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Occurrence of shear thickening in aqueous micellar solutions of CTAB with some added organic counterions

Received: 30 June 1997
Accepted: 20 October 1997

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Abstract In this experimental work we carefully investigate the influence of some organic counterions (having similarities): sodium salicylate (NaSal), sodium tosylate (NaTos) and sodium benzoate (NaBz) on the rheological properties of two aqueous solutions (0.1 and 0.05 M) of cetyltrimethylammonium bromide (CTAB). Here we are particularly interested in the occurrence of the shear thickening effect corresponding

to shear induced structures (SIS). All the rheological measurements presented in this work are realized with the same geometrical device (plan-cone) with controlled imposed shear stress. Conditions of occurrence and evolutions of the characteristics of the obtained shear thickening are given.

Key words Micellar – rheological – CTAB – salts – shear thickening

Introduction

The size and the structure of ionic micelles in solution depend on various parameters such as the surfactant concentration C_D , the ionic strength, the temperature, ... Structures can also be induced (or modified) under influence of a flow field. The situation is particularly rich and complicated in systems where the interactions between the charged micelles are not or only partially shielded by addition of appropriate amount of salt. It is well known that rod-like (or worm-like) micelles can form in ionic surfactant solutions with sufficient added simple salt [1, 2]. They also form in solutions containing strongly binding counterions such as, for example, salicylate (Sal^-) [3–5] or tosylate (Tos^-) [6].

In pure aqueous solution of CTAB, spherical micelles are present from the critical micellar concentration ($\text{CMC} = 9 \times 10^{-4} \text{ M}$ at 25°C) up to approximately 0.3 M. For greater concentrations the micelles start to grow in size, giving rod-like micelles and viscoelastic solutions. At

a concentration of the order of 0.6 M a nematic phase is obtained. The addition of counterions modifies the evolution of these structures. Cationic surfactant systems of alkyl trimethyl ammonium type RTA^+ (like CTAB) show pronounced viscoelastic behaviour when the salicylate counterion Sal^- is present [7–15]. NMR spectroscopy [16] has revealed that when $C_{\text{NaSal}} \leq C_{\text{CTAB}}$ ($R = C_S/C_D \leq 1$), almost all NaSal dissociate and Sal^- ions form complexes with CTA^+ to form thread-like micelles. Salicylate inserts its benzene ring into the hydrophobic part of the micelle as an intercalation of the salicylate between RTA^+ molecules [9, 17, 18]. For tosylate, Bunton et al. [19] found that the viscosity of a solution of CTAB increases very markedly when NaTos is added. The NaTos (as NaSal) promotes the increase of the length of the micelles. As for NaSal [12, 15, 20] when the Tos^- are in large excess, the rod-like micelles either break up or revert towards spherical micelles and, consequently, the micelles being shorter or more compact, a decrease of the zero shear viscosity is expected and observed. This fluidity at high salt content has been observed for

different systems and was tentatively explained by different processes [8, 16, 21, 22].

For a sufficient concentration of the surfactant and an appropriate added salt, the aqueous surfactant solutions, usually, for sufficiently high shear rates $\dot{\gamma}$, exhibit a shear-thinning behaviour [23–28]. This effect corresponds to a decrease of the apparent viscosity η against the shear rate and is generally interpreted as the consequence of the alignment of rod-like micelles or elongation and stretching of entangled coils (giving also orientational effects).

In other situations, less frequently seen, we can observe, after a Newtonian or eventually a thinning evolution, a relatively abrupt increase of the viscosity between two critical shear rates $\dot{\gamma}_{AB}$ and $\dot{\gamma}_{BC}$ (shear thickening effect). Such an evolution is only encountered with relatively small concentrations of the surfactant and also of the added counterions (such as Sal^- for a classical example). Often the viscosity, before the shear thickening, is of the same order of magnitude as that of the solvent. The pioneering workers in this domain are Hoffmann, Rehage, and coworkers who studied solutions of alkyl trimethyl ammonium and alkyl pyridinium salicylate [29–32]. Concerning shear thickening effect, different explanations of the mechanism involved and of the nature of the obtained phase (the shear induced structure SIS) have been proposed. For example, the SIS may correspond to: an orthokinetic coagulation [3, 33], a flow induced gelation by end-to-end collision [34], a growing of the micelles due to thermodynamic reasons [35, 36], an induced hexagonal liquid crystalline phase or nematic domains [30, 37], a coupling between electrostatic and shear flow [38] or formation of intricate cyclic pattern caused by elastic instabilities [39].

