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ORIGINAL CONTRIBUTION

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Rheological studies of freely suspended soap films

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Abstract We have developed an original viscometer that allows the determination of the viscoelastic properties of freely suspended soap films. We have found that soap films have a very high shear viscosity and a small shear modulus that might be associated with a glassy (gel-like) behavior. The incorporation of polymer chains inside the soap films does not change very significantly these viscoelastic properties that are governed by the dense packing of the surfactant in the interfacial films; nevertheless, the role of the polymer can be qualitatively described by scaling laws.

Key words Surface viscoelasticity · Soap films · Polymers · Confinement

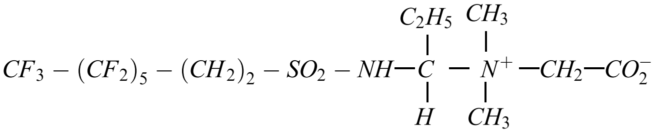
Introduction

In their metastable states (Newton and common black films), soap films are among the thinnest natural objects in everyday life. Their thicknesses typically range between 4 nm (Newton) and 20 nm (common) and they offer a very simple and unique opportunity to confine colloidal objects, such as polymers, for instance. The viscoelastic properties of soap films have received a lot of attention [1], but up to now only dilational properties have been studied and more specifically the elastic properties (Gibbs elasticity [2]) of a draining soap film [3] or of a single bubble [4]. In a previous paper [5], we described a new and original experimental device that we built to measure the shear dynamic properties of a single, freely suspended, black soap film. Quite surprisingly, we found that a soap film exhibits a small shear elastic modulus in spite of the fact that soap films are usually considered to be in a liquid state. In this paper, we confirm the existence of this small elasticity and we speculate about its origin and on the possible related glassy structure of the film. In addition, we present new experiments where polymers have been incorporated inside a soap film to investigate the influence of this forced confinement on the viscoelastic properties of the film.

Experimental

Materials

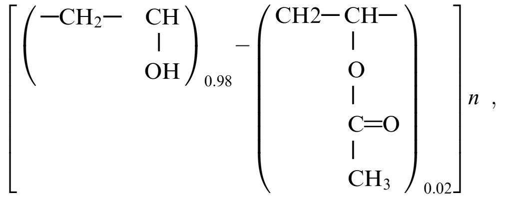
The soap films were made from surfactant aqueous solutions of a fluorinated betaine (FB) of formula



$$CF_3-(CF_2)_5-(CH_2)_2-SO_2-NH-\underset{H}{\overset{C_2H_5}{C}}-\underset{CH_3}{\overset{CH_3}{N^+}}-CH_2-CO_2^-$$

(a gift from Elf-Atochem) or from sodium dodecyl sulfate (SDS) purchased from Aldrich and used as received. The critical micellar concentrations (cmc) were 0.01% g/g for FB and 0.24% g/g for SDS. All samples were prepared with triply distilled water.

We incorporated in the soap films a 98%-hydroxylized poly (vinylalcohol) (PVA, purchased from Aldrich) of formula



$$\left[\left(-CH_2-\underset{OH}{CH}\right)_{0.98}-\left(CH2-\underset{\underset{\underset{CH_3}{C=O}}{O}}{CH}-\right)_{0.02}\right]_n,$$

We used two different weight-average molecular weights, M_w = 25,000 and 145,000 g. The polymer was first purified by precipitation in ethanol, dried for several hours in a stove under vacuum, then dissolved and stirred in water (3 h at 80 °C).

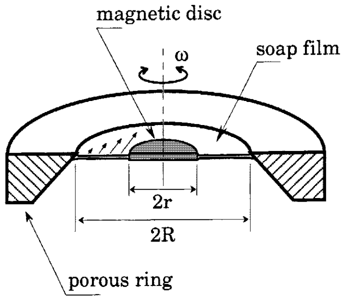


Fig. 1 Schematic section of the measurement device

Experimental setup and measurements

The principle of our original measurement setup consists of studying the response of a magnetic disc (of radius r) sustained in a single black soap film (of radius $R = 15$ mm and thickness e) (see Fig. 1) and submitted to an oscillating external magnetic field, $H(t) = H_0 \cos \omega t$, applied using a pair of Helmholtz coils. The film, that does not wet the magnetic disc, is suspended on a sintered glass ring which allows external control of the film thickness by monitoring the liquid pressure inside the ring. The magnetic disc is made from video recorder tape; it is centered inside the small deformation of the film caused by its weight (about 5 mg including the mirror) and it is magnetized inside the high magnetic field of the electromagnet gap of an electron spin resonance spectrometer in order to acquire a magnetic momentum, $\vec{\mu}$. The viscoelastic properties are deduced from the rotational response, $\theta(t) = A\cos(\omega t + \Phi)$, of the disc that is easily determined from the position of a laser beam reflected by a small mirror glued on the disc[1]. This response is governed by

