

Rheological and Shearing Conditions for the Preparation of Monodisperse Emulsions

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Monodisperse emulsions can be obtained by shearing a crude polydisperse one. The aim of this paper is to discover the required conditions for droplet fragmentation and resulting monodispersity, independently of the chemical nature of the system. We particularly examine (i) the role of the initial emulsion viscoelasticity and (ii) the nature of the applied shear. Taking into account these features, we show a large variety of monodisperse materials that are easily producible in reasonable quantity.

Introduction

Monodisperse emulsions may be obtained by a fractionated crystallization process¹ based on depletion interactions. This process is well-controlled and theoretically understood; however, it is quite fastidious to make use of it. More recently, another very convenient and rapid way of producing such characterized emulsions has been proposed^{2,3} and developed. It consists of shearing a premixed emulsion made of large polydisperse droplets. Though the emulsion fragmentation mechanism is not yet understood, Mason and Bibette found out experimentally that two conditions have to be fulfilled to get monodispersity: (i) shearing must concern a viscoelastic medium and (ii) the gap of the shear cell has to be as narrow as possible (on the order of 100 μm or less). On the basis of these observations, a new Couette cell⁴ has been built. With use of this tool, various systems have been investigated to determine the conditions for controlled droplet rupturing to occur. The influence of various parameters, such as the premixed emulsion rheology and the shearing conditions, are related to the final droplets size distributions.

This paper should be considered as the continuation of ref 3; we aim to present experimental aspects of the droplets rupturing on diverse systems without going into the complex fragmentation mechanism. We only consider amphiphilic species that adsorb rapidly at the oil/water interface. The outline is as follows: in the first part, we describe the Couette apparatus that makes this study possible. The second section gives an overview of the various systems we studied as well as the characterization methods. The third part is devoted to the presentation of the experimental results and to a general discussion of emulsion fragmentation.

I. Couette Cell Description

The Couette mixer⁴ consisting of two concentric cylinders is schematically shown in Figure 1. It is equipped with a syringe pump for injecting the premixed emulsion, allowing continuous production.

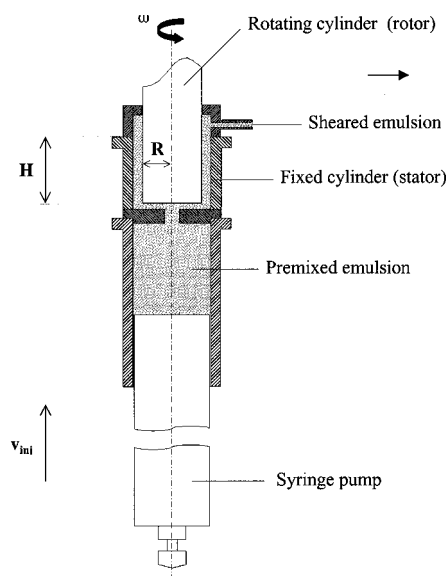


Figure 1. Scheme of the Couette mixer. The premixed emulsion, contained initially in the syringe, is pushed by a piston into the gap between the rotor and the stator. The sheared emulsion is recovered at the top of the mixer.

The inner cylinder of radius $R = 19.89$ mm is linked to a motor that rotates at a selected angular velocity ω which can reach up to $71.2 \text{ rad}\cdot\text{s}^{-1}$. The velocity v at the surface of this cylinder is $v = R\omega$. The outer cylinder is fixed. We can choose from three stators of different radii R_i so that the shearing can be applied in three different gaps, $d_i = R_i - R$ ($d_i = 100 \mu\text{m}$, $200 \mu\text{m}$, or 1 mm), depending on the chosen stator. This apparatus is manufactured by T.S.R. Co.⁵

For the maximum angular velocity, we are able to reach very high shear rates $\dot{\gamma} \sim R\omega/(R_i - R) \sim 14\,200 \text{ s}^{-1}$ for a gap of $d_i = 100 \mu\text{m}$. The Couette mixer is designed so that it is possible to change the covering height H between the outer cylinder (stator) and the inner one (rotor); see Figure 1. The premixed emulsion contained initially in the syringe is pushed at a selected injection velocity, v_{inj} , by a piston into the gap between the rotor and the stator. We chose to work here at maximum H and minimum v_{inj} so that the

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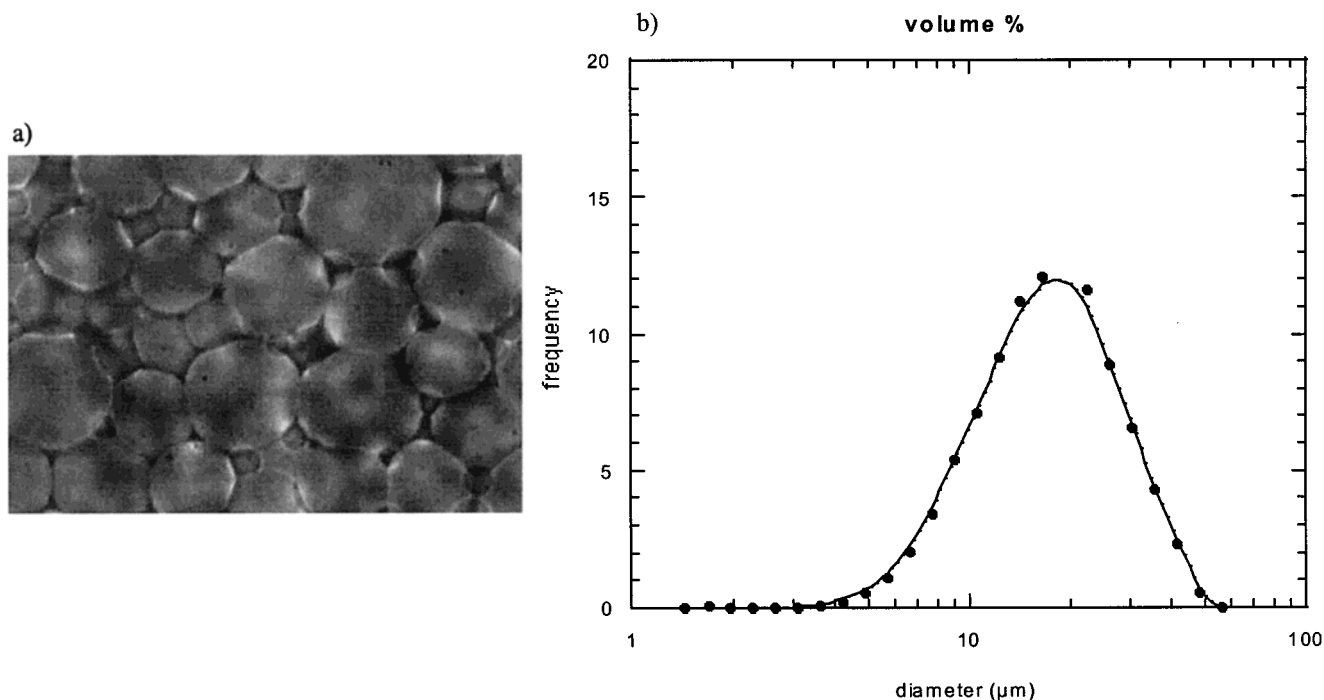


