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## Flows and heterogeneities with a vane tool: MRI measurements

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#### Synopsis

We study the local flow properties of various materials in a vane-in-cup geometry. We perform velocity and particle concentration measurements through MRI techniques during the flows of a Newtonian fluid, a yield stress fluid, and a dense suspension of noncolloidal particles in a yield stress fluid. In the Newtonian fluid, we observe that the  $\theta$ -averaged strain rate component  $d_{r\theta}$  decreases as the inverse squared radius in the gap, in agreement with a Couette analogy. However, this results in a torque significantly higher (by 12%) than the Atkinson and Sherwood (1992) prediction, which is strictly valid only for blades of negligible thickness embedded in an infinite medium. We also observe that the flow enters deeply the region between the blades, leading to a significant extensional flow. In the yield stress fluid, in contrast with the usually accepted picture based on simulation results from the literature, we find that the layer of material that is sheared near the blades at low velocity is not cylindrical. There is thus a significant extensional component of shear that should be taken into account in the analysis. Finally and surprisingly, in the suspension, we observe that a thin non-cylindrical slip layer made of the pure interstitial yield stress fluid appears quickly at the interface between the sheared material and the material that moves as a rigid body between the blades. This feature can be attributed to the non-symmetric trajectories of the noncolloidal particles around the edges of the blades. This new important observation is in sharp contradiction with the common belief that the vane tool prevents from slippage and tends to preclude the use of the vane tool for studying the flows of pasty materials.

## I Introduction

Experimental investigations of the rheological behavior of concentrated suspensions often involve a vane-in-cup geometry (see Barnes and Nguyen (2001) for a review). As compared to other classical geometries, the vane tool offers two main advantages. First, it allows studying the properties of structured materials with minimal disturbance of the material structure during the insertion of the tool [Dzuy and Boger (1983); Alderman *et al.* (1991)]. It is thus widely used to study the properties of gels and thixotropic materials [Alderman *et al.* (1991); Stokes and Telford (2004)] and for in situ study of materials as e.g. in the context of soil mechanics [Richards (1988)]. Second, it is supposed to avoid wall slip [Keentok (1982); Dzuy and Boger (1983); Saak *et al.* (2001)], which is a critical feature in dense suspensions [Coussot (2005)]; the reason for this belief is that the material sheared in the gap of the geometry is sheared by the (same) material that is trapped between the blades. Consequently, it is widely used to study the behavior of pasty materials containing large particles as fresh concrete [Koehler *et al.* (2006); Estelle *et al.* (2008); Wallevik (2008); Jau and Yang (2010)] and foodstuff [Stokes and Telford (2004); Martínez-Padilla and Rivera-Vargas (2006)].

The constitutive law of materials can be obtained from a rheological study with the vanein-cup geometry provided one knows the coefficients – called "geometry factors" – that allow

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translating the raw macroscopic data (torque, rotational angle or velocity) into local data (shear stress, shear strain or shear rate). However, in contrast with other classical geometries, even the *a priori* simple linear problem (for Hookean or Newtonian materials) is complex to solve with a vane tool. This linear problem was studied theoretically by Sherwood and Meeten (1991) and Atkinson and Sherwood (1992) in the general case of a *n*-bladed vane tool embedded in an infinite linear medium. The analytical expression found for the torque vs. rotational velocity is in rather good agreement with macroscopic experimental data [Sherwood and Meeten (1991)]. Note however two possible shortcomings of this theoretical approach for its use in practice: the blades are infinitely thin and there is no external cylinder.

There is no such approach in the case of nonlinear media (*i.e.* complex fluids). A practical method used to study the flow properties of non-linear materials, known as the Couette analogy [Bousmina *et al.* (1999); Aït-Kadi *et al.* (2002); Estelle *et al.* (2008)], consists in calibrating the geometry factors with Hookean or Newtonian materials. One defines the equivalent inner radius  $R_{i,eq}$  of the vane-in-cup geometry as the radius of the inner cylinder of a Couette geometry that would have the same geometry factors for a linear material. For any material, all macroscopic data are then analyzed as if the material was sheared in a Couette geometry of inner cylinder radius  $R_{i,eq}$ . The nonlinearity (that effects the flow field) is sometimes accounted for as it is in a standard Couette geometry [Estelle *et al.* (2008)]. This approach may finally provide constitutive law measurements within a good approximation [Baravian *et al.* (2002)].

