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Shear Thickening of an Aqueous Micellar Solution of Cetyltrimethylammonium Bromide and Sodium Tosylate

V. Hartmann and R. Cressely (*)

Laboratoire de Physique des Liquides et des Interfaces, Groupe Rhéophysique des Colloïdes, Université de Metz, 1 boulevard F. Arago, 57070 Metz, France

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Abstract. — In this experimental study we give results concerning the shear thickening behavior exhibited by an aqueous cationic surfactant solution of CTAB in the presence of added NaTOS. We carefully investigate the rheological characteristics of this phenomenon corresponding to a shear induced state (SIS), the reproductibility and also the transient regimes. We show that with steady experiments there exist no hysteresis effect. Finally we give the evolution of the shear thickening characteristics against temperature.

1. Introduction

It is well-known that under appropriate conditions, surfactant molecules can assemble with a reversible process to form various structures in solutions. One possibility is to obtain one-dimensional aggregates (rodlike or wormlike) which present similarities with polymeric system [1,2]. These systems are known as "living polymers". In more concentrated system it is possible, sometimes, to obtain mesophases like nematic or hexagonal, lamellar (bilayer) structures or disordered bicontinuous structures. Often it is possible, as indicated in the phase diagram, to pass from one structure to another by varying the temperature, the concentration or for example by addition of a cosurfactant or a salt. Shear flow can also induce, in an isotropic solution at rest, structures or transitions to another structure.

For a classical cationic surfactant intensively studied, the CTAB (cetyltrimethylammonium bromide) in water, increasing concentration gives spherical micelles at the CMC (critical micellar concentration) and after, elongated micelles, more or less flexibles, which leads near 0.7 M (at 25 °C), to a nematic phase followed by an hexagonal one. At low concentration of CTAB, the addition of a simple salt (like KBr, potassium bromide) facilitates the growing of the micelle, giving a wormlike micellar system which has particularly at rest, good analogies with semi-diluted polymer solutions [1,2]. It is well-known that addition of an organic compound like NaSal (sodium salicylate) is even more effective in promoting the growing of the micellar size. This dramatic increase of the length and appearance of entanglements correspond to

(*) Author for correspondence (e-mail: Cressely@pli.univ-metz.fr)

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a very important increase of the zero shear viscosity \( \eta_0 \) (of several order sometimes). These solutions ordinarily give a shear thinning effect for sufficiently high shear rate, i.e. a decrease of the apparent viscosity of the solution \([3,4]\). The origin of this effect is mainly in the orientation of the elongated particles under shear.

For surfactant solutions, in some less frequently encountered situations, it is possible to observe an increase of the apparent shear viscosity at a critical shear rate \( \gamma_{AB} \) in a relatively small interval of shear rate. Such behavior is called shear thickening and is related to a shear induced state (SIS). It has been observed especially for dilute micellar solutions and for the first time by Hoffman et al. \([5]\): they describe a thixotropic effect, the viscosity rising with time. This was explained by a shear induced structure. During the last decade some experimental \([4,21]\) and theoretical \([22-25]\) attempts have been made to describe and explain the shear thickening effect. If some progress has been realized, obviously a lack of complete experimental results explains the difficulties encountered for a good understanding of the phenomenon.

The two domains of questions which are posed are: what exactly are the shear induce structures that are formed with the shear thickening, and how are they created? Some researchers have tried to give an answer to these and related questions.

Rao et al. \([6]\) have proposed for the CTAB + NaSal system that the micelles form necklace type strings under shear which become so long that they can be aligned under shear. Hoffmann et al. \([7]\) had envisaged this hypothesis before they used the kinetic coagulation mechanism. They proposed that the micelles acquire enough energy to overcome the repulsive forces between them. When the micelles touch they may stick together. They may coalescence and form some kind of supermolecular structure during flow. This SIS can only grow in the direction of flow. With increasing shear rate two different processes can occur. It is either possible that more micelles undergo coalescence or the supermolecular structure can be stretched in the direction of flow. When the flow is stopped, the large molecules break and return to their initial form. This could explain results found later \([8-10]\).