$$I\frac{\mathrm{d}^2\theta}{\mathrm{d}t^2} + \alpha\frac{\mathrm{d}\theta}{\mathrm{d}t} + \beta\theta + \mu H_a \sin\theta = \mu H(t)\cos\theta \ , \tag{1}$$

where the angle θ is taken from the direction of the horizontal component of the Earth's magnetic field, H_a, and for an experimental geometry where we set the excitation field, $H(t)$ orthogonal to the Earth's horizontal field, H_a. I is the moment of inertia of the disc and the coefficients α and β are related to the shear viscosity, η_s, and the 2D shear modulus, E_s, by (assuming a small gap $R - r$):

$$\alpha = 2\pi\left(\frac{r^3}{R-r}\right)\eta_\mathrm{s} \tag{2}$$

$$\beta = 2\pi\left(\frac{r^3}{R-r}\right)E_\mathrm{s} \ . \tag{3}$$

Equation (1) is easily solved for small θ since it reduces to

$$\frac{\mathrm{d}^2\theta}{\mathrm{d}t^2} + \alpha'\frac{\mathrm{d}\theta}{\mathrm{d}t} + \beta'\theta = \frac{\mu H_0}{I}\cos\omega t \ , \tag{4}$$

with $\alpha' = \alpha/I$ and $\beta' = (\beta + \mu H_\mathrm{a})/I$. The solutions of this equation in a permanent regime are

$$A = \frac{\frac{\mu H_0}{I}}{\sqrt{(\alpha'\omega)^2 + (\beta' - \omega^2)^2}} \tag{5}$$

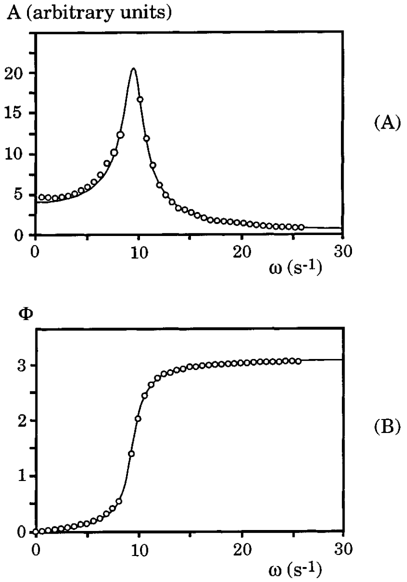


Fig. 2 A Rotational response amplitude, $A(\omega)$, and **B** phase, $\Phi(\omega)$, of the oscillation of the disc with respect to the angular frequency, ω, of the excitation magnetic field. *Solid lines* represent fits using Eqs. (5) and (6)

$$\Phi = \arctan\left(\frac{\alpha'\omega}{\beta' - \omega^2}\right) \ . \tag{6}$$

One of the difficulties of the experiment is to get rid of the Earth's magnetic field. The 2D elasticity and the viscosity of the soap film are obtained from the fits of the experimental amplitude and the phase of the response as shown in Fig. 2.

We reported in a previous paper [5] that the measured small elastic shear modulus ($E_\mathrm{s} \simeq 10 \cdot 10^{-6}\,\mathrm{N\,m^{-1}}$) of a black soap film may have several origins:

1. The shielding of the Earth's magnetic field, $\vec{H}_\mathrm{a}$, is not efficient enough.
2. A mode transformation: the motion of the magnetic disc on the film induces dilation/compression as well as shearing.
3. A soap film actually has a small shear modulus.

Effect of the Earth's magnetic field

In order to investigate the first point, we undertook additional experiments where instead of shielding the Earth's magnetic field (with a piece of so-called μ-metal), we applied an artificial magnetic counterfield of amplitude H_c to anneal the Earth's field, H_a.

The 2D elasticity and the viscosity versus the counterfield amplitude, H_c, relative to the horizontal component, H_a, of the Earth's field are shown in Fig. 3. It can be deduced from this figure that the perturbation resulting from the Earth's field is at most 25%.