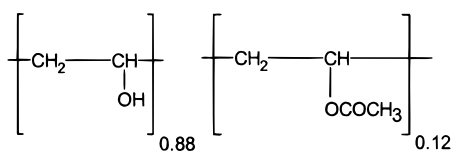
Figure 2. Example of a premixed emulsion made of a 40% Ifralan solution containing 75% silicone oil (10 Pa·s). The measured average size is about 20 μm and uniformity is around 40%.

emulsions are sheared in about 10 s. With this Couette mixer, it is possible, according to the viscoelastic properties of the premixed emulsion and the selected gap, to produce rapidly some significant quantities of emulsions with narrow size distributions (up to 1 or 2 L/h, depending on the system, at volume fractions ranging from 70 to 85%).

II. Material and Characterization Method

1. Systems. As already mentioned, we aim to describe some general features of fragmentation. For this reason our emulsions are of both the direct and reverse type, stabilized either by surfactants or emulsifying polymers. We also consider the possible addition of thickeners: polymers without significant interfacial properties.

To stabilize direct emulsions (oil in water),⁶ we choose some nonionic hydrophilic surfactants of low molecular weight (~ 500) and some polymers. The hydrophilic surfactants we used are Ifralan 205, a mixture of pentaethylene glycol mono *n*-dodecyl ether ($\text{C}_{12}\text{H}_{25}(\text{OCH}_2\text{CH}_2)_5\text{OH}$) and pentaethylene glycol mono *n*-decyl ether ($\text{C}_{10}\text{H}_{21}(\text{OCH}_2\text{CH}_2)_5\text{OH}$) purchased from Ifrachem, and Tergitol NP7, heptaethylene glycol nonylphenol ($\text{C}_9\text{H}_{19}(\text{C}_6\text{H}_5)\text{OH}(\text{OCH}_2\text{CH}_2)_7\text{OH}$) provided by Sigma. As a polymeric emulsifier, we used a copolymer of polyvinyl alcohol and polyvinyl acetate (PVAAc) of molecular weight $M_w \sim 15\,000$ purchased from Fluka Chemika, whose chemical formula is



This polymer can be easily solubilized in water. Like the dispersed phase, we employed various silicone oils (poly-

(dimethylsiloxane) or PDMS) provided by Fluka Chemika. Their viscosities were 100, 350, and 1000 mPa·s. Because they are immiscible in aqueous surfactant solutions, the possibility of spontaneous microemulsion formation and Ostwald ripening is considerably reduced. We have also used mineral oil (mixture of alkanes) of very low viscosity (50 mPa·s) purchased from Sigma.

For inverse emulsions, we used Span 80 (sorbitan monooleate) purchased from Sigma. The dispersed phase was 0.1 M NaCl in water to avoid fast destabilization.⁷

In some experiments, to increase the viscosity of the continuous phase, we added a polysaccharide (Alginate HF120L) of molecular weight $M_w \sim 54\,000$ provided by Disatec from Promova. It consists of D-mannuronate and L-guluronate. It acts as a thickener and has only weak interfacial properties because it does not stabilize an emulsion without the addition of a surfactant. Another example of an aqueous thickener is pectin: we used the Genu pectin type VIS/200 provided by Hercules. The emulsifier concentrations C are given in weight percent with respect to water.

2. Preparation. The first step consists of preparing a crude polydisperse emulsion as described in ref 3: the premixed emulsion. It is prepared by incorporating the chosen oil in a surface-active agent/water solution under gentle mixing to avoid the creation of small droplets. A photo of such a crude emulsion is reported in Figure 2a. As seen from the size distribution (Figure 2b), the droplets have sizes ranging from about 5 to 50 μm . This premixed emulsion is introduced into the Couette injector for controlled shearing.

3. Characterization. The obtained emulsions are observed with a reverse optic Axiovert Zeiss microscope and characterized by a Malvern Mastersizer. The collected scattered intensity as a function of the angle is converted into the size distribution using the Mie theory. The mean

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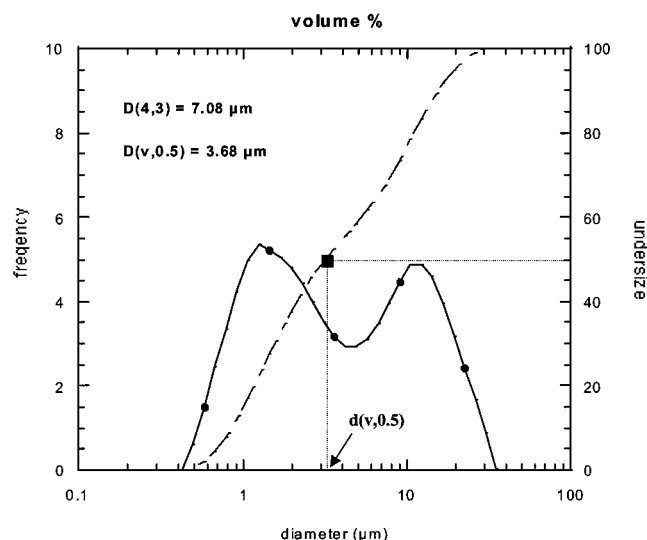


Figure 3. Relation between the size distribution and $d(v, 0.5)$.

size is defined as

$$D[4,3] = \frac{\sum_i V_i d_i}{\sum_i V_i} = \frac{\sum_i d_i^4}{\sum_i d_i^3}$$

where V_i and d_i are respectively the volume and diameter of the i th particle. The polydispersity of the emulsion is parametrized by the uniformity expressed in percentage and defined as

$$U = \frac{\sum_i V_i |d(v, 0.5) - d_i|}{\sum_i V_i} \frac{1}{d(v, 0.5)}$$

U is the volume weighted mean deviation from the median diameter $d(v, 0.5)$ of the distribution. This median value is determined from the cumulative undersized volume fraction as schematically shown in Figure 3. It corresponds to the diameter for which the cumulative undersized volume fraction is equal to 50%. In the following, we characterize the obtained emulsions through $D[4,3]$ and U only. Note that, the higher U , the larger the size distribution (because U represents a deviation from the median value). In the following we consider that an emulsion is monodisperse if U is smaller than 20%.