We can see a variety of explanations for the nature of the SIS in the literature. It might consist of very long micelles (e.g. with the system CTAB + NaSal \([8]\) and for others \([7,9-12]\)), of a network \([11,13]\), of a kind of liquid crystal (for example hexagonal for CODMABr (hexadecyloctyldimethylammoniumbromide) solution \([9]\), nematic for TDMAO (tetradecylidimethylaminoxide) + SDS (sodiumdodecylsulfate) system \([9,12]\)) or it might correspond to a phase transition (e.g. with solution of CPyCl (cetylpyridinium chloride) + NaSal \([15]\)). More recently, by direct imaging of the scattered light microscopy technique, Liu and Pine \([22]\) have observed shear-induced gelation in extremely dilute solutions of wormlike micelles (CTAB/NaSal system). This gelation is followed by a fracture of the gel which produces extremely elastic gel bands moving with different velocities and in different directions after the flow is stopped. These structures are unstable.

For the mechanisms of formation of the SIS different approaches have been proposed. Turner and Cates \([23]\) have suggested a colinear aggregation: two micelles can fuse under shear only if they are colinear. The system is pushed by shear from the sol into the gel state. Bruinsma et al. \([24]\) consider the effect of shear rate on the size of rodlike micelles in dilute and semidilute solutions and they proposed an orthokinetic coagulation. This phenomenon does not require attractive interactions. Here the Brownian motion is not the dominant transport mechanism: the aggregates are due to purely hydrodynamic interactions. Shear flow prevents the establishment of thermal equilibrium, it will increase the number of particle-particle collisions. For these two descriptions of the mechanism involved, the longer micelles and the better shielded electrostatic repulsive forces ought to support the formation of SIS. Wang \([25,26]\) thinks that the micelles can grow for thermodynamic reasons. So, a definitive explanation (in all situations) concerning SIS is not at present available.
The aim of this paper is to give for the classical cationic surfactant cetyltrimethylammonium bromide (CTAB) in the presence of sodium tosylate (NaTOS), strongly adsorbed on the micellar structure (like in the case of NaSal), the rheological behavior corresponding to steady measurements, but also the transient response of the system in the growing new state of equilibrium corresponding to the shear thickening phenomenon. The reversibility of the rheological behavior with the shear rate (hysteresis) is also studied. Lastly, the variation of the parameters characterizing the shear induce thickening with the evolution of the temperature is carefully investigated.

2. Materials and Methods

2.1. Products. — Products used in this work are commercially available. CTAB (cetyltrimethylammonium bromide) and NaTOS (sodium tosylate) were purchased from Acros. The solvent was pure water distilled twice in a quartz vessel. We prepare the used solution by weighing appropriate amounts of each compound (water, CTAB, NaTOS), so concentrations are given in % by weight or mole per kg. Then we mixed them in a glass vial. The solution was left to stand at least three days in order to reach equilibrium. The CTAB concentration $C_D$ was fixed for this study at 0.04 M (1.46%) and $C_S$, the one of NaTOS, at 0.017 M (0.33%).

2.2. Rheology. — The Carri-med CSL100 working in the constant shear stress mode was used to measure the rheological properties presented in this work. We utilized a cone and plate geometry (4 cm diameter, 0.5° cone angle) which allowed measurement of the shear rate $\gamma$ between approximately 0.1 and 5000 s$^{-1}$. A Peltier device allows the temperature of the solution to be adjusted. The sample was studied in the temperature range of 20 to 30 °C. To prevent errors due to evaporation, the measuring geometry was surrounded by a solvent trap containing water. 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. If it is not specially specified in the text, all the measures reported in this work correspond to a fresh solution, not previously presheared. If it is not mentioned, the rheological curves correspond to measurement at equilibrium. This is important to notice because the curve must be notably different if the scanning time is too short. Remarks concerning this problem will appear later in this text.