However, simulations and observations show that  $R_{i,eq}$  is not a universal parameter of the vane tool independent of the properties of the studied material. While the streamlines go into the virtual cylinder delimited by the blades in the case of Newtonian media [Baravian et al. (2002)], yielding an equivalent radius lower than the blade radius [Sherwood and Meeten (1991); Atkinson and Sherwood (1992), it was found from simulations [Barnes and Carnali (1990); Savarmand et al. (2007)] that the streamlines are nearly cylindrical everywhere for shear-thinning fluids if their index n is of order 0.5 or less, and thus that  $R_{i,eq} = R_i$  in these cases. Moreover, for yield stress fluids, simulations and photographs of the shearing zone around a 4-bladed vane rotating in Bingham fluids [Keentok et al. (1985)], simulations of Herschel-Bulkley and Casson fluids flows in a 4-bladed vane-in-cup geometry [Yan and James (1997)], and simulations of Bingham fluids flows in a 6-bladed vane-in-cup geometry [Savarmand et al. (2007)], all show that at yield (*i.e.* at low shear rates), the material contained in the virtual cylinder delimited by the blades rotates as a rigid body, and that it flows uniformly in a thin cylindrical layer near the blades. This is now widely accepted [Barnes and Nguyen (2001)] and used to perform a Couette analogy with  $R_{i,eq} = R_i$ ; the yield stress  $\tau_y$  is then simply extracted from torque T measurements at low velocity thanks to  $\tau_y = T/(2\pi H R_i^2)$ , where H is the vane tool height (neglecting end effects) [Nguyen and Boger (1992)].

The flow field in a vane-in-cup geometry and its consequences on the geometry factors have thus led to many studies. However, only theoretical calculations, macroscopic measurements and simulation data exist in the literature: there are no experimental local measurements of the flow properties of Newtonian and non-Newtonian materials induced by a vane tool except the qualitative visualization of streamlines made by Baravian *et al.* (2002) for Newtonian media, and the photographs of Keentok *et al.* (1985) for yield stress fluids. Moreover, while the main advantage of the vane tool is the postulated absence of wall slip, as far as we know, this widely accepted hypothesis was neither investigated in depth nor criticized. In order to provide such local data, we have performed velocity measurements during the flows of a Newtonian medium and of a yield stress fluid in both a coaxial cylinder geometry and a vane-in-cup geometry. We have also performed particle concentration measurements in a dense suspension of noncolloidal particles in a yield stress fluid, which is a good model system for complex pastes as fresh concrete [Mahaut *et al.* (2008a,b)]. Our main results are that:

(i) in the Newtonian fluid, the  $\theta$ -averaged strain rate component  $d_{r\theta}$  decreases as the inverse squared radius in the gap, in agreement with the Couette analogy, although our results

differ significantly from the Atkinson and Sherwood (1992) theory; the flow enters deeply the region between the blades, leading to a significant extensional flow;

- (ii) in the yield stress fluid, in contrast with results from the literature, the layer of material that is sheared near the blades at low velocity does not have a cylindrical shape;
- (iii) in the suspension of noncolloidal particles in a yield stress fluid, the noncolloidal particles are quickly expelled from a thin zone near the blades, leading to the development of a thin slip layer made of the pure interstitial yield stress fluid, in sharp contradiction with the common belief that the vane tool prevents from slippage.

In Sec. II, we present the materials employed and the experimental setup. We present the experimental results in Sec. III: velocity profiles obtained with a Newtonian oil and with a yield stress fluids are presented in Sec. IIIA and Sec. IIIB, while Sec. IIIC is devoted to the case of suspensions, with a focus on the slip layer created by a shear-induced migration phenomenon specific to the vane tool.

## **II** Materials and methods

## A Materials

We study three materials: a Newtonian fluid, a yield stress fluid, and a dense suspension of noncolloidal particles in this yield stress fluid.

The Newtonian fluid is a silicon oil of 20 mPa.s viscosity.