III. Results

1. Introduction. In this study, we explore a large set of materials and examine the effect of both the rheological properties of the premixed emulsion and the applied shear conditions on the final emulsion. We first show the crucial role of elasticity in controlling the quality of the premixed emulsion fragmentation. Let us consider a reverse emulsion made of 10% 0.1 M NaCl solution in pure Span 80. At such a low dispersed-phase volume fraction, the rheological properties of the premixed emulsion are mainly governed by those of the continuous medium.

The oscillatory measurements of pure Span 80 are reported in Figure 4a. They show that, at low frequencies, i.e., at long time scales, Span 80 behaves mainly as a viscous liquid, whereas at high frequencies, i.e., at short time scales, its elastic modulus becomes equivalent to its

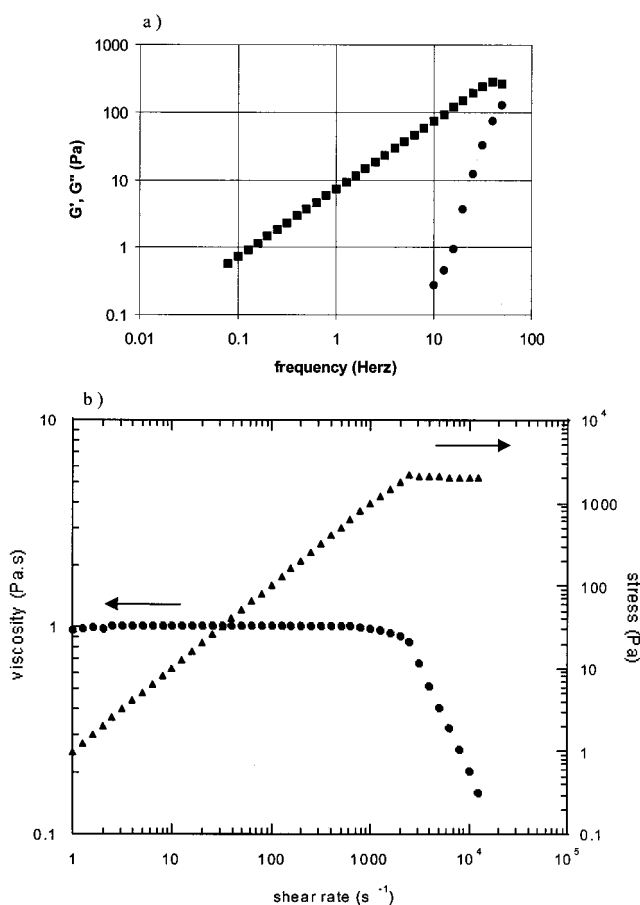


Figure 4. Rheology of pure Span 80: (a) oscillatory measurements: loss modulus (square), elastic modulus (circle); (b) steady-shear measurements: steady-shear viscosity (circle); measured stress (triangle).

loss modulus. Concurrently, the steady shear measurements of pure Span 80, reported in Figure 4b, show that the Span 80 behaves as a Newtonian fluid at low shear rates, which means that it behaves as a purely viscous fluid at long time scales, whereas at higher shear rates, the stress is no longer proportional to the shear rate. This non-Newtonian behavior probably results from the elasticity detected at high frequencies. Therefore, by choosing the appropriate shear rate, one can explore either the purely viscous regime ($\dot{\gamma} < 2000 \text{ s}^{-1}$) or the non-Newtonian regime ($\dot{\gamma} > 2000 \text{ s}^{-1}$). The inverse emulsions are characterized by dynamic light scattering at a detection angle of 90° . The measurements are also made at 60° and 120° for confirmation. At 50 s^{-1} the crude premixed emulsion is unchanged. Indeed, the estimated viscous pressure exerted on the droplets, $\tau_{\text{viscous}} = \eta \dot{\gamma} \sim 60 \text{ Pa}$, is not high enough to overcome the Laplace pressure, $\tau_{\text{Laplace}} = 2\sigma/R \sim 100 \text{ Pa}$, where σ is the interfacial tension between the two fluids. At 1000 s^{-1} , one can see, from the image in Figure 5c, that fragmentation has taken place because the droplets are on average smaller than those in the premixed emulsion, but the emulsion remains highly polydisperse. In this case, the viscous stress is approximately equal to 1200 Pa , which is sufficient to overcome the Laplace pressure. These results are fully consistent with the work of Taylor⁸ who considered the rupture of an isolated droplet in a viscous medium. By balancing the viscous stress responsible for the droplet

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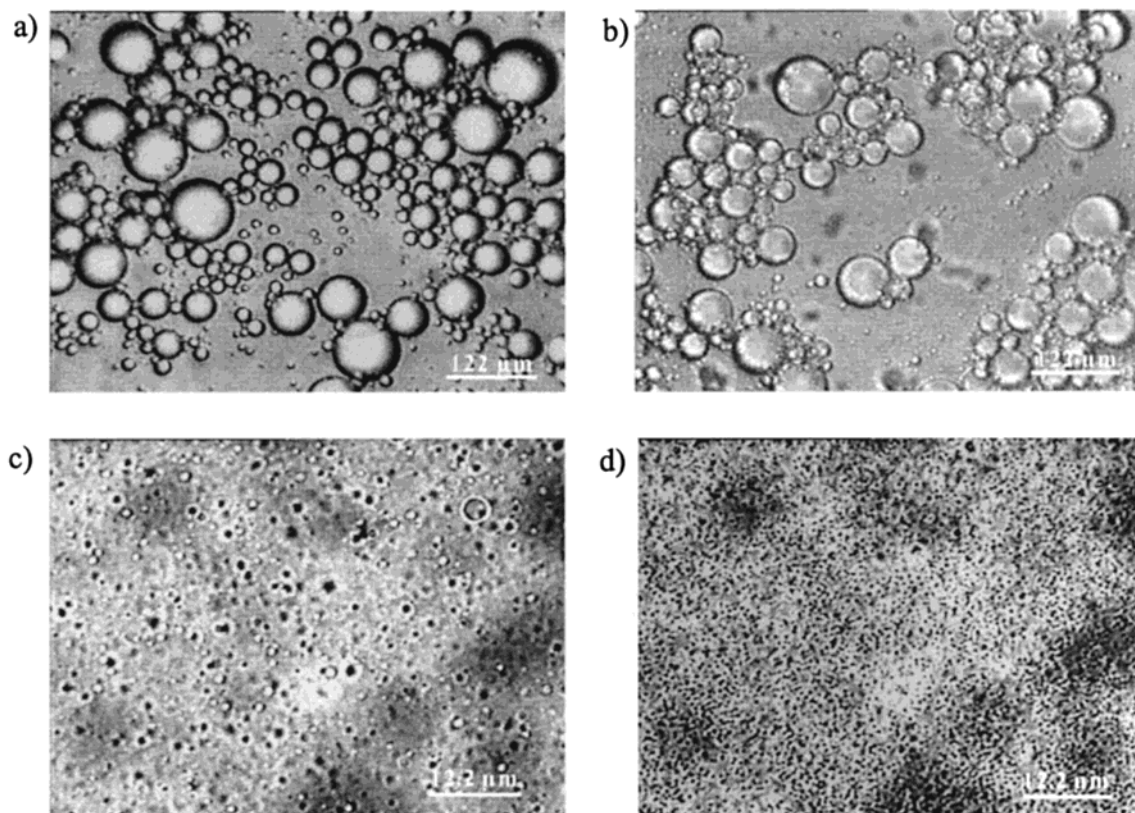


