after an amine mole fraction of about 0.25 if only A_2 and A_3B_1 complexes are present. This value corresponds quite well with our results.

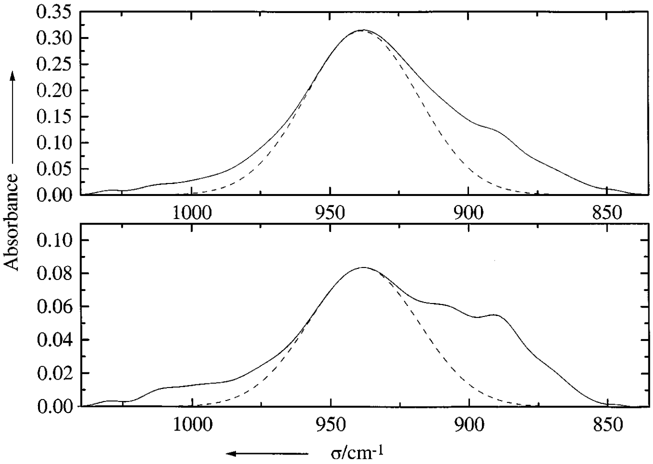

Fig. 3 The fitted (*dashed lines*) OH out-of-plane deformation vibration peak of heptanoic acid together with the measured FTIR spectra (*solid lines*) at room temperature for σ between 800 and 1050 cm^{-1} after a baseline correction, in the heptylamine–heptanoic acid system. The mass fraction of amine is 0 (*top*) and 0.1 (*bottom*)

The spectra for different amine contents for σ in the spectral region between 1800 and 1500 cm^{-1} are shown in Fig. 5. Of special interest is the peak at σ around 1700 cm^{-1}, which originates from the C=O stretching vibration in the undissociated acid. The absorbance of this peak is very high with low amounts of amine, but decreases on further addition and is totally absent for an equimolecular ratio of acid and amine. The addition of amine leads to proton transfer from the acid to the amine and the resulting COO^- group has a strong asymmetric stretching vibration for σ in the region between 1650 and 1550 cm^{-1}. Also the NH_3^+ group has an absorption band for σ between 1625 and 1560 cm^{-1} from the asymmetric deformation vibration and another band between 1550 and 1505 cm^{-1} from the symmetric deformation vibration. The peaks from both the ammonium and carboxylate ions heavily overlap leading to difficulties in making a quantitative analysis. A curve-fitting procedure was also undertaken in this region but with poor results. Qualitatively one can, however, see that apart from the decrease in the absorption peak from the undissociated acid on addition of amine, there are increases in the peaks of the ammonium and carboxylate ions. These peaks have their maxima at an equimolecular ratio of acid and amine, and they decrease on further addition of amine.

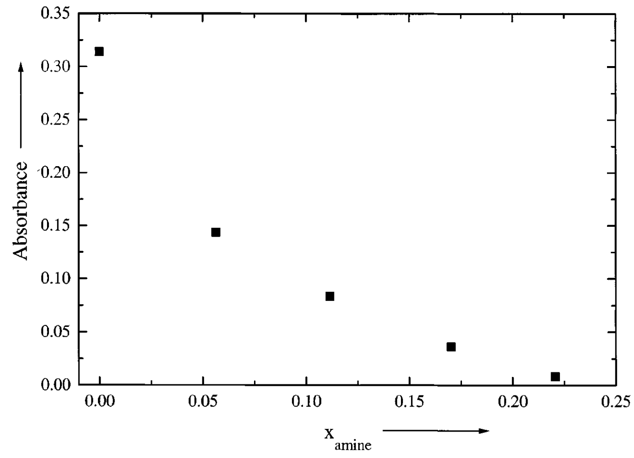

Fig. 4 The IR absorbance at room temperature as a function of mole fraction of amine, x_{amine}, for the OH out-of-plane deformation vibration peak at σ approximately 935 cm^{-1} in the heptylamine–heptanoic acid system