The yield stress fluid is a dense water in oil emulsion. The continuous phase is dodecane oil in which Span 80 emulsifier is dispersed at a 7% concentration. A 100 g/l CaCl<sub>2</sub> solution is then dispersed in the oil phase at 6000 rpm during 1 hour with a Sliverson L4RT mixer. The droplets have a size of order 1  $\mu$ m from microscope observations. The droplet concentration is 75%, and the emulsion density is  $\rho_f = 1.01 \text{ g cm}^{-3}$ . The emulsion behavior, measured through coupled rheological and MRI techniques described in Ovarlez *et al.* (2008), was well fitted to a Herschel-Bulkley behavior  $\tau = \tau_y + \eta_{HB}\dot{\gamma}^n$  of yield stress  $\tau_y = 22.5$  Pa, consistency  $\eta_{HB}=5.3$  Pa s<sup>0.5</sup>, and index n = 0.5.

The suspension is a suspension of monodisperse polystyrene beads (density  $\rho_p = 1.05 \text{ g cm}^{-3}$ , diameter  $d = 250 \ \mu\text{m}$ ) suspended at a 40% volume fraction in the dense emulsion described above. The density matching between the particles and the yield stress fluid is sufficient to prevent from shear-induced sedimentation of the particles in the yield stress fluid [Ovarlez *et al.* (2010)]; in all experiments, we check that the material remains homogeneous in the vertical direction thanks to MRI density measurements.

## **B** Rheometry

The rheometric experiments are mainly performed within a 6-bladed vane-in-cup geometry (inner cylinder radius  $R_i = 4.02$  cm, outer cylinder radius  $R_o = 6$  cm, height H = 11 cm). The blades thickness is 6 mm. Other experiments are performed with a wide-gap Couette geometry of slightly different inner cylinder radius  $R_i = 4.15$  cm, due to the presence of sandpaper (the other dimensions were identical). The inner cylinder of the Couette geometry, and the outer cylinder of both geometries are covered with sandpaper of roughness equivalent to the size of the largest elements of the materials studied in order to avoid wall slip.

In the rheometric experiments presented here, we control the rotational velocity of the inner cylinder, with values ranging from 0.1 to 100 rpm.

#### C MRI

Proton MRI [Callaghan (1991)] was chosen as a non-intrusive technique in order to get measurements of the local velocity and of the local bead concentration inside the sample. Experiments are performed on a Bruker 24/80 DBX spectrometer equipped with a 0.5T vertical superconductive magnet with 40 cm bore diameter and operating at 21 MHz (proton frequency). We perform our experiments with a home made NMR-compliant rheometer, equipped with the geometries described in the previous section. This device was already used in a number of previous rheo-nmr studies [Raynaud *et al.* (2002); Rodts *et al.* (2004); Ovarlez *et al.* (2006)], and is fully described in Raynaud *et al.* (2002). The volume imaged is a (virtual) rectangular portion of 40 mm in the axial (vertical) direction with a width (in the tangential direction) of 10 mm and a length of 70 mm (in the radial direction, starting from the central axis). This volume is situated at the magnet center (so as to damp the effects of field heterogeneities) and sufficiently far from the bottom and the free surface of the rheometer so that flow perturbations due to edge effects are negligible. We checked that the velocity and concentration profiles are homogeneous along the vertical direction in this slice.