Figure 5. Reverse emulsions in pure Span 80: (a) premixed emulsion; (b) emulsion after shearing at 50 s^{-1} ; (c) emulsion after shearing at 1000 s^{-1} ; (d) emulsion after shearing at 3000 s^{-1} . Note that for photos (a) and (b), the bars represent $122 \mu\text{m}$ whereas for photos (c) and (d) they represent $12.2 \mu\text{m}$.

Table 1. Dynamic Light-Scattering Results Obtained at 90° with a Multiexponential Analysis

$\dot{\gamma} \text{ (s}^{-1}\text{)}$	mean diameter (nm)	distribution limits (nm)
1000	300	from 90 to 735
3000	220	from 160 to 300
10000	170	from 114 to 314

deformation and the Laplace pressure that tends to restore its spherical shape, he related the droplet size to the applied shear rate: $R \sim \sigma/\eta\dot{\gamma}$. By increasing the applied shear rate, one reaches the non-Newtonian regime of the continuous phase attributable to its viscoelasticity. In Figure 5d one observes that fragmentation has occurred and simultaneously the size distribution has become narrower. This illustrates the role of elasticity to get monodispersity. In Table 1 we report the droplet diameters measured from DLS experiments and the distribution width extracted from a multiexponential analysis. Between 1000 and 3000 s^{-1} , the stress is approximately unchanged (see Figure 4b); however, the fragmentation is very different: at 1000 s^{-1} the obtained emulsion is highly polydisperse, while at 3000 s^{-1} , the resulting emulsion is quite monodisperse (see Table 1).

The role of viscoelasticity, as shown in the previous paragraph for a reverse emulsion, is more general and is also valid for direct emulsions. We consider a premixed emulsion made of 40% silicone oil dispersed in a purely viscous phase made of 15% Ifralan 205. This continuous phase exhibits a Newtonian behavior. After shearing, the emulsion remains polydisperse, though the mean size is reduced. An illustration of such a badly fragmented emulsion is given in Figure 6, where the mean diameter is about $17 \mu\text{m}$ and the uniformity is 50%. In the previous example, viscosities of both the dispersed and continuous phases are rather low because they are respectively equal

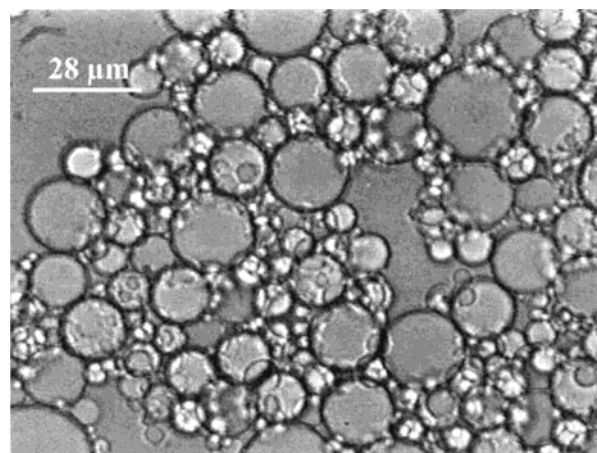


Figure 6. Polydisperse emulsion obtained by shearing a purely viscous premixed emulsion. Composition: $C = 15\%$ Ifralan 205; $\Phi = 40\%$.

to 350 and $20 \text{ mPa}\cdot\text{s}$. To show that a viscosity increase, and hence a viscous stress increase, is not sufficient to get monodispersity, we consider a more viscous continuous medium. This is achieved by adding 4% pectin to water. The viscosity of the continuous phase is then $700 \text{ mPa}\cdot\text{s}$. The obtained emulsion, after shearing, is made up of smaller droplets ($2.4 \mu\text{m}$) but is still badly fragmented with a uniformity around 32%. The wide distributions obtained with purely viscous premixed emulsions clearly demonstrate, as already pointed out by Mason and Bibette,^{2,3} that elasticity is a necessary condition to get monodispersity.

2. Quantitative Consequences of Elasticity on Fragmentation. The rheological properties of the premixed emulsion are controlled by those of the continuous