3. Results and Discussion

3.1. Shear Thickening. — In Figure 1 the evolution of the apparent steady shear viscosity $\eta$ (applied shear stress $\sigma$/equilibrium shear rate $\gamma$) against $\gamma$ is shown in a log-log representation for the CTAB 0.04 M and NaTOS 0.017 M at 24 °C. Each point of the curve is obtained from the $\gamma = f(t)$ curves (given later) and corresponds to the equilibrium value. We observe a very well-defined shear thickening behavior. When $\gamma$ is low, below the critical value $\gamma_{AB}$, the viscosity is constant and is equal to the zero shear viscosity $\eta_0$ (Newtonian domain, called part A). After this critical shear rate and below a second one $\gamma_{BC}$ (this domain corresponds to part B), the viscosity increases abruptly up to $\eta_{max}$, maximum of the viscosity. We notice that there exists experimental points in this part of the flow curve. So we have no discontinuity in the flow curve. When $\gamma$ is larger than $\gamma_{BC}$, shear thinning occurs: $\eta$ decreases linearly, in a log-log representation, with a slope equal to $p$ (part C). In the insert of Figure 1, the steady viscosity against the applied shear stress is shown in a lin-lin representation. We have here limited the domain studied but increased the number of experimental points to have a precise representation for the part B, in which the viscosity increases.
To test the reproducibility of the results, we have repeated these measures more than 20 times. We always obtain a similar regular curve exhibiting the shear thickening effect. Nevertheless the characteristics of the phenomenon ($\eta_0$, $\eta_{\text{max}}$, $\gamma_{\text{AB}}$, $\gamma_{\text{BC}}$, $p$) can vary a little from one measurement to another. If part C of the curve is apparently nearly the same, with a good approximation, the bigger difference appears for the value of $\eta_0$, corresponding to part A of the flow curve. We notice that this domain of measurement corresponds to the smaller imposed shear stress and that, with the small viscosity involved in these measurements, the sensitivity of the rheometer is worse. We need indicate that if we impose a smaller shear stress, apparent oscillations appear at the beginning of part A of the curve, this phenomenon is a rheological artefact of the measurement. Differences in the $\eta_0$ value obtained could come from a parasitic effect such as the small difference in the amount and centring of the solution put on the plane of the cone-plate device of the rheometer. We can see later, in Figure 4, that even in this domain A, if we do not change the sample, the reproducibility is good. Representations of experimental incertitude zones for the viscosity and critical shear rates are indicated in Figure 1. The preceding considerations concern curves corresponding to steady measurement. We have also done experiments with different continuous scanning of the shear stress (the viscosity is not yet the steady viscosity). The scanning can be linear or logarithmic. By varying the time of scanning we can observe differences with the curve obtained with equilibrium values. We have noticed that if the time is too short (less than approximately 10 min) the viscosity curve is strongly affected (see the curve 5c studied later). Increasing the time of scanning decreases the differences with steady measurements. The first part (A) of the curve below $\gamma_{\text{AB}}$ can show an appearance of shear thinning (more important if the scanning time is shorter). With our results, we estimate that the measurements made during 20 minutes (or longer), with a linear or logarithmic scanning, give practically same results as equilibrium values.

Fig. 1. — Steady state viscosity against shear rate in logarithmic coordinates. In the insert is reported the variation of the steady viscosity versus shear stress in a linear coordinates.
Hu et al. [8, 14] have found steady curves of the same type for the much less concentrated system CTAB/NaSal (the concentrations are of the order of the mM). For their solutions [14], the viscosity in part A is not clearly represented. In their measurements [8], the slope of the domain A depends on the temperature. For higher temperatures the evolution appears more Newtonian. We can also see a decrease in the first part of the viscosity curve in others systems such as 0.01 M CTAB-salicylic acid [13] or 30 mM CPyCl/NaSal [15]. In other cases a Newtonian domain A is obtained, for example for C_{14}TASal [16], C_{14}DMAO + SDS [7], TTASal (tetradecyltrimethylammoniumsalicylate) [11], TDMAO + SDS [12], and a system called 12-2-12 (dimeric surfactant ethanediyl-α, ω-bis(dodecyl dimethylammonium bromide) [17]. When the shear thickening effect exists, we can see, in the literature, experimental points [7,11–13,16,17] or not [8,14,15,18] in the Part B (where the viscosity rises abruptly with the shear rate). Sometimes, one can not obtain the part C [7] or after a maximum the viscosity stays approximately constant [18]. We notice that often the curves found in the literature are relatively badly defined, with irregularities. Regular curves, with a good representation of the three different domains such as those obtained with our system are exceptions in experimental shear thickening studies.