We are concerned here with the linear and nonlinear rheological behaviour (especially the shear thickening) of aqueous CTAB solutions at two concentrations: 0.1 and 0.05 M, which are relatively important in comparison with those used in similar studies. We studied the influence of three organic counterions: NaTos, NaSal, and NaBz at small concentrations, in domains in which it is expected to obtain shear thickening effect. In the case where this phenomenon exists, we give and compare the corresponding rheological curves and the evolution of the characteristics (critical shear rate, amplitude) of this phenomenon against nature and concentrations of the added counterions.

Experimental

Solutions

Products used are commercially available. The solvent was pure water distilled twice in a quartz vessel. The solutions

used in this work were prepared by weighing each compound. The CTAB concentration C_D was fixed at 0.05 or 0.1 M. The concentration C_S of the salts could vary between 0.01 and 1 M. In the study of the occurrence of shear thickening we are essentially interested in situations in which $R = C_S/C_D$ is small. Before utilization, the solutions were left to remain at rest for at least three days in order to reach equilibrium.

Rheology

The rheological measurements presented here were all performed with the Carri-med CSL100 working in the constant shear stress mode. Experimental results seem to indicate that the occurrence of the SIS depends on the geometry of the shearing device (the gap of the cell or the angle of the cone-plane for example) [40]. Consequently, all the experimental results presented in this work were obtained with the same device (to eliminate geometric influence in the studies of the evolution of the characteristics of the shear thickening effect): cône (angle 0.5° ; diameter 4 cm) and plate geometry. A Peltier device allows the temperature of the solution to be adjusted. All the measurements were carried out at 24°C except those corresponding to Fig. 1. All the results reported in this work correspond to experiments at the end of which the solution was apparently in a perfect state, as clear as at the beginning of the measure. For each experiment we have used a new sample of the solution. Results presented in this work correspond to measurements at equilibrium. Only some curves (as indicated on legends) correspond to a scanning time of twenty minutes. The results with such a scanning time (or greater) are like those corresponding to equilibrium measurements as we have verified and shown in a previous paper [41]. With this scanning time we obtain a better definition, with more experimental points on the flow curves.

Results and discussion

Linear behaviour

In Fig. 1 we have represented, in a lin–log coordinates, the evolution of η_0 , the zero shear viscosity, as a function of the concentration C_S of NaSal, the added salt, for three temperatures: 24, 28, and 46°C . The CTAB concentration C_D is here fixed at 0.1 M. As for the CPCl/NaSal system studied by Rehage and Hoffmann [5], the curves for the different temperatures exhibit two maxima. These results can be related to the specific interactions between

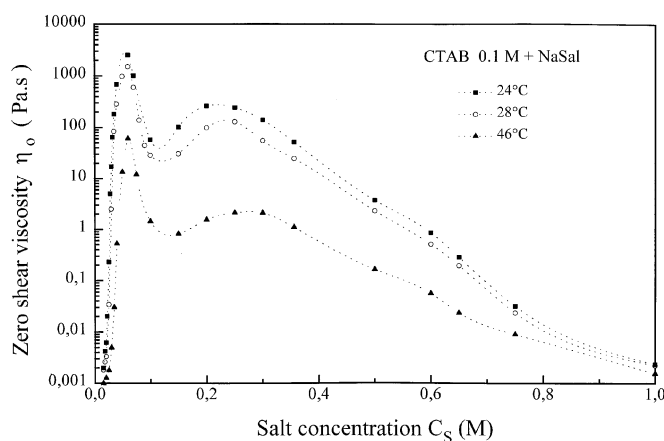


Fig. 1 Variation of the zero shear viscosity η_0 as a function of the NaSal concentration at fixed CTAB 0.1 M concentration and three temperatures: 24, 28 and 46 °C. The lines are only a guide for the eye