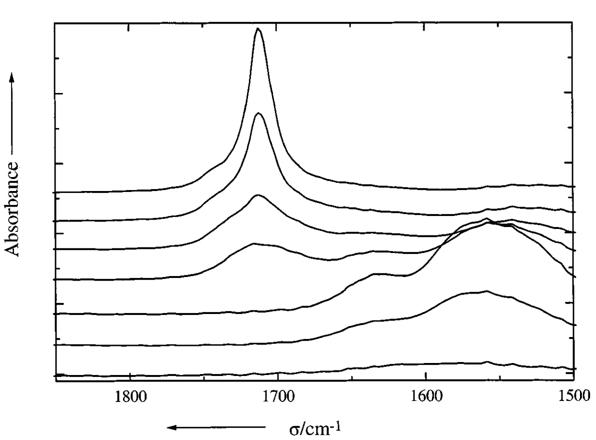

Fig. 5 FTIR spectra at room temperature for σ between 1900 and 1500 cm^{-1} with mass fraction of amine as a parameter in the heptylamine–heptanoic acid system. The mass fraction of amine is (*top* to *bottom*) 0.05, 0.1, 0.2, 0.3, 0.47, 0.7 and 1

^{13}C NMR

The chemical shift for the carbonyl carbon, $\delta_{\mathrm{C,obs}}$, in natural-abundance ^{13}C NMR was followed for the complexation between amine and acid and is seen in Fig. 6 as a function of $n_\mathrm{B}/n_\mathrm{A}$, where n_i denotes the amount of substance *i*. In pure acid almost all the acid is in the dimeric form [30]. On addition of amine there is a linear decrease until $n_\mathrm{B}/n_\mathrm{A} = 0.2$, which corresponds to an amine mole fraction of 0.17. After this there is a plateau for $n_\mathrm{B}/n_\mathrm{A}$ between 0.2 and 0.38 and a subsequent linear increase until an almost equimolecular ratio of acid and amine. For excess amine the chemical shift remains constant.

The chemical shift observed for a nucleus is the weighed mean value of the chemical shifts of the nuclei in different sites according to the equation

$$\delta_{\mathrm{C,obs}} = \sum_i P_i \delta_i \ , \tag{1}$$

where $\delta_{\mathrm{C,obs}}$ is the measured shift for the atom under consideration, δ_i is the shift for the nucleus in site *i* and P_i is the fraction of nuclei in site *i*. Equation (1) is only valid if the exchange of molecules between different sites is fast on the NMR time scale. Assuming only two kinds of complexes to be present at low amine content ($x_{\mathrm{amine}} < 0.2$), acid dimers, A_2, and A_mB_1, where $m = 2$, 3, 4 or 5, the following equation for the chemical shift of the carbonyl carbon can be written

$$\delta_{\mathrm{C,obs}} = P_{\mathrm{A_2}}\delta_{\mathrm{A_2}} + P_{\mathrm{A}_m\mathrm{B}_1}\delta_{\mathrm{A}_m\mathrm{B}_1} \ . \tag{2}$$

This can be rearranged to give

$$\delta_{\mathrm{C,obs}} = m(\delta_{\mathrm{A}_m\mathrm{B}_1} - \delta_{\mathrm{A_2}})\frac{n_\mathrm{B}}{n_\mathrm{A}} + \delta_{\mathrm{A_2}} \ . \tag{3}$$