3.2. TIME EFFECT. — In micellar shear thickening studies, it is often noticed that a relatively long time is necessary to obtain equilibrium values. This time is a function of the system investigated, of the concentration, of the shear rate or shear stress applied. We have noted some examples: approximately 100 s for 10 mM TDMAO/SDS solutions and γ = 50 s⁻¹ [10], 240 s for 0.9 mM CPySal and γ = 50 s⁻¹ [5], 2000 s for 5 mM CTASal and γ = 0.046 s⁻¹ [19], 3200 s for 0.6 mM CTASal and γ = 0.99 s⁻¹ [20]. With our system, we have studied the transient regime of γ for imposed shear stress giving experimental points in the different parts of the flow curve of the Figure 1 given before. Figure 2 gives examples of the evolution of the shear rate against time. We have applied a step function of the stress at t = 0 s. We can notice that for shear stress σ below that giving the shear thickening, i.e. in the part A, corresponding to σ ≤ 3 Pa (as it can be seen in the insert in the Fig. 1), a steady shear rate
Fig. 3. — Shear rate growth. A constant shear stress $\sigma = 6$ Pa was applied at time $t = 0$ and removed at $t = 60$ s (curve 1) during a time of 5 s giving $\gamma = 0$. Curve 2 corresponds to the shear rate growth when $\sigma = 6$ Pa was again applied (for the same conditions as before). Curve 3 corresponds to the next result, using the same procedure as mentioned for the two curves 1 and 2.

The value is reached monotonically in a relatively short time, less than twenty seconds. Figure 2 shows that also for high shear stress (for example 18 Pa), corresponding to a point in the well-defined shear thinning domain (part C) of the flow curve, a similar evolution is obtained, the steady value being also obtained relatively quickly. For intermediate values, within the range $3 \text{ Pa} < \sigma < 14$ Pa (part B and beginning of the part C), the evolution is more complicated. There is an initial more or less important overshoot of the shear rate, followed eventually (only in the part B) by a small number of damped oscillations. In these situations, the time $t_\infty$ necessary to reach the equilibrium shear rate is more important (of the order of 40 s). We notice that the curve obtained for $\sigma = 6$ Pa (and showing the more pronounced oscillations) corresponds to a point in the growing part (near the end of part B) of the flow curve. In this domain a shear induced state is building and the evolution corresponds to the creation of the induced structure. Figure 2 shows that in all situations, equilibrium values of the shear rate $\gamma$ are obtained in a maximum time of the order of forty seconds. These times are notably smaller than longer times mentioned in some works (cited previously), but it must be noticed that our surfactant solution is much more concentrated than the millimole concentration order often encountered in previous work. To be sure that the value of $\gamma$ obtained after a time of the order of one minute is the equilibrium value, specially in the part B of the curve where relatively sharp transition in the growing viscosity occurs, we have prolonged the time during which we imposed the shear stress up to an hour. The value of the corresponding $\gamma$ remains very approximately the same as that obtained after one minute. In particular, for points just after the beginning of the increase of viscosity (in B) we do not obtain with increasing time (more than an hour) a transition giving viscosity near the one of $\eta_{\text{max}}$ corresponding to the complete shear induce state.
Fig. 4. — Shear rate growth. A constant shear stress $\sigma = 2$ Pa was applied at time $t = 0$ and removed at $t = 60$ s (curve 1) during a time of 5 s giving $\gamma = 0$. Curve 2 corresponds to the shear rate growth when we applied $\sigma = 8$ Pa (same conditions as before). Curve 3 corresponds to the result when, using the same procedure as previously, we applied 2 Pa. In curve 4, the same method for 8 Pa was applied.