the surfactant and the counterion [16]. We can notice that the global evolution and the positions of the extrema of the curves are only a little affected by the variation of the temperature. The value of η_0 is logically, as expected, a decreasing function of the temperature (at the same C_S). If we concentrate our attention on the curve corresponding to 24 °C (the only temperature used in the rest of this work) we can note several points. In the first part of the curve (small values of C_S , of the order of 0.01 M) η_0 is small and close to the one of the solvent. In the next domain (II), η_0 rises very abruptly up to a first maximum corresponding to $C_S \approx 0.06$ M ($R \approx 0.6$). Then the zero shear viscosity decreases to a minimum ($C_S \approx 0.1$ M, $R \approx 1$). Thus equimolar concentrations of the surfactant and salt give here a minimum (and not a maximum as it is sometimes observed [16]). More increasing C_S induces a new increase of η_0 leading to a second maximum for $C_S \approx 0.2$ M ($R \approx 2$) inferior to the first one. Finally η_0 decreases up to approximately the viscosity of the water when C_S reaches 1 M ($R = 10$). For $T = 24$ °C, in Fig. 2, we have studied comparatively the influence of the nature and the concentration of the three added counterions (NaSal, NaTos and NaBz) on two CTAB solutions (0.05 and 0.1 M). The partial experimental results presented in this figure show that the η_0 value is a strong function of the nature of the salt (even if the structures of the counterions are similar in some aspects), of the CTAB and salts concentrations. For the CTAB 0.05 and 0.1 M solutions we can always notice that for same C_D and C_S : $\eta_0(\text{NaSal}) > \eta_0(\text{NaTos}) > \eta_0(\text{NaBz})$. The results concerning the different solutions presented in this figure indicate that, at small (≈ 0.01 M) or at high (1 M) concentrations of salt, all η_0 are of the same order of magnitude (approximately as that of water).

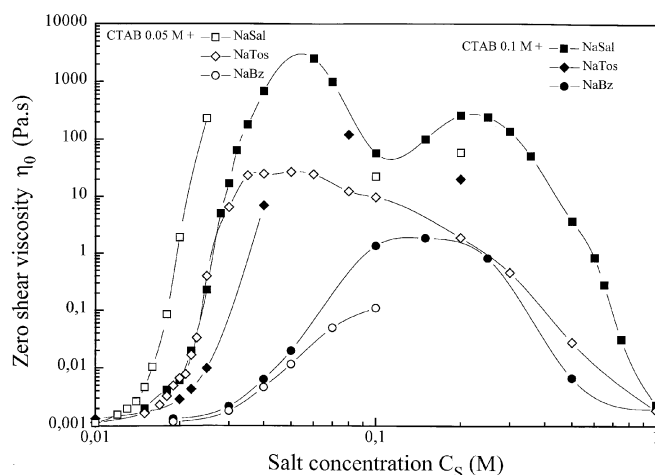


Fig. 2 Some results concerning the variation of the zero shear viscosity η_0 (for the three counterions: NaSal, NaTos and NaBz) versus concentrations C_S at two CTAB concentrations

But, between the extreme values the evolution for the three counterions are very different. The $\eta_0(\text{CTAB } 0.05 \text{ M}/\text{NaTos})$ curve can be decomposed into four domains. In the first domain, η_0 is close to the one of the solvent alone (distilled water). For the corresponding concentration range ($C_S \leq 0.01$ M) it is expected that the micelles are not sufficiently elongated to induce strong interactions between them. The relatively small micelles do not overlap and can freely rotate. In the next domain (II) ($0.02 \text{ M} \leq C_S \leq 0.03 \text{ M}$), the η_0 value increases abruptly.

