Experimental setup to study particle migration in a Couette cell with an optical method

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ABSTRACT

Understanding migration of particles in emulsions is essential for many industrial applications. One particular example from the petroleum industry is drilling fluids. We describe here an experimental setup which allows the visualisation of particles in a transparent emulsion in a Couette cell. When the internal cylinder of the Couette cell is rotating, we can measure the horizontal velocity field at each height in the Couette cell. When the emulsion is at rest, we can construct 3D picture of the emulsion to obtain the height and the position in the gap of each particle.

INTRODUCTION

Drilling fluid are often oil-in-water or water-in-oil emulsions embedded with dense barite particles. It is essential for well stability that they remain uniformly distributed when the fluid flows. Concentrated emulsions are yield stress fluids and when they are sheared, particle sediment due to gravity 1. Also, migration to the higher shear-rate regions 2 and alignments 3 can occur.

To study particle migration in an emulsion, Particle Image Velocimetry (PIV) is a potential technique. PIV is based on imaging tracer particles mixed in the fluid and cross-correlating the images to measure the particle displacement 4. It has been successfully implemented by several authors in a Couette geometry, for instance to study flow of particle suspensions 5 and wall slip in a polymeric yield stress fluid 6,7.

In this paper, we describe an experimental setup to study particle migration in an emulsion, in a Couette cell with PIV. We first describe the transparent emulsions and the devices. Then, we use PIV to study wall slip of emulsion on the solid surfaces and finally show how to obtain particle distribution in the Couette cell volume.

MATERIAL: EMULSION

We have chosen an emulsion preparation close to 8. Silicone oil (V350 provided by VWR) is mixed to an aqueous continuous phase (53wt% glycerol and 47wt% DI water, where an additional 3wt% of TTAB surfactant is dissolved). With this proportion of glycerol, optical indexes of the continuous phase and the oil are equal at 20°C and the emulsion is transparent. Details about how to formulate a transparent emulsion can be found in 9. We first prepare a mother emulsion with 82wt% of silicone oil with a Silverson emulsifier, by increasing the rotation velocity from 600 to 2400 rpm by steps of 600 rpm. Total mixing time is 110 min. The mother emulsion is stable for several months. For the tests described here, we dilute it with the same continuous phase to 65.4wt% of droplets and carry out the tests within few days after dilution to avoid emulsion drainage or coarsening.

To assess the size of the droplets, we have further diluted a drop of emulsion with a solution with no glycerol to change the optical index of the continuous phase, then we observed it with a microscope. Figure 1 shows an example of picture and a histogram of the droplet sizes. The average droplet radius, obtained from the analysis of these images, is about 1 µm.

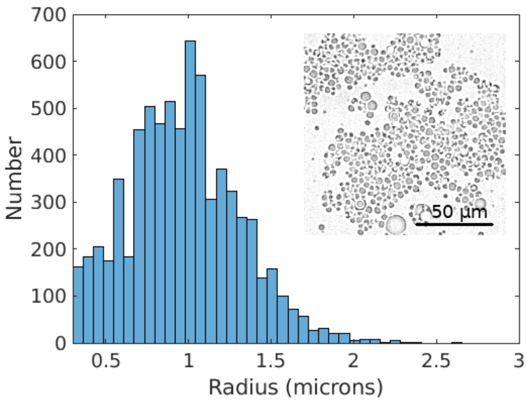

Figure 1. Histogram of the droplet size in the emulsion, obtained from a sample of 7000 droplets. Inset: A microscope picture illustrating the droplets.

EXPERIMENTAL SETUP

The experimental setup is schematically described in Figure 2. We use the Couette cell designed for PIV experiments by Anton Paar (C-LTD70/PIV) in a Anton Paar MRC 702 rheometer. The bob is black to avoid laser reflection, height is 16 mm and its diameter is 32 mm. The bottom of the rotating cylinder is hollow: the edges are 1.5mm lower than the bottom surface. The distance between the lower bob edge and the bottom of the cup is 2 mm. The default value is 5 mm, but we have chosen to decrease it to reduce attenuation of the light crossing the bottom part of the cell. The cup is transparent, liquid circulate between the double-wall to control temperature, and the internal diameter is 35 mm. We set the temperature to 20°C.