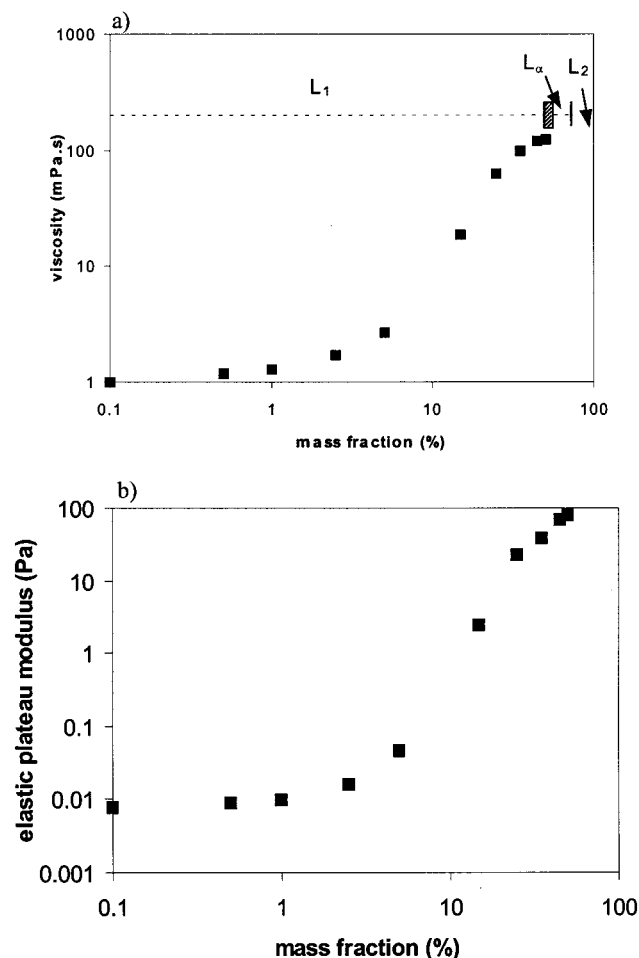


Figure 7. Rheology of the aqueous phases made with Irfalan 205 and phase limits: (a) zero-shear viscosity and (b) elastic plateau modulus as a function of surfactant concentration.

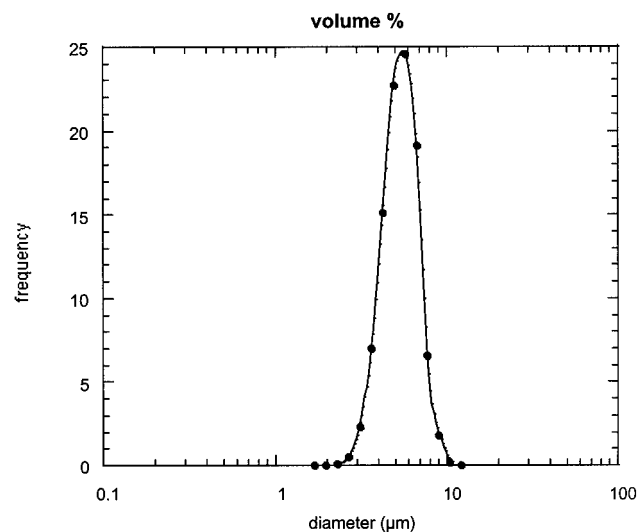


Figure 8. Size distribution of an emulsion obtained by shearing, at 2500 s^{-1} , a premixed emulsion whose elasticity is mainly provided by the continuous phase: $C = 30\%$; $\Phi = 40\%$; $D[4,3] = 5.5 \text{ μm}$; $U = 19.3\%$.

phase and are modified by the presence of droplets at given volume fractions. Hence, by changing both the composition of the continuous phase and the dispersed phase volume fraction, it is possible to tune the rheological properties of the premixed emulsion: elasticity can be obtained either from the intrinsic continuous phase rheological properties (by using appropriate emulsifier

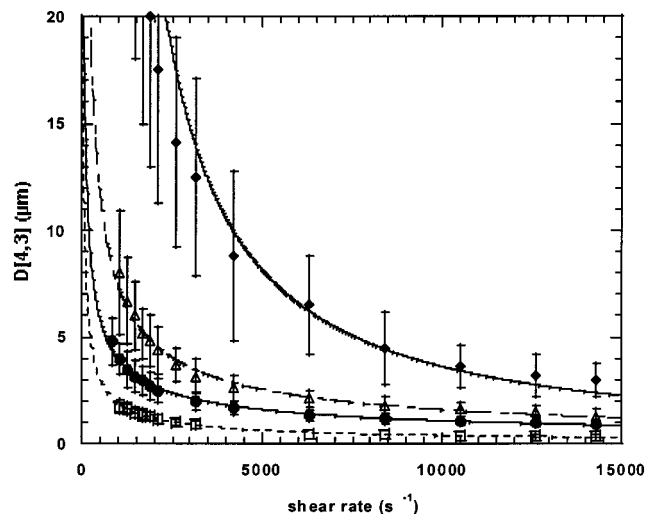


Figure 9. Influence of the emulsifier concentration on the size profiles: diamond, $C = 15\%$; triangle, $C = 25\%$; circle, $C = 30\%$; square, $C = 45\%$ Irfalan 205. The dispersed phase is kept constant at 75% silicone oil of 350 mPa·s viscosity. The lines are guides to the eye.

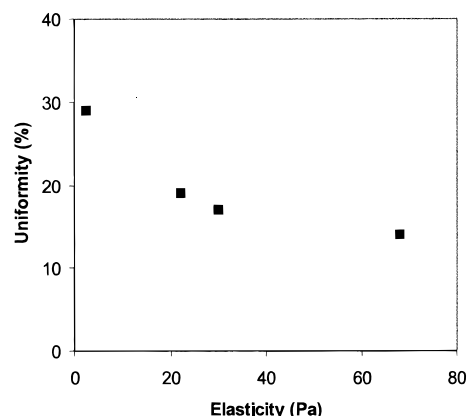


Figure 10. Correlation between uniformity and elasticity in the L_1 phase at $\dot{\gamma} = 2100 \text{ s}^{-1}$, deduced from Figures 7 and 9 (see text).

and/or polymer amounts) or from the packing of the dispersed droplets (for volume fractions above Φ_{rcp}).

a. Viscoelastic Continuous Phase. The viscoelasticity of the continuous phase may be varied by modifying the emulsifier concentration or by adding some thickeners. The emulsifier has a double role, a small amount of it stabilizes the water–oil interface and its excess governs the continuous-phase characteristics: phase behavior and rheological properties. In Figure 7 we report the rheological properties: viscosity and elasticity of aqueous solutions made of Irfalan 205 as a function of emulsifier concentration. The viscosity is the zero-shear viscosity η_0 obtained from oscillatory measurements and the elasticity is the plateau modulus G_0 extracted from a Maxwellian fit of the measured elastic and loss moduli as a function of pulsation: $G' = G_0[(\omega^2\tau^2)/(1 + \omega^2\tau^2)]$ and $G'' = G_0[(\omega\tau)/(1 + \omega^2\tau^2)]$, where G_0 is the elastic plateau modulus, ω the pulsation, τ the characteristic time, and $\eta_0 = G_0\tau$ is the zero-shear viscosity. As seen in Figure 7, by varying the concentration C of surfactant in water, it is possible to alter remarkably the viscoelastic properties of the continuous phase: an increase of the surfactant concentration leads to an increase of the viscoelasticity of the continuous phase.