Mode transformation

We performed several tests to ensure that the measured modulus cannot be attributed to a transformation (see point 1 above) from a shear mode to a compression/dilation mode: then, in this case, the measured elasticity would be related to the Gibbs elasticity.

Our first test was to change the amplitude of the applied field, $H(t)$; indeed, in the case of the existence of mode transformation, the resonance should change in accordance with the amplitude,

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[1] This mirror is tiny ($3 \times 2 \times 0.15\ \mathrm{mm}^3$) and is centered on the disc so that it does not disturb the rotational symmetry

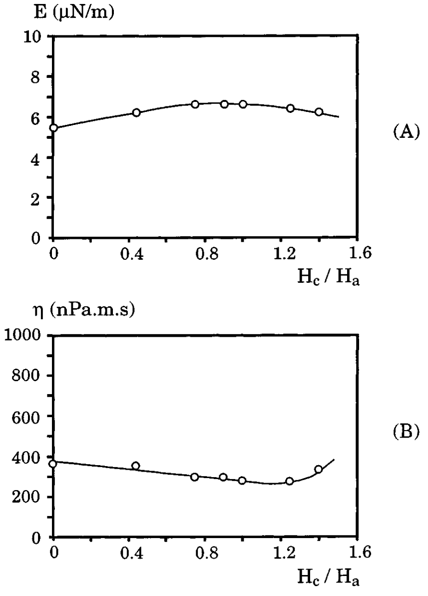

Fig. 3 **A** Surface shear elasticity and **B** viscosity of a common black film ($e \simeq 20$ nm) of fluorinated betaine (*FB*) (0.03% g/g) measured for different amplitudes of the static magnetic counter-field, H_c

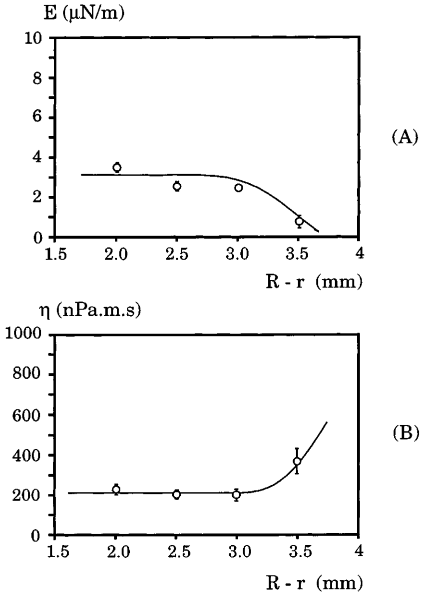

Fig. 4 **A** Surface shear elasticity and **B** viscosity of a common black film (e $\simeq$ 20 nm) of FB (0.03% g/g) measured with discs of different radii

i.e. with the amount of mode mixing. We did not notice any change in the viscoelastic properties of a soap film when the amplitude, H_0, of the exciting field was changed. The second test is to use magnetic discs of different radii. Indeed, the amplitude of any mode transformation should be strongly dependent on the radius of the magnetic disc. The 2D elasticity and viscosity of a black soap film for four different gaps ($R - r$) of the rheometer are shown in Fig. 4. Here we used the exact value of the shear [6] and not the small-gap approximation, $R - r \ll r$, as in Eqs. (2) and (3). It can be seen that the 2D elasticity and the viscosity remain constant as expected except for the largest gap of $R - r = 3.5$ mm: we could attribute the decrease in elasticity to the fact that the gap could be larger than a typical crystallite size (or a typical gel-like glassy domain size) but this cannot explain the increase in viscosity. Anyway, all subsequent measurements were performed with the smaller gap (2 mm) and using the small-gap approximation of $R - r \ll r$.

Another undesirable physical phenomenon that could affect our determination of the viscoelastic properties of soap films is the presence of meniscii at the borders of the film (i.e. along the porous ring and along the magnetic disc). We determined the influence of the meniscii by studying the response of a magnetic disc floating at the surface of clean water contained in a beaker: of course, in this case there should not be any shear modulus since we are dealing with a simple liquid, but we observed an elastic restoring torque because of the magnetic field of the Earth and the presence of a meniscus (of height equal to the capillary length of water, i.e. 2 mm) that centers the magnetic disc. The equivalent shear modulus that we can deduce from this elastic torque is less than 10% of the value that we measured with a black soap film in a comparable geometry (same viscometer gap).

