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Layer-formation of non-colloidal suspensions in a parallel plate rheometer under steady shear

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ABSTRACT

Suspensions are subject to confinement induced structuring, i.e. layering, at the confining surfaces. While most of the previous work focused on layering in Couette cells, the present study aimed to characterize the resulting layers at the plates of a parallel plate rheometer with regard to their relative particle concentration. The particle concentration profile over the radial distance was characterized for various mean concentrations and gap heights. To this end, we mapped the distribution of fluorescently dyed tracer particles in density and refractive index matched suspensions. The results indicate that layering at the surfaces stabilizes as the ratio between gap height and particle diameter increases. For lower gap heights, i.e. as the suspension approaches a two dimensional state, the layer concentration was non-uniform over the plates. In general, results were quantitatively different for the upper and lower plate and the concentration profiles were noticeably asymmetric. We conclude that this is probably the result of the rheometer loading or the start-up process. The stable layers as well as the inhomogeneous particle distribution in general offer an explanation for the lack of transferability of viscosimetric results between different setups.

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1. Introduction

Accurate knowledge of a fluid's viscosity is of great importance to many applications. The measurement of the viscosity of suspensions and other complex fluids is, however, frequently impeded by changes of the microstructure that are caused by the geometry of the measurement device. Some of these microstructural effects, such as particle migration, are well understood and have been studied in great detail [1]. Others such as confinement induced layering appear to be general features of complex fluids [2] but are usually not considered for non-colloidal suspensions. While the consequences of such confinement related effects are often recognized (see for example [3–5]), the underlying processes at the confining surface have rarely been studied. The result of the failure to account for these effects is that even well established models often fail to represent simple experiments accurately, see for example [6].

One of the earliest accounts of layering was provided by Abbott et. al in 1991 [7] when they combined NMR imaging with a Couette cell. Husband et al. [8] reported in 1994 that the particles are arranged in a hexagonal pattern in these layers at the walls of this device. Little progress has been made since then. The current knowledge of these processes was mainly derived from simulation of Couette cells, which were also used in the earliest experimental studies, or inferred from indirect measurements. Most of these studies reported a lower concentration limit for strong ordering at the walls between 48% and 50% [9–11]. The resulting layers are mostly found to be of a hexagonal structure [8,10,12]. This layering is not always of equal strength at both confining surfaces [13]. Gondret [14] reported that, like other structuring phenomena, layering benefits from monodispersity.

Studies of shear flow between rotating parallel plates, both experimental [15] and by simulation [16], show confinement induced effects even in dilute suspension if the ratio of gap width to particle diameter is reduced far enough. Since the confinement influence is probably quantitatively different at parallel plates, as the circumferential speed is not constant on the surface, these results do not necessarily contradict the simulations of Couette cells. The simulation of Couette flow by Yeo and Maxey [9] revealed that a structured layer forms at the confining surface and a homogeneous core is present, separated from the structured layers by a transitional zone. Blanc et al. [17] found a similar effect in their experimental investigation of a suspension's microstructure in a Couette cell. While more than one parameter, such as particle roughness [18] or shear rate [19], can explain the formation of layers at confining surfaces, the range of the wall influence into the bulk appears to be limited [9]. The wall layers are independent of the

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gap to particle size ratio, but depend on particle volume fraction, if a critical ratio between gap size and particle radius of approx. 30 [20] is exceeded. In any case, layering is the result of local interactions. The volume fraction dependence also leads to a strong dependence on particle migration [4] and, therefore, the shear history of the suspension [12]. The formation process of layers may be considered a form of particle migration. It therefore shares many of the properties associated with the migration of particles in a suspension's bulk. Among these characteristics is a lack of susceptibility to shear reversal reported by Petit and Noettinger [21] and Gondret and Petit [22], such as is present in oscillatory tests.

The parallel plate geometry is extremely important for rheological tests of high viscosity fluids and suspensions. However, the formation of layers at the plate's surfaces has not been studied in detail under steady shear flow. Neither have previous studies shown the relationship between layer formation and distance from the rotation axis nor has any study been done that penetrated the suspension beyond the first layer of particles. It is extremely unlikely that the layering behavior should be the same between parallel plates as in the more widely studied Couette device, mainly because the velocity is constant over the confining surfaces in a Couette device but not over rotating parallel plates.

The aim of this work was to characterize the layering of non-colloidal suspensions in a parallel plate geometry. Particularly, the development of such layers as a function of the ratio between gap height and particle diameter as well as the influence of the bulk concentration is investigated.