It is also interesting to study the stability (during the time) of the shear induced structure after stopping the shear flow. First, as shown in Figure 3, we have chosen to apply a constant shear stress equal to 6 Pa (corresponding to part B) during one minute with a fresh solution and then we stop the shear flow. When $\gamma = 0$, we wait for 5 seconds and then we apply again 6 Pa during one minute. We repeat this procedure six times with the same solution. We remark that all the curves are practically the same. For clarity, we have represented only the first three curves in the Figure 3. We have chosen 6 Pa because with this shear stress we obtain a viscosity which is near the maximum of the viscosity, at the end of the part B, when the SIS is practically fully obtained. So we have created a shear induced state when we apply 6 Pa during one minute. We have made the same experiment with different shear stress corresponding to different points in part B and also with 8 Pa which give a viscosity near $\eta_{\text{max}}$ but in the part C. We have obtained identical results: the different curves are practically superimposed. Strivens [13] has done approximately the same experiment (two-5 min shearing periods separated by different recovery period of time) with 0.01 M CTAB/salicylic acid solution. He saw that 150 seconds recovery time is required for the sample to return to its original state after the removing of the imposed shear. Hu et al. find a necessity of 300 s for the solution of CTAB/NaSal (1 mM equimolar) [8] and 100 seconds for the 10 mM TDMAO/SDS solution [10]. We can notice that these times are much higher than our value of 5 seconds.

Another procedure has been used to confirm that the induced structure (or its effect on the transient response) disappears a few seconds after the cessation of the flow. For that, a shear stress of 2 Pa (giving a viscosity in the Newtonian domain) is applied during one minute. This curve (numbered 1 in Fig. 4) is the reference curve since it was obtained with a fresh solution (not presheared). At the end of the first step, we stop the shear flow, keeping the solution in the rheometer. We wait again for 5 seconds after the stop of the cell. Then we applied 8 Pa
during one minute (curve numbered 2). The structure is completely induce at the end of this step. Again, after 5 s we impose again 2 Pa during one minute (curve 3). We stop 5 s and we imposed again 8 Pa during one minute (curve 4). We have represented these results in the Figure 4. We notice that the curves which have been made at the same constant imposed shear stress are superimposed. We can conclude that after creating the SIS and after an interruption of 5 s of the flow, the fluid has found again its initial state and the SIS has totally disappeared, without memory effect. If we applied again an appropriate value of the shear stress, we find again the SIS with the same evolution (and with the same zero shear viscosity). It would be interesting to study the disappearance of the structures on shorter times, however with our rheometer, with its inertia (the viscosity is very small) it is unrealistic to study it for times less than a few seconds. The preceding curves (Figs. 2 and 3), from an another point of view,
show the good reproducibility of the results for a same solution even after different preshearing procedures. In the Figure 5, we have looked for a possible hysteresis effect. For these surfactant systems, showing shear thickening, this phenomenon was never studied to our knowledge in the literature.

In this figure we can compare the evolution of the apparent steady shear viscosity for an increase (curve 1) followed (without changing the solution tested) by a decrease (curve 2) of the imposed shear stress. We have also reported results concerning for the stress decrease (curve 3) an experiment in which a fresh solution (not presheared) was used, the Newtonian domain being reached at the end. We can notice that all of the three curves presented in Figure 5a, within the experimental incertitude domains, are superimposed. The conclusion is that with measurements at equilibrium there is no hysteresis effect. The results are independent of the sense of the scanning of the stress with a fresh solution as with a solution submitted to the cycle of an increase followed by a decrease (or the inverse) of the shear stress. If the cycle of scanning is made in a finite time, an apparent hysteresis is clearly visible on the flow curves. The importance of the effect is most important for the shorter time of scanning of the stress (e.g. 5 or 10 min). The hysteresis effect practically disappears (Fig. 5b) for sufficiently long time of scanning (e.g. 20 or 30 min). The apparent hysteresis effect is evidently a consequence of the finite time necessary needed to reach the equilibrium value: during the scanning of the shear stress, the transient measured value is not the same when the stress increased or decreased, giving an apparent hysteresis effect (e.g. in the Fig. 5c).