Then, after this careful review and elimination of (hopefully) all artifacts, we can affirm that our setup constitutes a unique way to determine the shear viscoelastic properties of soap films.

Measurements of the thickness of the film

On top of the viscometer we fitted an interferometric device to measure the thickness, e, of the film while it is sheared. We measured the intensity, I^r, of monochromatic light (we used a laser diode with a wavelength, λ, of 640 nm) reflected by the film under nearly normal incidence. Actually we measured the contrast, Δ, defined in Ref. [7] as a function of time while the film drained from its formation until its final metastable state:

$$\Delta = \frac{I^r - I^r_{\min}}{I^r_{\max} - I^r_{\min}} = \frac{(1 + F_0^2)^2 \sin^2\left(\frac{2\pi n e}{\lambda}\right)}{(1 - F_0^2)^2 + 4F_0^2 \sin^2\left(\frac{2\pi n e}{\lambda}\right)} \ , \tag{7}$$

where $I^r_{\min}$ and $I^r_{\max}$, are the relative minimum and maximum respectively, of the reflected intensity before the film reaches its metastable state (black film) and F_0 is the Fresnel coefficient defined as $F_0 = \left(\frac{1-n}{1+n}\right)$, with n being the refractive index of the film, which we assume to be homogeneous and equal to 1.33 (i.e. the water value).

Results and discussion

Surface properties of a soap film

The surface shear modulus and viscosity for soap films made from FB and SDS are shown in Table 1.[2]

The time refers to how long after their formations the films were studied and it does not have a pronounced influence: all the films studied were common black films of measured thickness of about 20 nm. The concentrations expressed as percentages (in g/g) represent 1 cmc

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[2] The surface shear viscosity is often expressed in the CGS unit, namely the surface Poise (sP): 1 sP = 10^{-3} Pa m s

Table 1 2D shear elasticity and viscosities of common black soap films made from sodium dodecyl sulfate (*SDS*) or (*FB*) fluorinated betaines

	E_s (10^{-6} N m^{-1})	η_s (10^{-9} Pa m s)
SDS (6 h) 0.24%	8.6 ± 0.1	320 ± 20
SDS (20 h) 0.24%	8.6 ± 0.2	280 ± 30
FB (6 h) 0.03%	6.4 ± 0.2	322 ± 40
FB (6 h) 0.1%	8.6 ± 0.2	355 ± 30

for the SDS and 3 and 10 times the cmc for the fluorinated surfactant FB.

If we consider that a common soap film is made of two surfactant molecule monolayers separated by a soap solution layer (of thickness of about *e*), we can write

$$\eta_s = 2\eta_{\text{mono}} + e\eta \simeq 2\eta_{\text{mono}} \ , \qquad (8)$$

where η_{mono} is the shear viscosity of a monolayer and η the bulk viscosity of the solution ($\eta = 0.92 \times 10^{-3}$ Pa s at 25 °C), and it is clear that the measured viscosity η_s is entirely determined by the surfactant monolayers. It is then naturally interesting to compare our determination of the viscosity of a SDS monolayer of a soap film with the numerous determinations of the shear viscosity of a SDS Gibbs layer. For instance, Petkov et al. [8] and Goodrich [9] have measured for SDS a surface shear viscosity value η_{mono} of 2×10^{-6} Pa m s, i.e. a value about 10 times larger than the present value. This requires further comment.

Our measured value does not depend at all on a model of the hydrodynamics of the subphase that is needed for Langmuir and Gibbs layers [10]. The values of Petkov et al. and Goodrich were obtained with an approximately 10 cmc SDS solution and we can wonder whether the state of the molecules is then the same as in a soap film. The only indication that we have is the value of the area per polar head, $\Sigma = 45 \pm 2$ Å^2, that has been measured by neutron reflectivity [11] at the air/solution interface obtained at the cmc to be compared to $\Sigma = 34$ Å^2 measured by X-ray reflectivity on a SDS common black film [12]: this suggests that the packing of SDS molecules is higher in a soap film than in a Gibbs monolayer and so we should expect a higher surface viscosity for soap films that we do not measure experimentally. Nevertheless, it is difficult to draw any conclusion on the dynamics of the molecules and thus on the viscoelastic properties since the volume in which the molecules can diffuse and subsequently the time of exchange between bulk and surface is drastically limited in a soap film.