A plane of particles is illuminated by a horizontal laser (Nitron nanopiv laser) sheet of thickness 500 µm. A PCO Pixelfly Camera is coupled with a zoom lens at aperture 2 and a 0.5 magnifying lens from Navitar; the focal length with the lenses is about 20 cm. A mirror allows the observation of the particles from below the cell. We obtain pictures of a 4-mm long portion of the cell, with spatial resolution 340px/mm.

The camera, the mirror and the laser are fixed together on a vertical translation stage. We can therefore obtain images from the bottom to the top of the Couette cell.

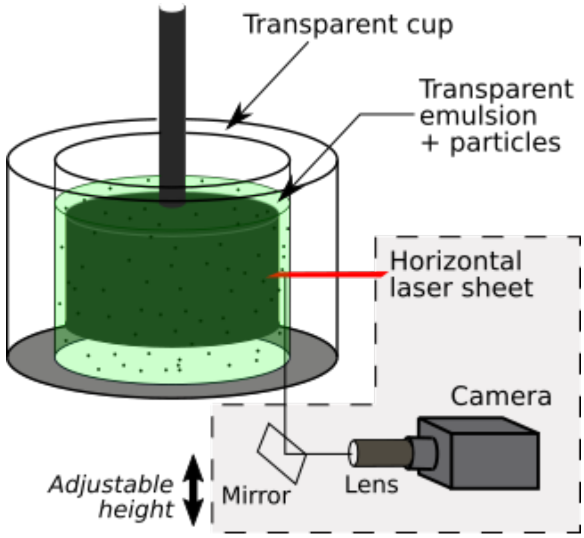

Figure 2. Schema of the setup.

In the following, we will describe two uses of this setup.

- At a fixed vertical height, we can measure the velocity field in the gap with PIV. This allows us in particular to measure the wall slip at the bob and cup surfaces. We use in this case neutrally-buoyant 5-µm particles.

- In the absence of shearing, we can take pictures from the top to the bottom of the cell to observe the distribution of the particles. This can be used to measure the sedimentation velocity of dense particles.

STUDY OF WALL SLIP

The surface of the bob and the cup are smooth, which can induce wall slip in emulsions 10. In order to use the setup to investigate the effect of shear on an emulsion with particles, we need to assess the conditions when wall slip occurs.

Flow curve

The first approach consist in studying the shape of the flow curve. Here, we have placed the emulsion without particles in the cup, pre-sheared it for 1 min at 100 s^{-1}, allowed stress relaxation for 30 s, and finally decreased the shear rate from 100 s^{-1} to 0.01 s^{-1}. The obtained curve is shown in Figure 3, it can be fitted by a Herschel-Bulkley behavior above 1 s^{-1}, which can suggest that the wall slip is limited in this range. This will be checked by the PIV experiment presented in the next section.

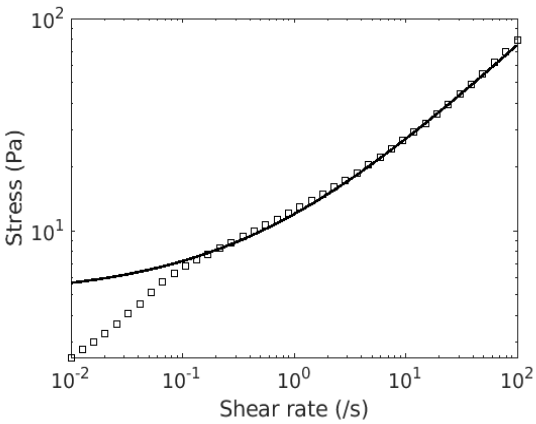

Figure 3. Flow curve of the transparent emulsion obtained wit the transparent Couette cell (squares) and fit with a Herschel-Bulkley model (stress in Pa and shear rate in s^{-1}):$\tau = 5 + 7\dot{\gamma}^{0.5}$.