According to Eq. (3) the plot of $\delta_{\mathrm{C,obs}}$ versus $n_\mathrm{B}/n_\mathrm{A}$ should give a straight line with a slope equal to $m(\delta_{\mathrm{A}_m}B_1 - \delta_{\mathrm{A_2}})$ and an intercept of $\delta_{\mathrm{A_2}}$. If a straight line is fitted in the amine-poor region one gets a slope of −8.554 and an intercept of 180.882. Now the value for $\delta_{\mathrm{A_2}}$ from the linear regression can be compared with the measured one, i.e. 180.949, which is slightly higher. A similar approach can be adapted to the second linear part, for $n_\mathrm{B}/n_\mathrm{A}$ values between 0.38 and 0.89, using the chemical shift obtained for the A_mB_1 complex from the previous regression and assuming that the only complexes present are A_mB_1 and A_1B_1. The best result is obtained for the complex A_4B_1 and results in a chemical shift for this complex of 178.74. When the coexistence of A_2 and A_4B_1 complexes is assumed, there should be no acid dimers left after $x_{\mathrm{amine}} = 0.2$; however, the IR results clearly show the existence of acid dimers up to $x_{\mathrm{amine}} = 0.225$, which indicates that A_4B_1, if it exists, cannot exist with A_2 alone. Furthermore, an A_4B_1 or A_5B_1 complex seems more unlikely than an A_3B_1 or A_2B_1 complex when considering hydrogen bonds and geometric constraints. On the other hand, if one assumes the coexistence of A_2 and A_2B_1 complexes, dimers should be present up to $x_{\mathrm{amine}} = 0.33$. From the IR measurements it is evident that this is not the case.

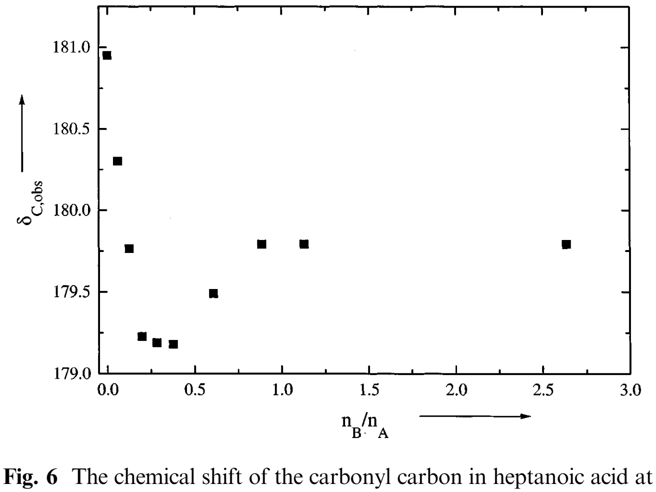

Fig. 6 The chemical shift of the carbonyl carbon in heptanoic acid at 298.15 K as a function of $n_\mathrm{B}/n_\mathrm{A}$, where n_i is the amount of substance *i*, in the heptylamine–heptanoic acid system

Behaviour according to spectroscopic measurements

From the spectroscopic measurements the following can be concluded. Heptanoic acid exists as dimers in the pure form. Addition of amine leads to proton transfer from the acid to the amine and a complex with an equimolecular ratio forms. This complex is, in the presence of excess acid, able to attract acid molecules through hydrogen bonding, thereby disrupting the acid dimers. At low amine content ($x_{\mathrm{amine}} < 0.2$) the existence of A_2 and A_3B_1 complexes seems realistic but at higher amine content ($0.2 < x_{\mathrm{amine}} < 0.5$) several kinds of complexes are present. The breakdown of the A_3B_1 complexes is probably caused by the strong interactions between the A_1B_1 complexes when the amount of these complexes is increased. At excess amine ($x_{\mathrm{amine}} > 0.5$) the A_1B_1 complex is the only form of the acid.

Viscosity and conductivity

For comparison macroscopic quantities, i.e. viscosity and conductivity, were also measured for the heptanoic acid–heptylamine binary system. The viscosity and the conductivity are shown as a function of the mole fraction of amine in Fig. 7. The viscosity has a maximum very close to the equimolecular ratio of acid and amine. This is an indication that the A_1B_1 complexes are able to interact and form a three-dimensional network responsible for the increase in viscosity. This is further seen in Fig. 7 because the viscosity starts to