Due to the increase of C_S , the electrostatic repulsion between the charged polar head of the surfactant molecules decreases, so the growth of the length of the rod-like micelles is facilitated. When the particules are sufficiently large they can strongly overlap, giving a network exhibiting a large η_0 viscosity. Then, in the third domain (III), when C_S lies between 0.035 and 0.06 M, η_0 is relatively insensitive to the salt content: the zero shear viscosity is nearly constant (there exists however a maximum value of η_0 at $C_D \approx C_S = 0.05$ M, $R = 1$). Bunton et al. [19] have also observed an evolution of η_0 with only one maximum, at $R = 1$, with the system CTAB 0.025 M/NaTos, $T = 25$ °C. We can notice that for this system, in comparison with CTAB 0.1 M/NaSal, the two maxima (and the minimum) have disappeared. In the last domain (IV), for large amount of salt, η_0 is a strong decreasing function of the concentration of NaTos as it is found in other systems. The reason of this evolution remains to be satisfactorily explained without ambiguity as mentioned before. For $C_S = 1$ M ($R = 20$), the obtain viscosity is again near that of water, the solvent.

An even more regular evolution, with one maximum is obtained with NaBz, as it can be seen for the CTAB

0.1 M/NaBz in Fig. 2. The viscosity η_0 is smaller in this case. Similar evolutions have been reported for similar systems [42]. If we compare the η_0 values for the same low salt concentrations (before the first or unique maximum) but with $C_D = 0.1$ or 0.05 M we can notice that two very different behaviours are obtained. For the sodium benzoate in this salt domain (more over, as in all the 0.01 – 1 M salt domain investigated): $\eta_0(\text{CTAB } 0.1 \text{ M/NaBz}) > \eta_0(\text{CTAB } 0.05 \text{ M/NaBz})$. On the contrary, for NaSal and NaTos, in the first part of the salt domain (II) of the curve η_0 , increasing CTAB concentration from 0.05 to 0.1 M induces a decreasing value of the zero shear rate viscosity. As we will see later, it is only with these two counterions, in a part of this domain of salt concentration, that we can observe the shear thickening effect. For higher salt concentrations in particular after the maximum of η_0 , we have verified the existence of the more logical behaviour: η_0 for fixed salt concentrations increases with increasing CTAB concentration from 0.05 to 0.1 M (as it can be seen in Fig. 2 from experimental points for $C_S = 0.2$ M e.g.). Detailed study of the influence of the surfactant concentration will be the subject of a further paper [43].

Nonlinear behaviour

In Fig. 3, the evolution of the apparent viscosity η against the shear rate γ is represented in a lin-log representation for the lower concentrations of NaTos. We notice a well-defined shear-thickening behaviour when C_{NaTos} lies between 0.015 and 0.022 M. We can decompose the curves into three parts A, B, C. In A, the first one, for low values of the shear rate, the viscosity is constant ($C_{\text{NaTos}} < 0.020$ M) or decreases with a slope increasing with C_S . This part ends at a critical shear rate γ_{AB} . Between this critical shear rate and γ_{BC} , a second one, the viscosity increases (part B). The third part C corresponds to the decrease of η when $\gamma > \gamma_{BC}$.

We can notice that the phenomenon of shear thickening occurs in the salt concentration domain (II), at the beginning of the abrupt increasing part of the zero shear viscosity curve (Fig. 2). For $C_S < 0.02$ M, shear rates smaller than γ_{AB} are insufficient to orient significantly the micelles. So in such situations an approximately Newtonian evolution (part A) is observed. For greater salt concentrations, such as 0.022 M, the salt induces elongation of the micelles permitting the particles to have a sufficient axial ratio to be oriented by shear rates smaller than γ_{AB} . So the first part A of the curve exhibits a shear thinning effect, the apparent viscosity decreasing as a consequence of orientational (and eventually deformational) effects of the shear rate. For sufficiently great shear rates,

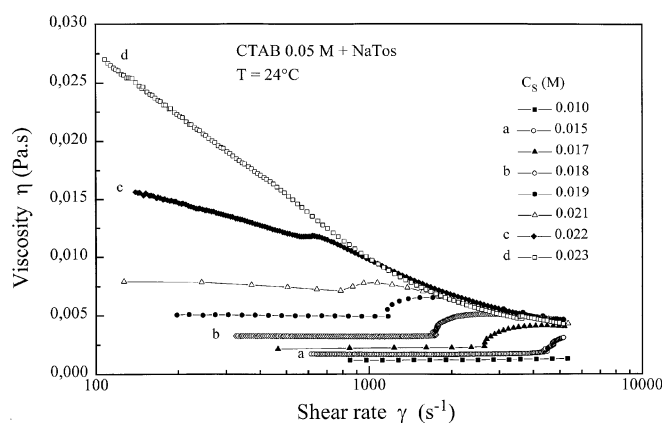


Fig. 3 Apparent viscosity η as a function of shear rate γ for solutions of CTAB 0.05 M and different salt (NaTos) concentrations giving shear thickening (curves a, b, c and d in 20 min with a logarithmic stress sweep, the others at equilibrium)