PIV measurements

For PIV measurements, 5-microns neutrally-buoyant particles at concentration 2 g/L are added to the emulsion. A test showed that the flow curve from Figure 3 is not affected by the presence of particles. Tracer particles are not well dispersed, i.e. agglomerates can be observed, but this does not affect the PIV analysis.

For the PIV tests, two pictures of the samples are taken with a short time difference dt. dt ranges from 800 ms to 1 ms when the shear rate varies from 0.1 s^{-1} to 100 s^{-1}, it is selected so that the maximal displacement of a particle between the images is about 20 pixels. PIV is performed within the boundaries of the gap, the other parts of the images (cup wall and bob) being masked. PIV consists in dividing the first image of the pair into regions of interest, and identifying the location of each region in the next image by cross correlation 4. Here, regions of interests are 128 pixel square windows. To improve the accuracy of the measured velocity fields, cross-correlation is calculated from an ensemble of 80 pairs of images. An example of obtained velocity field is shown in Figure 4 at shear rate 100 s^{-1}. We observe in this case that, as expected, the velocities of the emulsion close to the rotating cylinder are higher than the velocities near the external wall.

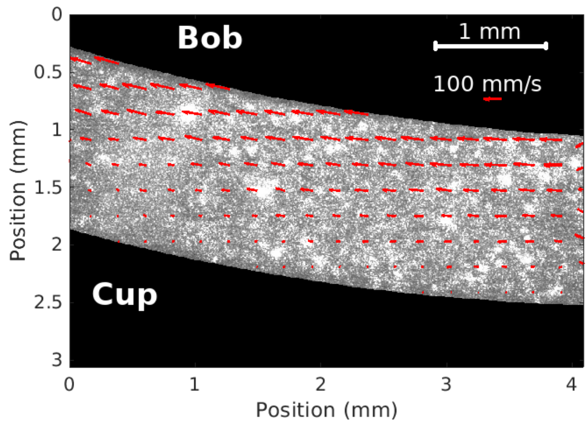

Figure 4. Velocity field (in red) obtained with PIV at 100 s^{-1}, superimposed with one of the corresponding pictures of the particles.

We have calculated the median value of the velocities at each position r (distance from the rotation axis) to obtain the velocity profiles from Figure 5. When the shear rate is very low (1 s^{-1}), the velocity is uniform: the emulsion does not flow, its moves as a plug at a smaller velocity than the bob surface. There is therefore wall slip on both the bob and the cup surfaces. At 0.3 s^{-1}, the velocity profiles shows that the emulsion flows close to the bob (r <16.8 mm) whereas the velocity profile is flat close to the cup: the emulsion is sheared only close to the bob. At high shear rate, emulsion is sheared in the whole gap. The velocity of the emulsion is smaller than the theoretical velocity for Newtonian fluids, indicating some slip at the surface of the rotating cylinder. The slip velocity at the bob wall is 20% of the velocity of the solid surface for shear rates for 1 s^{-1} to 100 s^{-1} (respectively solid velocities from 1.5 mm/s to 150 mm/s).

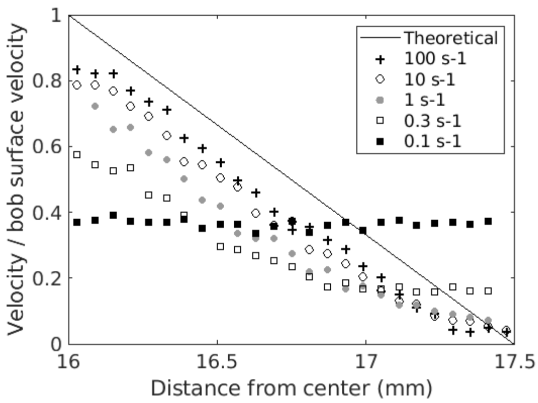

Figure 5. Reduced velocity profiles in the gap for five different shear rates.