Details on the sequence used to obtain velocity profiles can be found in [Raynaud et al. (2002); Rodts et al. (2004)]. While it is possible to get 2D or 3D maps of 2D or 3D velocity vectors [Rodts et al. (2004), such measurements may actually take minutes and imply complex synchronization of the MRI sequences and of the geometry position. However, although there is generally no orthoradial invariance of flow with the vane-in-cup geometry, we will show in the following that the orthoradial velocity alone provides a valuable information that can be sufficient for most analyses; in particular it allows computing the  $\theta$ -averaged strain rate component  $\bar{d}_{r\theta}$  and thus the  $\theta$ -averaged shear stress  $\bar{\tau}_{r\theta}$  (and the torque T) in the case of the Newtonian oil. That is why we have chosen to limit ourselves to 1D profiles of 1D velocity measurements, namely the orthoradial velocity  $V_{\theta}(r,t)$  as a function of the radius r and time t, for which a single measurement may take as less as 1s; this has allowed us to perform a sufficient amount of experiments, with various materials, geometries, and rotational velocities. Depending on the time over which this measurement is averaged as compared to  $2\pi/(N\Omega)$  where N=6 is the number of blades and  $\Omega$  is the rotational velocity, this measurement may provide either a time (or  $\theta$ )-averaged orthoradial velocity  $\bar{V}_{\theta}(r) = (1/2\pi) \int_{0}^{2\pi} V_{\theta}(r, \theta) d\theta$  or an instantaneous (transient) orthoradial velocity  $V_{\theta}(r,t)$ . In this latter case, the  $\theta$  dependence of the orthoradial velocity  $V_{\theta}(r,\theta)$  at a given radius r can then be easily reconstructed by simply replacing the time t dependence by an angular  $\theta$  dependence with  $\theta = \Omega t$ . It should also be noted that due to incompressibility of the materials we study, the  $V_r(r,\theta)$  field can be reconstructed thanks to  $(1/r)\partial_r(rV_r) + (1/r)\partial_\theta(V_\theta) = 0$  with  $V_r(R_o,\theta) = 0$ ; however, this derivation from the experimentally measured values of  $V_{\theta}(r,\theta)$ cannot be very accurate. Finally, from  $V_r(r,\theta)$  and  $V_{\theta}(r,\theta)$ , we are also able to evaluate the strain rate components  $d_{rr}(r,\theta) = -d_{\theta\theta}(r,\theta) = \partial_r V_r$  and  $d_{r\theta}(r,\theta) = (1/2) [(1/r)\partial_{\theta}(V_r) + r\partial_r (V_{\theta}/r)].$ 

The NMR sequence used in this work to measure the local bead concentration is a modified version of the sequence aiming at measuring velocity profiles along one diameter in Couette geometry [Hanlon *et al.* (1998); Raynaud *et al.* (2002)], and is described in full details in Ovarlez *et al.* (2006). The basic idea is that during measurements, only NMR signal originating from those hydrogen nuclei belonging to the liquid phase of the sample (*i.e.* both the oil and water phase of the emulsions) is recorded: the local NMR signal that is measured is thus proportional to  $1 - \phi$  where  $\phi$  is the local particle volume fraction. A rather low absolute uncertainty of  $\pm 0.2\%$  on the concentration measurements values was estimated in Ovarlez *et al.* (2006).

## **III** Experimental results

In this section, we study successively the flow properties of the Newtonian oil, the yield stress fluid, and the suspension.

#### A Newtonian fluids

In this section, we study the flows observed with a Newtonian fluid. We first present a basic theoretical analysis of the flows in a vane-in-cup geometry as compared to flows in a standard Couette geometry, which provides the basis for a Couette analogy. The  $\theta$ -averaged orthoradial profiles  $\bar{V}_{\theta}(r)$  are then shown, and are compared to predictions of the Couette analogy. The full velocity field  $V_{\theta}(r, \theta)$ ,  $V_r(r, \theta)$  is finally presented and analyzed.

#### 1 Couette analogy: theoretical analysis

Throughout this paper, we use cylindrical coordinates  $(r, \theta, z)$ . All flows are supposed to be z invariants (*i.e.* there are no flow instabilities). We define the  $\theta$ -average  $\bar{f}(r)$  of a function  $f(r, \theta)$  as  $\bar{f}(r) = (1/2\pi) \int_0^{2\pi} f(r, \theta) d\theta$ .

The stress balance equation projected along the orthoradial axis reads:

$$(1/r)\partial_r(r^2\tau_{r\theta}) + \partial_\theta(\tau_{\theta\theta}) - \partial_\theta p = 0 \tag{1}$$

where  $\tau_{ij}$  is the deviatoric stress tensor and p the pressure.

The strain rate tensor component  $d_{r\theta}$  reads:

$$d_{r\theta}(r,\theta) = \frac{1}{2} \Big( (1/r)\partial_{\theta}(V_r) + r\partial_r(V_{\theta}/r) \Big)$$
(2)