the particles in the flowing solution can grow, with alignment and eventually fusion building a structure having a higher shear viscosity. This can be the explanation for the occurrence of the shear thickening. In steady state measurements, we can notice the existence of experimental points after γ_{AB} and before the maximum of η . The stable structure can be reinforced in a domain of shear rates up to γ_{BC} . For greater shear rates, $\gamma > \gamma_{BC}$, the more important effect of γ can be a dominant orientational effect (with eventually a decrease of the length of the micelles) giving a decrease of the apparent viscosity as it is seen in Fig. 3 (more or less important, depending on the salt concentration). For C_S less than 0.015 M (i.e. 0.01 M), we only see a Newtonian evolution, η is constant in all the accessible shear rate domains. Perhaps there is a shear thickening effect but it will occur for a greater shear rate that our rheometer cannot reach. However, when C_S is very small, the shear thickening effect must disappear because without salt, at such low concentration of the surfactant, the micelles are spherical and consequently induced structures are highly improbable.

To compare the relative effect of NaSal and NaTos we have represented in Fig. 4 the results obtained with the system CTAB 0.05 M/NaSal for small concentrations of NaSal. We observe a shear thickening effect if the concentration of salt lies approximately between 0.010 and 0.017 M. The needed minimum value of C_S is smaller for NaSal. This shows that NaSal is more efficient. We can notice that here in the first part A of the curves $\eta = f(\gamma)$, η is always constant whatever the concentration of NaSal. We can also remark with this system that the only domain of concentrations of NaSal giving shear thickening corresponds to the beginning of the part of the $\eta_0 = f(C_{\text{NaSal}})$

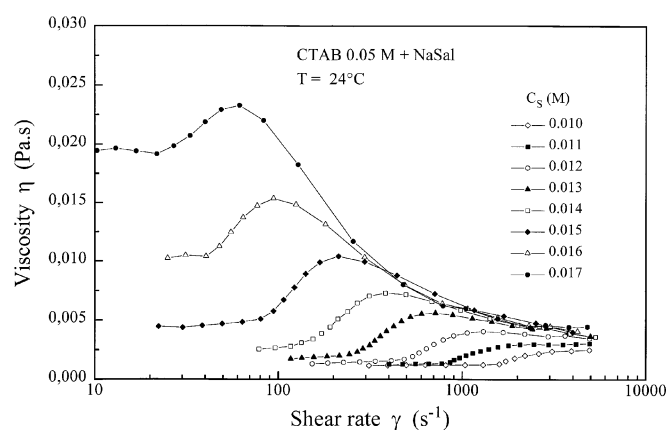


Fig. 4 Apparent viscosity η as a function of shear rate γ for solutions of CTAB 0.05 M and different concentrations of NaSal giving shear thickening (equilibrium curves)

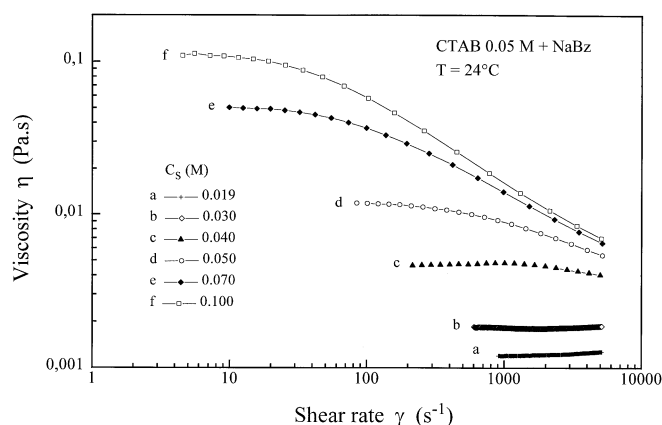


Fig. 5 Apparent viscosity η as a function of shear rate γ for solutions of CTAB 0.05 M and different concentrations of NaBz (equilibrium curves except curves numbered a and b corresponding to a logarithmic stress sweep in 20 min)

curve (Fig. 2) in which the η_0 viscosity increases sharply (domain II).