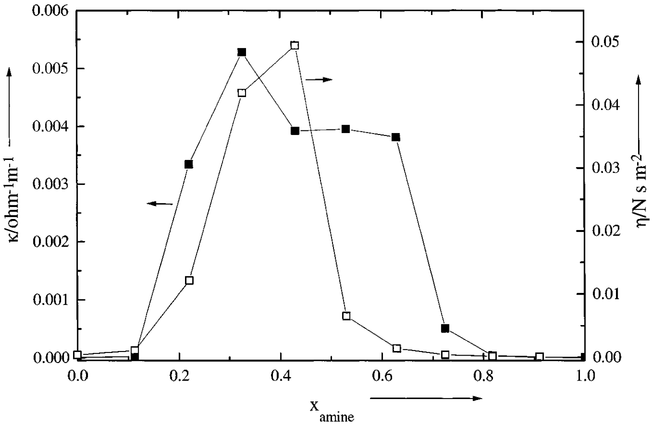

Fig. 7 The viscosity, η, (*open squares*) and conductivity, κ, (*filled squares*) at 298.15 K as a function of mole fraction of amine, x_{amine}, in the heptylamine–heptanoic acid system. The *solid lines* are not a result of a mathematical model but are merely drawn to help visualize the graph

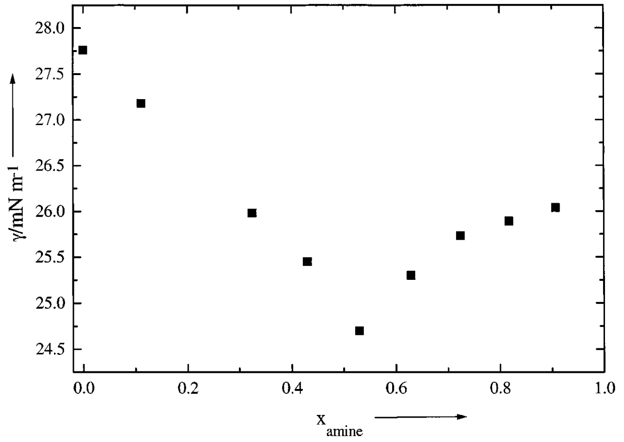

Fig. 8 The surface tension, γ, at room temperature as a function of the mole fraction of amine, x_{amine}, in the heptylamine–heptanoic acid system

increase at approximately $x_{\mathrm{amine}} = 0.20$, i.e. at an amine content where the equimolecular complexes start to form. The conductivity, on the other hand, has its maximum at a somewhat lower mole fraction of amine. This could partly be due to the high viscosity of the liquid at an equimolecular ratio.

Surface tension

The surface tension for the heptanoic acid–heptylamine binary system is shown as a function of the x_{amine} in Fig. 8. The surface tensions for the pure substances are quite similar. A minimum in the surface tension is obtained for the equimolecular mixture. This can be understood if the A_1B_1 complex is considered as a separate compound with a lower surface tension than both the acid and the amine. The curve in Fig. 8 is then merely a composition of two similar curves, one for the heptanoic acid–A_1B_1 system and one for the heptylamine–A_1B_1 system.

Conclusion

The strong equimolecular complex heptanoic acid:heptylamine (A_1B_1) can be considered as a separate compound, i.e. a catanionic surfactant, and the binary system heptanoic acid–heptylamine is thus composed of two binary systems. The first one is the A_1B_1–heptanoic acid system and the second one is the A_1B_1–heptylamine system. The systems differ in that the acid forms complexes with the equimolecular compound while the amine does not. This is evident from the ^{13}C NMR and FTIR measurements. At low A_1B_1 content in the first system, a complex consisting of three acid molecules and one amine molecule is predominant, but on increasing the A_1B_1 content a gradual breakdown of the A_3B_1 complexes occurs due to strong interaction between the A_1B_1 complexes. In this system the existence of different kinds of complexes is probable. In the second system no evidence of complex formation between the amine and A_1B_1 could be detected. The strong interaction between the A_1B_1 compounds is reflected in the high viscosity in the heptanoic acid–heptylamine binary system and a low water–solubilizing capacity in the heptanoic acid–heptylamine–water ternary system for an equimolecular ratio of acid and amine. Further work to characterize the heptylamine–heptanoic acid–water ternary phase diagram is currently being performed.

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