The effective shear rate in the emulsion is calculated from the slope of the linear regression of the velocity profiles (see Table 1) in the central part of the gap (r between 16.25 and 17.25 mm). When the imposed shear rate is 100 s^{-1}, the measured value is the same. The relative difference increases when the shear rate decreases.

Imposed shear rate	Measured shear rate 16.25 < r < 17.25 mm
100 s^{-1}	101 s^{-1}
10 s^{-1}	9.4 s^{-1}
1 s^{-1}	0.80 s^{-1}
0.3 s^{-1}	0.15 s^{-1}
0.1 s^{-1}	9.10^{-4} s^{-1}

Table 1. Comparison of the imposed shear rates with the values obtained from the slope of the velocity profiles in the central part of the gap.

Finally, we have checked that the velocity profiles are not affected by the observation plane. Figure 6 shows that the same profiles are obtained at height from 0 to 10 mm at 10 s^{-1}. We were however not able to achieve PIV analysis at the top of the cell, because of light attenuation due to the presence of the particles. Using a smaller amount of particle should solve this problem.

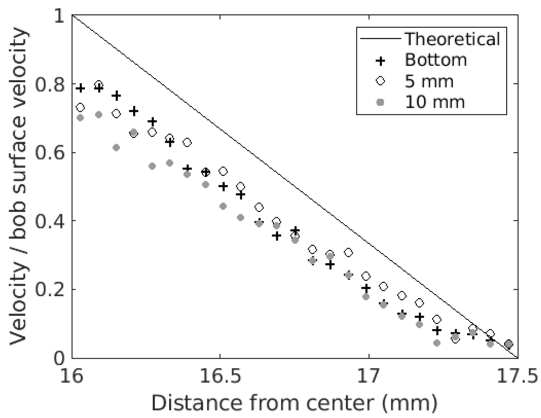

Figure 6. Reduced velocity profiles at 10 s^{-1}, for different observation plane height

To conclude this part, we are able to measure the presence of wall slip and the resulting effecting shear rates in the PIV cell. When the shear rate is above 1 s^{-1}, the effective shear rate is close to the imposed shear rate in the whole Couette cell, except close to the wall. Therefore, the set-up can be used to study particles suspension in the transparent emulsion when the shear rate is high enough.

3D RECONSTRUCTIONS OF PARTICLE DISTRIBUTION IN THE CELL

In this last part, we will describe our method to identify the position of particles in the volume of the cell. This can be used to study the migration of particles under shear.

The graphs of Figures 7 and 8 show results obtained with 40 μm diameter glass particles. We have also carried out similar tests with 100 μm glass particles. Glass particles are washed with acetone to remove impurities and isopropanol to remove traces of oil .

Pictures of the particles are taken when no shear is applied to the material. A sphere in a yield stress fluids at rest do not sediment as long as 11,12:

$$\tau_y > \frac{1}{21}\Delta\rho g d \quad (1)$$

, where$\Delta\rho$is the density difference between the particles and the fluid and d is the particle diameter. Therefore, glass particles (density difference about 1500 kg/m^3) of diameter below 100 μm do not sediment if the yield stress is above 0.7 Pa. The yield stress of the emulsion, obtained from the Herschel-Bulkley fit in Figure 3, is 5 Pa. Therefore, such particles do not sink at rest, so particle segregation under shear can be studied by stopping the bob rotation to take the images. For the same reason, the bubbles which are entrained in the emulsion during the preparation remain in the emulsion where it is at rest. We need to shear the fluid for a few minutes to make them rise to the top of the cell. The bubbles size is about ten times bigger than the particles, therefore, particle have hardly started to sediment when the bubbles are removed from the emulsion.