We consider a premixed emulsion comprised of 40% oil and 60% of a continuous phase containing 30% surfactant

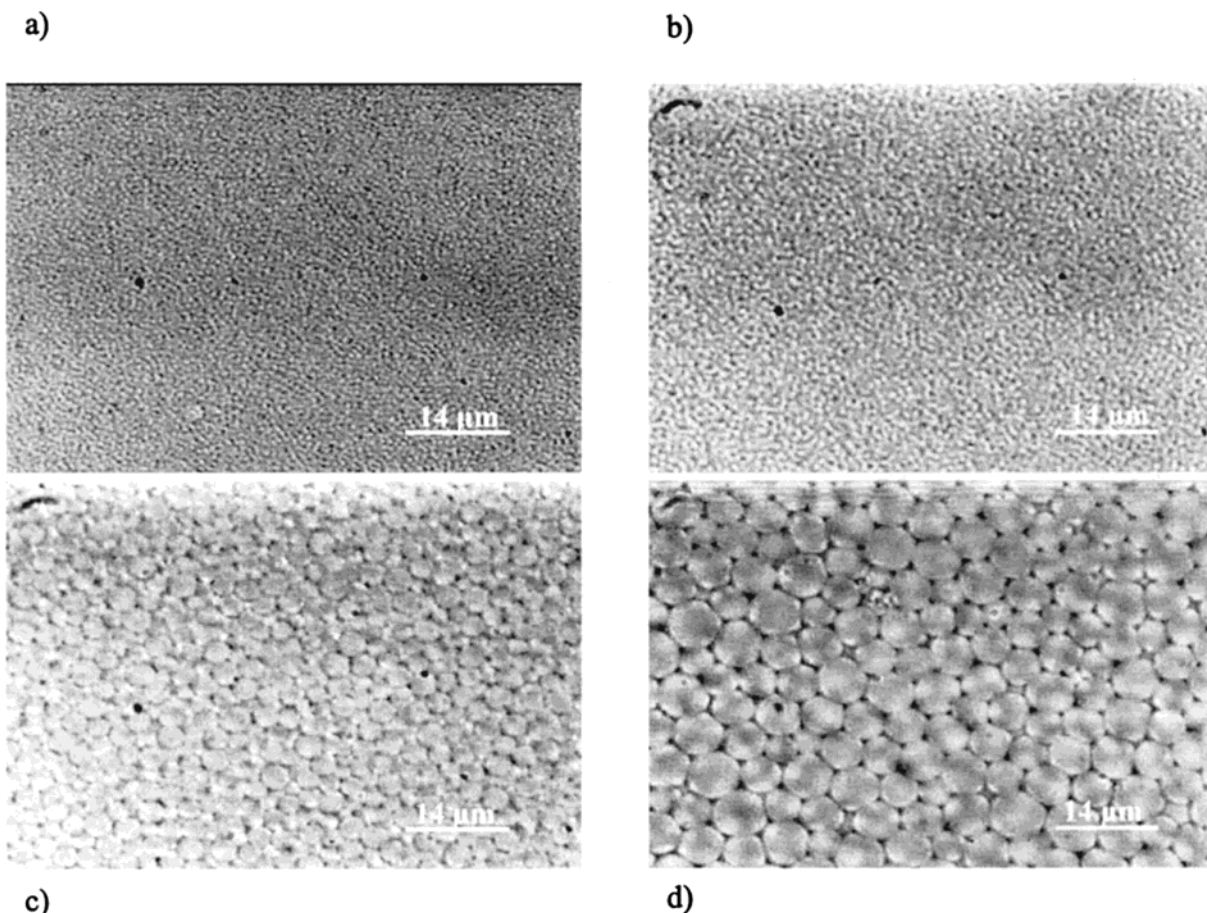


Figure 11. Photos of monodisperse emulsions obtained from viscoelastic premixed emulsions: (a) Ifralan 55%, $\dot{\gamma} = 8400 \text{ s}^{-1}$; (b) Ifralan 45%, $\dot{\gamma} = 2600 \text{ s}^{-1}$; (c) Ifralan 25%, $\dot{\gamma} = 4700 \text{ s}^{-1}$; (d) Ifralan 15%, $\dot{\gamma} = 7350 \text{ s}^{-1}$.

(viscoelastic regime). In Figure 8 we report the size distribution of the sheared emulsion which is centered around $5.5 \mu\text{m}$ with a uniformity of 19%, as expected from the rheological properties of the continuous phase.

The influence of the emulsifier concentration on fragmentation is put into evidence in Figure 9 where we plotted the mean diameter versus the applied shear rate. The dispersed phase volume fraction is kept constant at 75%, while we only vary the continuous medium composition. The reported error bars represent the uniformity of the emulsion. At a given shear rate, both smaller droplets and better fragmentation, i.e., lower uniformity, are achieved by increasing the viscoelasticity (see Figure 9). For example, at 45% Ifralan 205 the uniformity never exceeds 15%, whatever the applied shear rate, whereas it is of the order of 25% for 15% Ifralan 205. The uniformity at 2100 s^{-1} , as a function of the elastic plateau modulus (which depends on C), is plotted in Figure 10. Some microscopic pictures of emulsions involved in the experiment shown in Figure 9 are given in Figure 11.

For industrial and environmental reasons, it would be beneficial to reduce the amount of surfactant, while maintaining the elasticity of the continuous phase. This can be achieved by the addition of a nonadsorbing polymer. Alginate is a good candidate because a small amount (4%) allows one to decrease the NP7 quantity from 30% to 3%, keeping the same size profile and a good fragmentation quality ($1 \mu\text{m}$ and 14% of uniformity for $\Phi = 30\%$ and $\dot{\gamma} = 14\,280 \text{ s}^{-1}$) (see Figure 12).

Some polymeric molecules can themselves act as emulsifiers and replace the surfactant in stabilizing the water–oil interface.⁹ In this case, where the polymers exhibit amphiphilic properties, no surfactant is necessary.