If we compare the results obtained for the the same concentration of CTAB ($=0.05$ M) and the same concentrations of NaSal and NaTos, we can see that NaSal leads, for $C_S > 0.010$ M, to a higher value of η_0 . For the value 0.010 M the η_0 value for the two salts are nearly the same, but shear thickening is only observable with NaSal in the range of γ accessible with our rheometer. For $C_S = 0.018$ M, the η_0 for NaSal is more than one order of magnitude greater than that for NaTos and shear thickening effect has disappeared with NaSal. All these results indicate unambiguously that NaSal creates longer micelles at rest. These micelles are easier aligned and can interact to form the SIS at a γ_{AB} smaller than the one necessary for NaTos. With the two CTAB solutions we have also investigated the influence of sodium benzoate, being particularly interested by the eventual occurrence of shear thickening. We have carefully investigated the domain of salt corresponding to the increasing part of the $\eta_0 = f(\text{NaBz})$ curves (Fig. 2), the part giving shear thickening effect for NaSal and NaTos. We have never been able, with NaBz, to induce SIS giving shear thickening in the accessible shear rates range. Examples of obtained results are given in Fig. 5. The flow curves are Newtonian or exhibit a classical shear thinning effect.

Finally we have investigated the CTAB 0.1 M/NaTos system in the domain of salt concentration where the shear thickening was expected to occur. We have not been able to observe the phenomenon with this system. Concerning the occurrence of the shear thickening, the necessary conditions to observe the effect, from experimental results obtained in this work, are difficult to give. What we can say is that when we have obtained shear thickening, the

zero shear viscosity was relatively low, between 10^{-3} and 10^{-1} Pa.s and the domain of salt concentration corresponds to the beginning of the sharply increase of $\eta_0 = f(C_S)$. Furthermore, it was necessary that $C_S < C_D$ (more precisely in this study R lies between 0.15 and 0.44). It was shown that the nature of the salt (in particular with the same benzene ring), the structure and polarity of the head of the counterion play a major role in the possible formation of the SIS.

In Fig. 6, we have summarized the results concerning the critical shear rate γ_{AB} at which the shear thickening occurs. A linear decrease of γ_{AB} (in a log–lin representation) is observed in all cases. It can be explained by the fact that when C_S increases, the micelles grow in length (by a change in the packing parameter and a reduction of the surface charge). Interactions and orientations of micelles are easier and so smaller values of γ_{AB} are needed to create supermolecular structures.

If we observe the results concerning NaSal and two CTAB concentrations we notice that the domain of salt where exists the shear thickening behaviour, is shifted to greater values of C_S when the concentration of the surfactant increases. With the same salt and at identical C_S concentrations, $\gamma_{AB}(C_D = 0.05 \text{ M}) < \gamma_{AB}(C_D = 0.1 \text{ M})$. Since with less CTAB (and same C_S) there exists less repulsive electrostatic interactions between micelles, the particles can approach other micelles easier and so it needs a smaller critical shear rate γ_{AB} to allow the creation of the SIS.

In Fig. 7, we have represented the jump of viscosity occurring between γ_{AB} and γ_{BC} . The amplitude A_η of the shear thickening is defined as $A_\eta = \eta_{\max}/\eta_{AB}$, η_{AB} being the viscosity at the shear rate γ_{AB} . With the system CTAB 0.05 M/NaSal, when the concentration of the salt NaSal