The origin of a shear modulus in soap films

Our measurements with films made with FB and SDS show that these films have similar shear viscoelastic properties, i.e. a very small shear modulus (about 10 μN m^{-1}) and a high surface viscosity (around 300 nPa m s). It is instructive to derive a 3D equivalent viscosity, η, from the surface shear viscosity, η_s, from

$$\eta = \eta_s/\delta \ , \qquad (9)$$

where δ is the thickness of the sheared surface; for instance, for the SDS common black film, taking $\eta_s \simeq 150$ nPa m s (for a single monolayer of the film) and $\delta \simeq 10$ Å [12], we obtain $\eta \simeq 150$ Pa s, i.e. more than 10^5 times the viscosity of water at room temperature, a very high value indeed. This high viscosity is characteristic of lyotropic films [13–15] and has, to the best of our knowledge, not received a sound explanation. Most theoretical models concern the viscosity of thermotropic liquid crystals that do not exhibit such high viscosity values [16]. The solution has to be searched for in the extension of the alkyl chain out of the interface plane that should be related to high friction. It is possible to build a characteristic time, τ, from these high viscosities by using the surface pressure, Π, (in the case of an adsorbed monolayer) and the scaling relation $\eta_s = \Pi\tau$ [17]. The surface pressure can be estimated by $k_B T/\Sigma$, with $k_B T$ being the thermal energy and Σ being the surface area occupied by each surfactant at the interface, and is thus of the order of 10 mN m^{-1}. We obtain a characteristic time of the order of 10 μs, much larger than expected for a conventional bulk liquid (10^{-11} s); this would correspond to an energy barrier of $k_B T \ln\left(\frac{10 \cdot 10^{-6}}{10^{-11}}\right) \simeq 14 k_B T$ in a hopping model of viscosity. Since we have determined a shear modulus for common black films, we can also use a similar derivation for a characteristic time: $\eta_s = E_s\tau$. Using the average values of Table 1, we obtained $\tau \simeq 30$ ms, corresponding to an energy barrier of $\simeq 20 k_B T$. We have no microscopic model to explain these rather high energies, but the latter characteristic time estimation is compatible with the experimental observation of a resonance peak for a angular frequency $\omega_c \simeq 10$ s^{-1} (Fig. 2), corresponding to an experimental excitation time $\tau_c = \frac{R-r}{\omega_c r} \simeq 0.04$ s.[3] We then arrive at the conclusion that a soap film behaves as a solid (or at least as a glass) at short times (smaller than τ). Note that recent experiments [18, 19] even claim that the thinner Newton black films (of thickness of approximately 4 nm) have a long-range solid order.

The role of confined polymer chains

Polymers are often added to aqueous foams to slow down their drainage rate [20–22] and might have an

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[3] A possible explanation could reside in a collective tilt of the alkyl chains of the surfactant as suggested by X-ray data [12]: shearing would then induce a symmetry-breaking associated with high energies (J. Prost, private communication)

Table 2 Characteristics of the polymer in water. M_w: molecular mass (weight-average), $[\eta]$: intrinsic viscosity, c^*: bulk overlap concentration, $R_{F_3}^{GPC}$: chain radius (determined by gel permeation chromatography), N: number of monomers, $R_{F_3}^{calculated}$: chain radius (calculated from scaling laws)

M_w (g mol^{-1})	$[\eta]$ (ml g^{-1})	c^* (g l^{-1})	$R_{F_3}^{GPC}$ (nm)	N	$R_{F_3}^{calculated}$ (nm)
25,000	28	45	–	60	12
145,000	86	10	30	370	38

effect on surface rheology and 2D turbulence [23]. We thus studied the influence of the confinement of a hydrosoluble polymer on the viscoelastic properties of a soap film. More specifically, we studied black films made from a FB–PVA solution (see Materials). The low-molecular-weight polymer (25,000 g) is not confined in a 20-nm film in contrast to the high-molecular-weight polymer (145,000 g) that is squeezed by the soap film (Table 2). The thickness of the film is not changed by the incorporation of polymers.