3.3. Temperature Effects. — In Figure 6, we show the evolution of the shear thickening phenomenon with temperature for the same solution of CTAB 0.04 M and 0.017 M NaTOS. We have represented in this figure the steady shear viscosity at various temperatures between 20 and 30 °C. Individually, for each temperature, the flow curves are similar to the one presented before. We can see that the shear thickening exists in this range of temperature and is shifted
to higher shear rates with increasing temperature. On the last curve, at 30 °C, we can not
detect the phenomenon any more but only a Newtonian evolution. By continuity we can think
that perhaps the shear thickening effect exists, but for a greater shear rate, inaccessible with
our device and rheometer. A qualitative evolution of the curve is the “rounded” transition (at
\( \gamma_{AB} \)) occurring when the temperature decreases. We have measured all parameters of Figure 1:
\( \eta_0, \eta_{\text{max}}, \gamma_{AB}, \gamma_{BC}, p \). They describe the evolution of the shear thickening occurring in this
system. We give a synthesis of these results in Figures 7, 8 and 9.

In Figure 7, we have plotted the zero shear viscosity \( \eta_0 \) and the critical shear rate \( \gamma_{AB} \) against
the inverse of the temperature \( T \) (K) in a semi-log representation. We remark that \( \eta_0 \) increases
and \( \gamma_{AB} \) decreases linearly when \( 1/T \) rises. Thus, we can calculate an activation energy \( \Delta E \).
We find \( \Delta E \approx 98 k_B T \) for \( \gamma_{AB} \). Previously, we have found a value of 99 \( k_B T \) [4] with a similar
system (0.1 M CTAB and 0.022 M NaSal). However, these values are appreciably greater
than the 21.5\( k_B T \) given by Hu et al. [8] for a much less concentrated equimolar system of
CTAB/NaSal (1, 2, 5 mM). The origin of the difference could be found in the strong difference
of concentration and ratios of CTAB/NaSal in the different experiments. For \( \eta_0 \), we find an
Arrhenius behavior with \( \Delta E \approx -40k_B T \). The evolution of \( \eta_0 \) with the temperature is well-
known: with increasing temperature the size of the particles decreases and consequently so does
the zero shear viscosity. If the occurrence of shear thickening is a consequence of alignment
of the particles with the shear flow (which allows the building of structures), logically this
orientation is more difficult for smaller particles, and so \( \gamma_{AB} \) increases with temperature. Our
experimental results are in agreement with this conclusion.

The amplitude \( A(= \eta_{\text{max}}/\eta_0) \) and the difference between \( \gamma_{BC} \) and \( \gamma_{AB} \) are represented against
\( T \) in Figure 8. We notice that \( A \) and \( \gamma_{BC} - \gamma_{AB} \) both rise approximately linearly with \( T \) in the
investigated temperature domain.

A careful study of the evolution of part C for the different temperatures shows that in the
thinning domain the evolution of the viscosity can be represented by a power law character-
ized by the slope \( p \) of the linear evolution in a log-log representation. In Figure 9, we have
plotted the measured values of \( p \) against the temperature \( T \). We can notice that when \( T \) rises
\( p \) increases from \(-0.42 \) to \(-0.27 \). These values are far from the value of \(-1 \) which correspond

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**Figure 7.** Zero shear viscosity \( \eta_0 \) and critical shear rate \( \gamma_{AB} \) versus \( 1/T \) in a semi-log representation.
Fig. 8. — Amplitude $A (= \eta_{\text{max}}/\eta_0)$ of the shear thickening effect and difference of the critical shear rates ($\gamma_{\text{BC}} - \gamma_{\text{AB}}$) as a function of the temperature $T$.

Fig. 9. — Slope $p$ of the shear thinning part of the flow curve against the temperature $T$.

to the presence of a shear plateau ($\sigma$ being constant) against the shear rate, the situation observed at higher concentrations of the products CTAB and/or NaTOS [21] (or in CTAB/NaSal system [4]).
4. Conclusion

In this experimental work we have given precise and critical rheological results concerning the shear thickening exhibited by an aqueous micellar solution of CTAB/NaTOS. The exact nature of the shear induced state and the mechanisms involved are not at the present time completely elucidated. The appearance of the growing viscosity is accompanied by anisotropic effects in the sheared micellar solution. Optical measurements (flow birefringence for example) and small angle neutron scattering under shear can give valuable information about the shear induced structure. Such measurements are in progress and will be presented in a further paper.

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