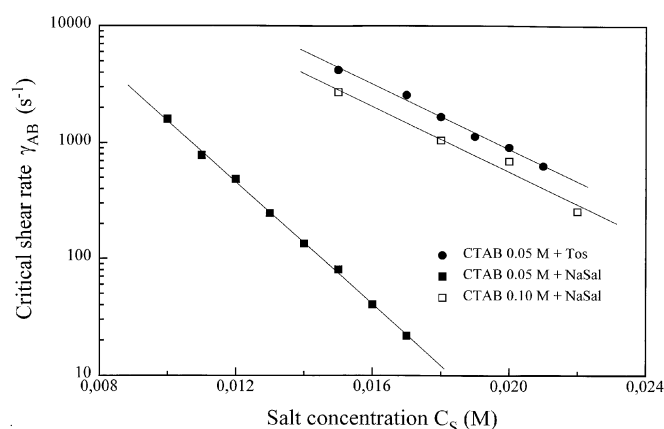


Fig. 6 Critical shear rate γ_{AB} (shear rate at which occurs the shear thickening effect) versus the concentration of the added salt for three micellar systems: CTAB 0.05 M/NaTos, CTAB 0.05 M/NaSal and CTAB 0.1 M/NaSal ($T = 24^\circ\text{C}$)

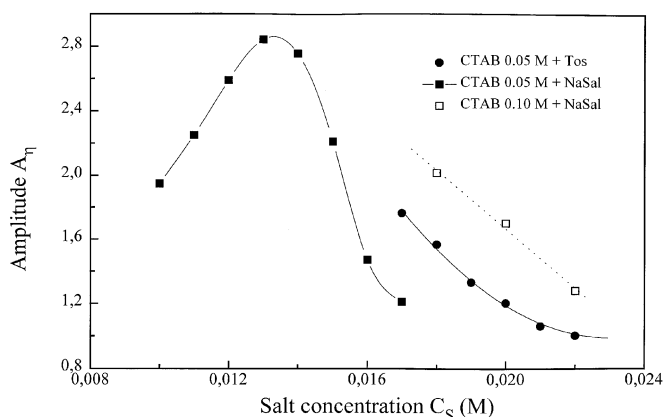


Fig. 7 Amplitude A_η of the shear thickening effect versus the concentration of the added salt for three micellar systems: CTAB 0.05 M/Tos, CTAB 0.05 M/NaSal and CTAB 0.1 M/NaSal ($T = 24^\circ\text{C}$). The lines are a guide for the eye

increases, A_η shows a maximum value for $C_s \approx 0.013\text{ M}$ and then decreases to disappear for too high added salt concentrations. For the other two systems, we can only observe a decreasing evolution. Probably the shear thickening effect still exists for smaller salt concentrations (superior to a threshold value) but we cannot see this

behaviour because the corresponding critical shear rate γ_{AB} is too high to be reached with our rheometer.

Conclusion

In this experimental work, we have carefully studied the linear (zero shear viscosity η_0) and the nonlinear rheological behaviour of aqueous micellar solutions of CTAB with added organic counterions. The effects of three salts, the sodium salicylate (NaSal), the sodium tosylate (NaTos) and the sodium benzoate (NaBz), have been investigated. We have been particularly interested in this study in the conditions of occurrence of shear thickening. Two concentrations of CTAB (0.05 and 0.1 M) were used. The three added anions Tos^- , Sal^- and Bz^- have some similarities: they have a benzene ring and a volume of the same order of magnitude but, different polar heads. In our experimental conditions, with NaBz we only observe a shear thinning behaviour. For the two other salts we have found a shear thickening effect only in a very narrow range of the concentration of the salt. So the effect exists only in very restricted situations with only a small amount of added salt. So restrictive conditions can explain why the shear thickening effect is not often seen in experiments. For these solutions, the zero shear viscosity η_0 is still small (in general less than 10 or 20 times the viscosity of water). Furthermore, the salt concentrations for which the phenomenon exists correspond to the beginning of the very sharp increase domain of the zero shear viscosity. But as it was shown, the influence of the shear rate in these situations is not always sufficient to create the SIS. Subtle electrostatic interactions must also exist to permit the building of the structure by an adequate coupling of such electrical effects and the orientational influence of the shear flow.

More experimental studies, with selected micellar systems and various geometrical devices, with the useful support of other rheophysical techniques, must be performed to hope a complete understanding of the mechanism and structures involved in the different situations. Simultaneously, a theoretical approach, with an appropriate model, must be conducted to clearly understand the shear thickening effect.

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