There is no fundamental difference, concerning fragmentation, between emulsions stabilized by a polymer or a surfactant. The only feature that has to be pointed out is the importance of the interfacial tension. Indeed, in Figure 13 we report, for comparison, the fragmentation profiles of two premixed emulsions, one stabilized by the PVAAc polymer and the other by Ifralan 205. The compositions have been chosen so that the continuous phases have the same elastic plateau modulus and the same zero-shear viscosity. This is achieved with 15% PVAAc (see Figure 14) and 35% Ifralan 205 in water: in both cases the elastic modulus and viscosity are respectively equal to 40 Pa and 100 mPa·s. The two size profiles differ by a factor of about 3; the sizes obtained for the polymeric stabilized emulsion are 3 times larger than those obtained, at the same shear rate, with the surfactant. This can be related to the observation that the interfacial tension between silicone oil and the continuous medium is also approximately 3 times larger with the polymer ($17.2 \text{ mN}\cdot\text{m}^{-1}$) than with the surfactant ($5 \text{ mN}\cdot\text{m}^{-1}$).

b. Close-Packed Materials. Elasticity of the premixed emulsion can also be obtained by increasing the dispersed phase volume fraction; it then results from the contact between the droplets and their deformation. The elasticity increases considerably above the close-packed state.^{10–12} We examined the influence of the droplet volume fraction on fragmentation. For this purpose, we used a 15% Ifralan water solution and incorporated, respectively, 40 and 90% oil. The two premixed emulsions were then sheared at the

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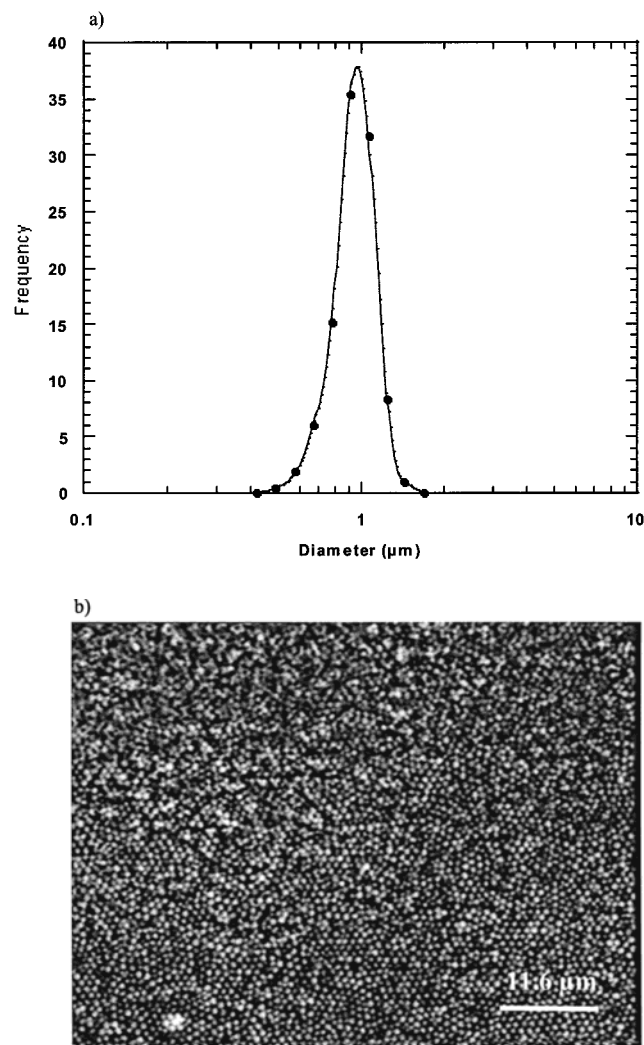


Figure 12. Emulsion obtained by shearing a premixed emulsion in the presence of alginate. Composition: Alginate 4%; NP7 3%; $\Phi = 30\%$.

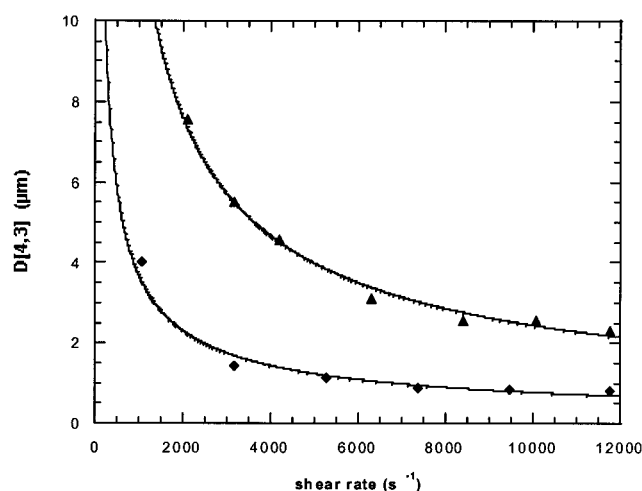


Figure 13. Influence of the interfacial tension on fragmentation. Diamond, Ifralan 5%, $\sigma = 5$ mN/m; triangle, PVAAC 15%, $\sigma = 17$ mN/m. The lines are guides to the eye.

same shear rate $\dot{\gamma} = 14\,280\,s^{-1}$. The size distributions of the two obtained emulsions are reported in Figure 15. This figure clearly shows the effect of the packing on both the average droplet size and the uniformity. The elasticity

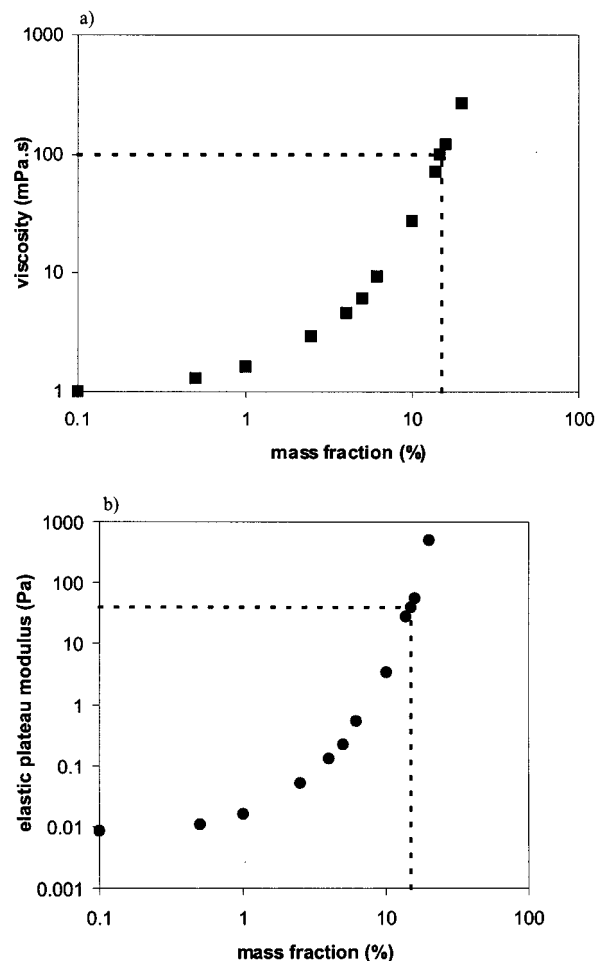


Figure 14. Rheology of the aqueous phases made with polymer: (a) zero-shear viscosity and (b) elastic plateau modulus as a function of polymer concentration.