2. Materials and methods

2.1. Materials

In the present study, we used poly(methyl methacrylate) (PMMA) particles, trade name Degacryl M449, kindly provided by Evonik Industries. The diameter range was 100 to 125 μm obtained via air jet sieving. The matrix fluid was chosen so as to closely match both the refractive index ($n_{D20} = 1.4900$) and density ($\rho = 1190$ kg/m^3) of the particles at 20.5 °C. While there are many index matching fluids described in the literature [23], we chose a mixture of 54.76 wt% Triton-X100, 25.23 wt% poly(ethylene glycol) 400 and 20.01 wt% of a 60.00 wt% solution of sodium iodine in deionized water. The dynamic viscosity of the resulting Newtonian fluid was $\eta_0 = 0.908$ Pa · s. The fluid matched the particle's properties within very narrow tolerances, $\Delta n_{D20} \approx 3 \cdot 10^{-4}$ and $\Delta\rho/\rho \approx 2 \cdot 10^{-3}$, as determined from the settling velocity of a dilute suspension. Since these properties are never perfectly uniform in a given batch of particles, further reduction of these tolerances was not feasible. While the refractive index matching provided optical transparency, the main purpose of the density matching was to prevent sedimentation and inertia related effects. A density difference between the dispersed phase and the fluid is also one of the main causes of yield stresses [24]. Furthermore, the high ion content of the fluid shields electrostatic charges of the particles that may otherwise contribute to a yield stress [25]. Weak interactions between particles were not expected to cause a noticeable yield stress for particle sizes used in the present study [26]. The influence of the fluid's surface tension on the yielding behavior was also negligible as the mean particle volume fraction was not close enough to the maximum packing fraction [24,27] in any of our experiments. All samples were sonicated to remove air bubbles.

We obtained the tracer particles necessary for optical flow visualization by dyeing a fraction of the particles with Rhodamine B. To this end, 0.75 g/l of the dye were dissolved in ethanol at 60 °C. In this fluid, 20.0 g/l of particles were stirred for one hour. The fluid was then removed by filtration, we rinsed the particles with pure ethanol. The standard concentration of these tracer particles was 0.2% in our samples. The transparency of a suspension deteriorates quickly with increasing particle content. To compensate for this, we lowered the tracer concentration for the most highly filled samples; the number of images was increased proportionally.

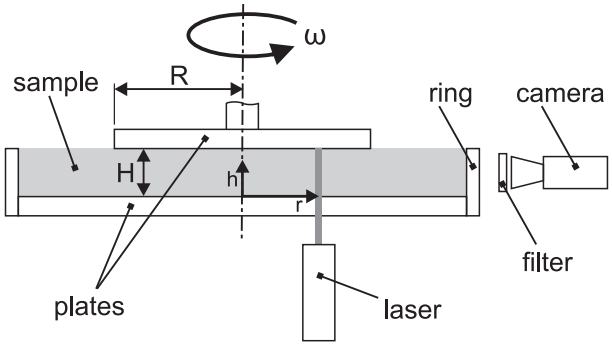

Fig. 1. Schematic illustration of the set-up.

2.2. Setup

The samples were subjected to a steady shear rate in the parallel plate setup shown in Fig. 1 at 20.5 ± 0.5°C. The diameter of the upper and lower plate was 25.0 mm and 45.6 mm, respectively. These were mounted to an Anton Paar MCR501 stress controlled rheometer. The surrounding ring was planar at one side perpendicular to the optical axis of the camera to allow for observation of the sample without optical distortion. Preliminary experiments showed that the distance between the outer edge of the upper plate and the ring was large enough to preclude any influence of the ring on the velocity field between the plates. Both the ring and the lower plate were made of PMMA, whereas the upper plate was covered with a smooth black surface that sufficiently inhibited reflection. The laser with light sheet generating lenses, Blau Optoelektronik MVNano DL 520 nm 30 mW, illuminated a plane corresponding to a specified radial distance at which the CCD camera DantecDynamics EO 4M recorded the light of the fluorescent particles. A long pass edge filter, cut-off wavelength $\lambda_c = 536.4$ nm, was placed in front of the camera to ensure that only the fluoresced light reached the sensor. The window observed by the camera spanned the entire gap height H and was 2.00 mm wide. From these dimensions we estimated the maximum difference between the radial distance of two particles in any image. It was 0.10 mm $< \Delta r <$ 0.18 mm, i.e. approximately equal to the particle diameter. Several radii and gap widths as well as various apparent shear rates were studied; these are denoted in the corresponding diagrams. All experiments were repeated three times, error bars in the diagrams indicate the respective standard deviation.

2.3. Concentration evaluation

A well-established method for the determination of concentration profiles is to dye the matrix fluid instead of a fraction of the particles [28]. The resulting images show all the particles that are intersected by the light sheet, provided the light sheet thickness is much smaller than the particle diameter. The inherent errors of this method were discussion in detail by Blanc et al. [17]. However, since the average distance between particles is small, this technique is vulnerable to motion blur that may occur at higher velocities. To ensure accurate measurements of the concentration profiles at higher shear rates, we instead mapped the density of the tracer particles. Since the tracer particles were from the same batch as the non-dyed particles they were characterized by the

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