We recall that the constitutive law of a Newtonian fluid of viscosity  $\eta$  reads:

$$\tau_{ij} = 2\eta d_{ij} \tag{3}$$

#### Couette geometry

In a standard – coaxial cylinders – Couette geometry, due to cylindrical symmetry, Eq. 1 reads  $\partial_r(r^2\tau_{r\theta}) = 0$  which means that the whole shear stress distribution  $\tau_{r\theta}(r)$  in the gap is known whatever the constitutive law of the material is. If a torque  $T(\Omega)$  is exerted on the inner cylinder driven at a rotational velocity  $\Omega$ ,  $\tau_{r\theta}(r)$  reads:

$$\tau_{r\theta}(r) = \frac{T(\Omega)}{2\pi H r^2} \tag{4}$$

For a Newtonian fluid of viscosity  $\eta$ , it follows that the strain rate component  $d_{r\theta}(r)$  reads:

$$d_{r\theta}(r) = \frac{T(\Omega)}{\eta 4\pi H r^2} \tag{5}$$

As Eq. 2 reads  $d_{r\theta}(r) = (1/2) r \partial_r (V_{\theta}/r)$  with cylindrical symmetry, due to the boundary conditions  $V_{\theta}(R_i) = \Omega R_i$  and  $V_{\theta}(R_o) = 0$ , from  $\int_{R_o}^{R_i} d_{r\theta}(r)/r \, dr = \Omega$ , one gets alternatively  $d_{r\theta}(r) = \Omega R_i^2 R_o^2/[r^2(R_o^2 - R_i^2)]$ . The orthoradial velocity profile then reads:

$$V_{\theta}(r) = \Omega \frac{R_i^2}{r} \frac{R_o^2 - r^2}{R_o^2 - R_i^2}$$
(6)

Finally, the viscosity  $\eta$  of a Newtonian fluid is obtained from the measured torque/rotational velocity relationship  $T(\Omega)$  through

$$\eta = \frac{T(\Omega)}{\Omega} \frac{R_o^2 - R_i^2}{4\pi H R_o^2 R_i^2} \tag{7}$$

These equations will be used for the comparison with the flows observed in a vane-in-cup geometry, in particular to determine the radius  $R_{i,eq}$  of the equivalent Couette geometry.

#### Vane-in-cup geometry

In a vane-in-cup geometry, there is a priori no cylindrical symmetry and all quantities a priori depend on  $\theta$ . However, averaging Eq. 1 over  $\theta$  yields  $(1/r^2)\partial_r(r^2\bar{\tau}_{r\theta}) = 0$ . This means that what is true in a Couette geometry,  $\tau_{r\theta}(r) = \tau_{r\theta}(R_i)R_i^2/r^2$ , is still true on average with a vane-in-cup geometry:  $\bar{\tau}_{r\theta}(r) = \bar{\tau}_{r\theta}(R_i)R_i^2/r^2$  independently of the material's constitutive law. Note that this derivation is true only between  $R_i$  and  $R_o$ ; this is not true for the material between the blades as the unknown  $\tau_{ij}$  distribution in the blades contributes to the  $\theta$ -average. The link between this stress distribution and the torque  $T(\Omega)$  exerted on the vane tool may then seem difficult to build. However, it can be equivalently computed on the outer cylinder where it reads  $T = \int_0^{2\pi} \tau_{r\theta}(R_o, \theta) H R_o^2 d\theta = 2\pi H R_o^2 \bar{\tau}_{r\theta}(R_o)$ . This means that Eq. 4 is still valid for the  $\theta$ -averaged shear stress in the vane-in-cup geometry:

$$\bar{\tau}_{r\theta}(r) = \frac{T(\Omega)}{2\pi H r^2} \tag{8}$$

From the  $\theta$ -averaged Eq. 3, this means that the  $\theta$ -averaged strain rate distribution in a Newtonian fluid reads:

$$\bar{d}_{r\theta}(r) = \frac{T(\Omega)}{\eta 4\pi H r^2} \tag{9}$$

As the  $\theta$ -averaged Eq. 2 reads  $\bar{d}_{r\theta} = (1/2) r \partial_r (\bar{V}_{\theta}/r)$ , this means that the  $\theta$ -averaged orthoradial velocity profile of a Newtonian fluid of viscosity  $\eta$  in a vane-in-cup geometry, with a boundary condition  $\bar{V}_{\theta}(R_o) = 0$  reads:

$$\bar{V}_{\theta}(r) = \frac{T(\Omega)}{\eta} \frac{R_o^2 - r^2}{4\pi H R_o^2 R_i^2}$$
(10)

Finally, the only difference with a standard Couette flow, as regards these  $\theta$ -averaged quantities, is that we do not know the value of  $\bar{V}_{\theta}(R_i)$ ; we only know that  $V_{\theta}(r, 2\pi k/n) = \Omega R_i$ , for k integer, where n is the number of blades. This means in particular that  $\bar{d}_{r\theta}(r)$  and  $\bar{V}_{\theta}(r)$  do not follow the same scaling with  $\Omega$  as in the standard Couette geometry, whereas they follow the same scaling with r.