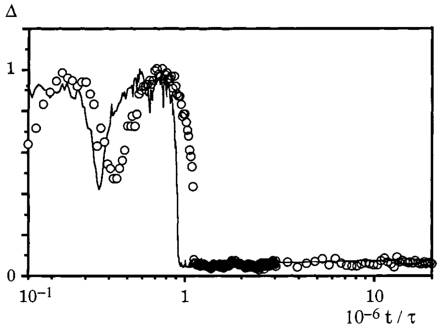

Fig. 5 Thinning of a film made from a FB solution (FB: 0.03% g/g) without polymer (○) and incorporating polymer (*continuous line*) [polyvinyl alcohol (*PVA*): 145,000 g mol^{-1}, 1% g/g]: the contrast (Δ) defined in Eq. (7) is plotted versus a reduced time, $\left(\frac{t}{\tau}\right)$ [22] with $\tau = \kappa^{-1}\frac{\eta}{\gamma}$, where κ^{-1} is the capillary length $\sqrt{\frac{\gamma}{\rho g}}$, γ is the surface tension of the film, ρ is the volumic mass and g is the acceleration due to gravity. We can deduce from this plot that the film containing polymer drains faster: the slope of $\Delta(t/\tau)$ just before the film abruptly reaches the black film is larger for the film containing polymer because of the additional attractive interaction between the surfaces of the film due to the formation of a depletion layer

Contribution of the polymer to the shear modulus: theoretical predictions

To the best of our knowledge, only the influence of confined polymers in lamellae on the compression modulus (and not on the shear modulus) has been studied theoretically [24, 25], and only in the particular case where the polymer does not adsorb on the surfactant monolayers (depletion regime [26]).

The polymer contribution, G, to the compression modulus of a swollen bilayer was calculated from the solution free energy using scaling laws [27]. Two situations have been described according to the possibility of the polymer to exchange with an external reservoir: constant volume fraction, ϕ, or constant chemical potential, μ. The theoretical predictions [24, 25] for the contribution, G, to the total compression modulus are reported in Table 3.

In order to use these theoretical predictions, we have to check that the polymer chains of PVA do not adsorb on the FB surfactant monolayers and to decide whether the shearing is done at constant volume fraction of the polymer or at constant chemical potential. Drainage measurements (Fig. 5) show that FB films containing PVA thin slightly faster than FB films without polymer. This behavior can be attributed to depletion layers at the interfaces, inducing an additional attractive interaction [21]. We can derive predictions for the effect of polymers on the shear modulus, E_s, from the predictions of the compression modulus, G, by using the Poisson ratio, v:

$$\frac{G}{E_s} = 2(1 + v) \ . \tag{10}$$

We have supposed that the Poisson ratio remains constant with polymer concentration and does not depend on the frequency of the mechanical deformation. Shearing a polymer chain increases its chemical potential: we can then assume that, when a soap film incorporating polymer is sheared, the polymer molecules will tend to escape the regions of high shear (close to the magnetic disc) and move towards the unsheared regions (the outer plateau border in contact with the porous ring) in order to homogenize the chemical potential in the film: the constant chemical potential hypothesis should clearly apply for the theoretical predictions of Table 3.

Table 3 Theoretical polymer contribution, G, to the total compression modulus of a nonadsorbed polymer (number of monomers N) confined between two parallel planes separated by a distance e (good solvent regime). a is a microscopic length (monomer size)

Regimes	G (in units of $\frac{k_B T}{d^3}$)	
	ϕ constant	μ constant
3D dilute	$\frac{1}{N}\left(1 + 4\left(\frac{R_{F_3}}{e}\right)\right)\phi$	$-4\left(\frac{a}{e}\right)^2 N^{1/5}\phi$
3D semidilute	$\frac{15}{2}\left(\frac{a}{e}\right)\phi^{3/2} + \frac{45}{6}\phi^{9/4}$	$-\frac{16}{5}\left(\frac{a}{e}\right)^2\phi^{3/4}$
2D dilute	$\frac{40}{9}\left(\frac{a}{e}\right)^{5/3}\phi$	$-\frac{25}{9}N\left(\frac{a}{e}\right)^{10/3}\phi$
2D semidilute	$2\left(\frac{e}{a}\right)\phi^3 + \frac{40}{9}\left(\frac{a}{e}\right)^{5/3}\phi$	$-\frac{25}{54}\left(\frac{a}{e}\right)^{13/3}\phi^{-1}$

Experimental results

FB–PVA 25,000 films in the 3D regime: Films containing short polymer chains have a smaller elasticity and their viscosity remained the same for all concentrations studied (Fig. 6).