To scan the total height of the 16 mm high cell, we take 100 pictures separated by dz = 165 μm. Because of the thickness of the laser sheet, there is a superposition between two consecutive layers. Each particles appears therefore on 3 consecutive pictures, which ensures that no particle is missed.

The stack of images is then analysed with Matlab. We first binarize each of the 100 images of the scan and use imopen and imclose filters to remove isolated white and black pixels. We reconstruct a 3D image of dimensions 1392 px, 1040 px and 100 px, on the x, y and z axis respectively. Then watershed transform is performed on the 3D image to separate neighbour particles. Finally the objects in the 3D image are counted using the Matlab function “regionprops3”.

At this point, we have to mention that the size of the obtained objects is not representative of the size of the particles. First, diffraction makes the particles look bigger in the images than in the reality. In addition, due to the thickness of the laser sheet, the apparent size of the particles in the vertical direction is also increased.

However, the position of the center of the objects is the position of the center of the particles. By using particle with known diameter, we can reconstruct the particle distribution in the cell, as shown for example in Figure 7, where each identified object has been replaced by a 40 μm sphere.

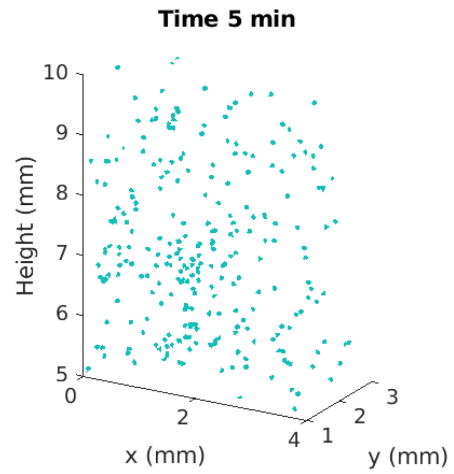

Figure 7. Example of a portion of 3D reconstruction of a suspension of 40 µm particles, after 5 min shearing at 100 s^{-1}.

Knowing the position of each particle, we can plot the vertical or radial distribution, as shown as an example in Figure 8. We see in this graph that the amount of particles at the top of the cell decreases when shearing time increases.

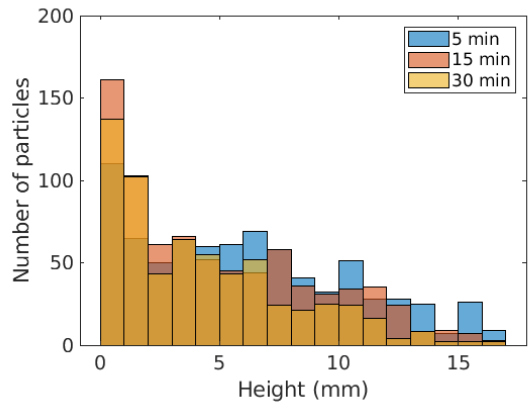

Figure 8. Histogram of the vertical distribution of 40 µm diameter particles after shearing at 100 s-1. Note that the total number of particles is not constant because particles can fall in the emulsion layer below the bob layer.

CONCLUSIONS AND PERSPECTIVES

The described setup consists in a transparent Couette cell, a transparent emulsion and a camera coupled with an horizontal laser sheet for the visualisation of particles.

With PIV, with observe that wall slip occurs at the wall of the smooth Couette geometry. Plug flow occurs when the applied macroscopic shear rate is too low (< 1 s^{-1}). When the applied shear rate is higher, the emulsion flows, and we can obtain from the PIV measurements the effective shear rate. The effective shear rates does not depend on the vertical position of the observation plane.

Finally, we show that we can use the same setup to scan the height of a portion of Couette cell and obtain the 3D distribution of particles in the gap. This can be used to observe particle migrations in the emulsion.

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