Nevertheless, these equations provide a new insight in the Couette analogy. The usual way of performing the Couette analogy consists in defining the radius of the equivalent Couette geometry  $R_{i,eq}$  as the radius that allows measuring the viscosity  $\eta$  of a Newtonian fluid with the standard Couette formula. From Eq. 7,  $\eta$  should then be correctly obtained from the torque/rotational velocity relationship  $T(\Omega)$  measured in a vane-in-cup geometry with:

$$\eta = \frac{T(\Omega)}{\Omega} \frac{R_o^2 - R_{i,eq}^2}{4\pi H R_o^2 R_{i,eq}^2} \tag{11}$$

Here, from Eqs. 10 and 6, we see that from the local flow perspective, there is a Couette analogy in the sense that the  $\theta$ -averaged orthoradial velocity (and shear) profiles will be exactly the same as in a Couette geometry. This defines a radius  $R_{i,eq}$  of the equivalent Couette geometry, such that  $\bar{V}_{\theta}(r)$  and  $\bar{d}_{r\theta}(r)$  read:

$$\bar{V}_{\theta}(r) = \Omega \frac{R_{i,eq}^2}{r} \frac{R_o^2 - r^2}{R_o^2 - R_{i,eq}^2}$$
(12)

$$\bar{d}_{r\theta}(r) = \Omega \frac{R_{i,eq}^2 R_o^2}{r^2 (R_o^2 - R_{i,eq}^2)}$$
(13)

This point of view provides an additional meaning to the Couette analogy, namely the similarity of the average flows, and offers a more accurate experimental mean to determine  $R_{i,eq}$  than calibration: in rheological measurements, the  $T(\Omega)$  relationship has to be corrected from end effects [Sherwood and Meeten (1991)] whereas the  $\bar{V}_{\theta}(r)$  or  $\bar{d}_{r\theta}(r)$  measurements provide directly the value of  $R_{i,eq}$  without any correction. This will be illustrated in the following.

#### 2 $\theta$ -averaged profiles

We first study the  $\theta$ -averaged orthoradial velocity profiles  $V_{\theta}(r)$  observed during the flows of a Newtonian oil (Fig. 1). As shown above, these profiles can be used to check the validity of the Couette analogy and to determine the Couette equivalent radius  $R_{i,eq}$ . The orthoradial dependence of the velocity profiles between two adjacent blades of the vane tool will be considered after.



Figure 1: a) Dimensionless velocity profile  $V_{\theta}(r, \Omega)/\Omega R_i$  of a Newtonian oil in a Couette geometry  $(R_i = 4.15 \text{ cm})$ , at various rotational velocities  $\Omega$  ranging from 2 to 20 rpm; the solid line is the theoretical profile for a Newtonian fluid. b) Dimensionless  $\theta$ -averaged velocity profile  $\bar{V}_{\theta}(r, \Omega)/\Omega R_i$  of a Newtonian oil in a 6-bladed vane-in-cup geometry  $(R_i = 4.02 \text{ cm})$  for  $\Omega$  ranging from 1 to 9 rpm; the dashed line shows the radius of the blades; the dotted line is the theoretical profile for a rigid body rotation (for  $r < R_i$ ); the solid lines are the theoretical profiles for a Newtonian fluid in Couette geometries of radii, from right to left: (i)  $R_i = 4.02 \text{ cm}$ , (ii)  $R_{i,eq} = 3.88 \text{ cm}$ , and (iii)  $R_{i,th} = 3.67 \text{ cm}$  corresponding to the Atkinson and Sherwood (1992) theory.

In Fig. 1a we observe that the velocity profiles in the gap of a Couette geometry are, as expected, in perfect agreement with the theory for a Newtonian flow (Eq. 6). This first observation can be seen as a validation of the display.