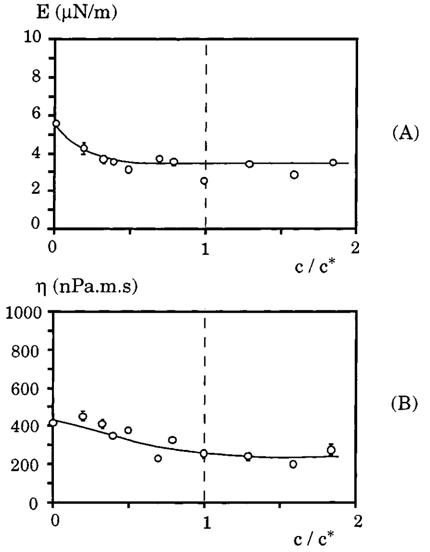

Fig. 6 **A** Surface shear elasticity and **B** viscosity of a common black film (e ≃ 20 nm) of FB (0.03% g/g) containing PVA (M_w = 25,000 g mol^{-1}) as a function of polymer concentration

FB–PVA 145,000 films in the 3D regime: Films made with FB–PVA 145,000 solutions contain polymer chains which are confined in the common black film. Their bidimensional viscoelastic properties in shearing according to polymer concentration are pictured in Fig. 7. E_s and η_s have been plotted with respect to (c/c_2^*), where c_2^* is the 2D overlap concentration. This concentration has been determined from $\eta_s(c)$ measurements as is usually done for polymer solutions. c_2^* corresponds to the concentration where the surface viscosity, η_s, increases drastically. Below c_2^*, the polymer contribution to the elastic shear modulus of the film is negative and the viscosity remains the same as for that measured without polymer. In the 2D semidilute regime, $c \gg c_2^*, E_s$ and η_s increase with polymer concentration.

These results are qualitatively described by the model corresponding to a shear deformation with a constant chemical potential. Figures 6 and 7 show that the contribution of polymers to the shear modulus of the film is always negative except in the 2D semi-dilute regime, where E_s increases with polymer concentration with the predicted $-\phi^{-1}$ behavior. Nevertheless, we do not observe a decrease in the elasticity in the semidilute regime (Fig. 6A) as for the $-\phi^{3/4}$ prediction.

The shear viscosity increases significantly above a concentration threshold, defining a 2D overlap concentration, c_2^*, which is about twice the overlap concentration in the bulk. The confinement disentangles the chains, as expected from the scaling law prediction:

$$\frac{c_2^*}{c^*} \sim N^{\frac{3}{10}}\left(\frac{a}{D}\right)^{\frac{1}{2}} , \qquad (11)$$

where D represents the thickness of confinement.

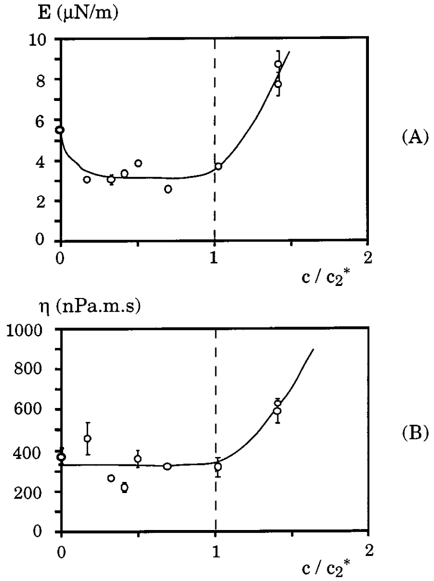

Fig. 7 **A** Surface shear elasticity and **B** viscosity of a common black film (e ≃ 20 nm) of FB (0.03% g/g) containing PVA (M_w = 145,000 g mol^{-1}) as a function of polymer concentration

Conclusions

We have measured the shear viscosity and elasticity of common black soap films using an original viscometer. The films studied (made from SDS or from a FB) have a very small shear modulus ($E_s \simeq 10\ \mu$N m^{-1}) and a high surface viscosity ($\eta_s \simeq 300$ nPa m s). These values are characteristic of a viscoelastic behavior with a relaxation time of about 30 ms: the origin of the elastic regime (a glassy state) has not yet been elucidated.

The confinement of PVA in the FB films induces a decrease in the elastic shear modulus, except in the 2D semidilute regime, where this modulus increases with polymer concentration. The effect of the polymer is qualitatively described by using a simple scaling law theory that assumes that polymer chains can be exchanged with a reservoir during shearing. Nevertheless, this effect is very limited: the rheological behavior of a soap film containing polymer chains is ruled by the dense packing of the surfactant molecules at the interfaces of the film.

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