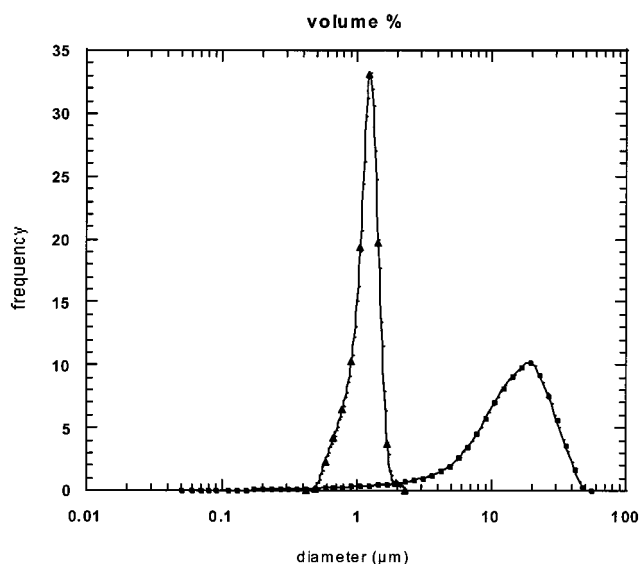


Figure 15. Comparison of the size distribution obtained with 40% (squares) and 90% (triangles) dispersed-phase volume fractions in a 15% Ifralan 205–water solution.

provided from the high droplet packing for the 90% emulsion is sufficient to get a monodisperse emulsion.

3. Shearing Conditions. The shearing device has a set of stators allowing a gap variation from 100 μm to 1 mm. We investigate the effect of the gap on fragmentation. By changing the gap, one also modifies the accessible range

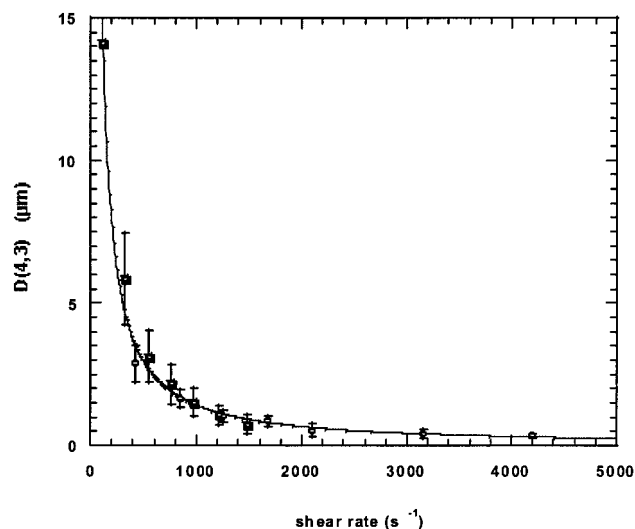


Figure 16. Influence of the gap on fragmentation of a premixed emulsion made of 55% Ifralan 205, $\Phi = 75\%$. Square, 1 mm gap; circle, 100 μm gap. The line is a guide to the eye.

of shear rates. We chose to report all the results obtained with the larger and smaller gaps on the same profile for comparison (Figure 16). Again, the error bars give the uniformity. As can be seen from this figure, the mean diameter is not affected by the gap size, but the fragmentation quality is strongly gap-dependent: an emulsion sheared in a 100 μm gap is more monodisperse than the same emulsion sheared in a 1 mm gap.

We also study the influence of the surface on emulsion fragmentation by comparing the profiles obtained either with polished stator and rotor or with sand-blasted surfaces. No noticeable difference on both the average diameter and polydispersity could be observed (Figure 17). These two effects (independent of both the gap and surface state on the droplets sizes) allow us to conclude that for our systems there is no noticeable slip at the Couette walls.

Conclusion

This experimental investigation, based on various systems, allows one to draw some general conclusions about emulsion fragmentation. (i) Elasticity of the premixed emulsion is absolutely necessary to get monodispersity, whatever its origin. As clearly shown, elasticity

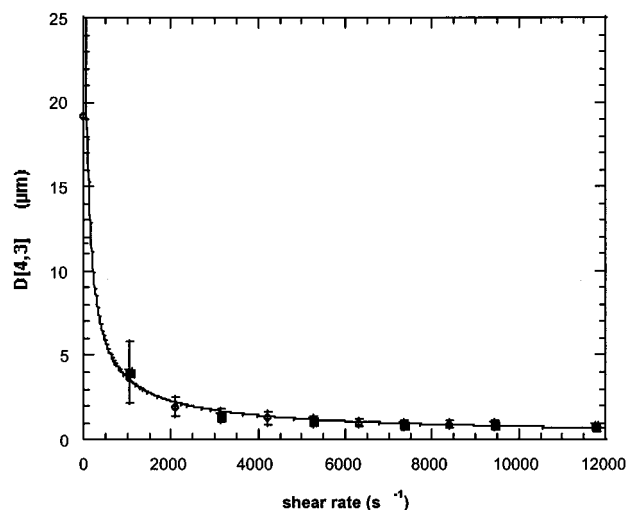


Figure 17. Influence of the surface roughness on fragmentation. Square, sand-blasted; circle, polished surfaces. The line is a guide to the eye.

may result either from the rheology of the continuous medium or from dense droplet packing. (ii) The applied shear rate must be well-controlled, as it is in the built Couette apparatus. Moreover, we found that the thinner the gap, the more monodisperse the final emulsion. This is likely due to heterogeneous flows that may develop in the gap. The residual polydispersity may hence be related to nonideal flow, even inside a 100- μm gap cell. Study of these possible heterogeneities is the key to understanding the fragmentation mechanism.

Once these two conditions are met, monodisperse emulsions are obtained. The final drop size depends on the applied shear rate, viscoelasticity of the premixed emulsion (controlled by both the surface-active agent concentration and dispersed phase volume fraction) and interfacial tension between the dispersed and dispersing fluids.

Finally, we showed that large quantities of monodisperse emulsions may be produced and that the drop size may be tuned from 0.3 to 10 μm . The homemade apparatus is now commercialized by TSR.

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