In the vane-in-cup geometry (Fig. 1b), we first note that the  $\theta$ -averaged dimensionless orthoradial velocity profiles  $\overline{V}_{\theta}(r, \Omega)/\Omega R_i$  measured for several rotational velocities  $\Omega$  are superposed, as expected from the linear behavior of the material. We also remark that the material between the blades rotates as a rigid body only up to  $r \simeq 3.1$  cm, indicating that the shear flow enters deeply the region between the blades (the blades radius is 4.02 cm). The whole limit between the sheared and the unsheared material in the  $(r, \theta)$  plane will be determined in Sec. IIIA3 (Fig. 3b). We finally observe that the theoretical velocity profile for a Newtonian fluid in a Couette geometry of radius equal to that of the blades falls above the data, as expected from the literature. This is also consistent with the observation that the shear flow enters the region between the vane blades.

In order to test the Couette analogy and determine the radius  $R_{i,eq}$  of the equivalent Couette geometry, we have chosen to plot the  $\theta$ -averaged strain rate  $\bar{d}_{r\theta}$  vs. the radius r in Fig. 2. This allows distinguishing more clearly the difference between the experimental and theoretical flow properties than the velocity profiles, because in any case the velocity profile tends towards the same limit ( $V(R_o) = 0$ ) at the outer cylinder whereas the strain rate profile does not. Note that velocity measurements could not be performed at proximity of the blades, which explains why strain rate data are missing from 4 to 4.2 cm.



Figure 2:  $\theta$ -averaged strain rate  $\bar{d}_{r\theta}$  vs. radius r for a Newtonian oil sheared at 1 rpm in a 6-bladed vane-in-cup geometry. The dashed line shows the radius of the blades. The solid lines are the theoretical strain rate profiles for a Newtonian fluid in Couette geometries of radii: (i)  $R_i = 4.02$  cm (light grey), (ii)  $R_{i,eq} = 3.88$  cm (black), and (iii)  $R_{i,th} = 3.67$  cm (dark grey) corresponding to the Atkinson and Sherwood (1992) theory in a infinite medium.

In Fig. 2, we first note that  $\bar{d}_{r\theta}$  is naught up to  $\simeq 3$  cm, which corresponds to the limit of the rigid motion of the material;  $\bar{d}_{r\theta}$  then increases when r tends towards  $R_i$  as the material is more and more sheared between the blades. In the gap of the geometry,  $d_{r\theta}$  decreases when r increases. As expected, the theoretical strain rate profile for a Couette geometry of radius equal to that of the blades falls well above the data at any radius r (it was less obvious on the velocity profiles). We then observe that the data are well fitted to the theoretical profile Eq. 13 that would be observed in a Couette geometry of radius  $R_{i,eq} = 3.88$  cm. This confirms that the  $\theta$ -averaged strain rate  $\bar{d}_{r\theta}$  decreases as the inverse squared radius in the gap, in agreement with the Couette analogy. From Eq. 8, it means that the torque exerted by the vane tool is decreased by a factor  $R_{i,eq}^2/R_i^2 = 0.93$  as compared to the torque exerted by the inner cylinder of a Couette geometry of radius  $R_i = 4.02$  cm at a same rotational velocity. This is significantly different from the Atkinson and Sherwood (1992) prediction, namely that the torque should be decreased approximately by a factor 1 - 1/n for an infinite *n*-bladed vane-in-cup geometry (let us recall that we do not need to consider end effects here because we measure only the contribution to the torque from the material sheared in the gap). For a 6-bladed vane tool, this would imply a theoretical decrease of the torque by a factor 0.83, corresponding to a theoretical "equivalent radius"  $R_{i,th} = 3.67$  cm. We observe in Fig. 2 that the strain rate profile corresponding to this theory falls well below the data. We also plot the velocity profiles corresponding to Eq. 12 with  $R_{i,eq}$  and  $R_{i,th}$  in Fig. 1 for illustration; they lead to the same conclusion. Note that the comparison between velocity profiles is discriminant only near the blades.

Finally, for a given rotational velocity, the torque with our vane tool is significantly higher (by 12%) than that predicted by the theoretical approach, and is thus much closer to that of a Couette geometry of same radius as the vane blades than expected. The difference between our results and the Atkinson and Sherwood (1992) theory may be due to two features that are absent from the theory: the finite blades thickness and the presence of the outer cylinder. Sherwood and Meeten (1991) actually found a very good agreement between the theory and experimental torque measurements on oils, with a geometry of inner radius 13.4 mm and thickness 1 mm, and of outer cylinder radius 102.5 mm. Sherwood and Meeten (1991) argue that, as the stress distributions varies as  $1/r^2$  in a Couette geometry,  $(R_i/R_o)^2$  should be of the order of 1% or less

to consider that the presence of the outer boundary can be neglected; this is clearly the case in their experiments and in field experiments where the vane is embedded e.g. in a soil; this is clearly not the case in our experiments and in most rheological experiments that make use of a vane-in-cup geometry. Note that, due to non-zero thickness, the distance from the center of the geometry to the blade end varies between 4.02 cm at the middle and 4.05 cm at the edges; this ambiguity on the geometry radius has negligible impact on our observations. However, this does not rule out the blades thickness as a possible origin of this difference as it may change the flow field. Whatever the real reason of this difference is, this shows that the Atkinson and Sherwood (1992) theory is probably of no use if the gap is not that large as compared to the vane radius and/or when the blades thickness is not negligible (in this later case, a criterion is not easy to formulate although it probably involves the thickness to radius ratio, which is of order 0.2 in our experiments and of order 0.08 in the Sherwood and Meeten (1991) experiments). In such case, it seems that numerical investigations are still needed, and that, at this stage, a calibration has to be performed to get the geometry factors.

#### **3** $\theta$ dependent profiles

To better characterize the flow field, we now study the dependence of the velocity profiles on the angular position  $\theta$ . We have performed experiments in which we measure one orthoradial velocity profile per second while the vane tool is rotated at 1 rpm, yielding 10 profiles between two adjacent blades.



Figure 3: a) Orthoradial velocity profile  $V_{\theta}(r, \theta)$  of a Newtonian oil sheared at 1 rpm in a 6bladed vane-in-cup geometry, for various angular positions  $\theta$ , from a blade ( $\theta = 0^{\circ}$ ) to midway between adjacent blades ( $\theta = 30^{\circ}$ ). The dashed line shows the radius of the blades. The dotted line is the profile for a rigid body rotation (for  $r < R_i$ ) and the theoretical profile for a Newtonian fluid in a Couette geometry of radius  $R_i$  (for  $r > R_i$ ). b) Two-dimensional plot of the limit between rigid motion and shear (empty circles) for a Newtonian material in the 6-bladed vane-in-cup geometry; the grey rectangles correspond to the blades.

In Fig. 3a, we plot the velocity profiles  $V_{\theta}(r, \theta)$  measured at different angles  $\theta$ . We first observe that the velocity profile which starts from a blade tip (corresponding to  $\theta = 0^{\circ}$  by definition) is very different from the velocity profile in a Couette geometry of same radius: it starts with a much steeper slope, which means that the blades tips neighborhoods are regions of important shear as already observed by Barnes and Carnali (1990). We then observe that, as expected from the  $\theta$ -averaged velocity profiles, the shear flow enters more and more deeply the region between the blades as  $\theta$  tends towards 30° (corresponding to midway between two adjacent blades); at this angular position, the rigid rotation stops at  $R_l \simeq 3.05$  cm. From all the velocity profiles, we finally extract a 2D map of the limit  $R_l(\theta)$  between rigid rotation and shear, which is depicted in Fig. 3b. This provides an idea of the deviation from cylindrical symmetry, and will be compared in the following to the case of yield stress fluids.



Figure 4: Radial velocity profile  $V_r(r, \theta)$  of a Newtonian oil sheared at 1 rpm in a 6-bladed vanein-cup geometry, for various angular positions  $\theta$  between two adjacent blades (from  $\theta = 0^{\circ}$  to  $\theta = 60^{\circ}$ ).

As explained in Sec. II, from the  $V_{\theta}(r,\theta)$  measurement and from the material incompressibility, we are able to reconstruct the radial velocity profile  $V_r(r,\theta)$  (see Fig. 4). This also allows us to compute the strain rate components  $d_{r\theta}(r,\theta)$  and  $d_{rr}(r,\theta) = -d_{\theta\theta}(r,\theta)$ , which are plotted in Fig. 5. Of course, due to the limited amount of profiles between two adjacent blades, such method provides only a rough estimate of these quantities. In addition to their interest for future comparison with modelings and simulations, these data allow us to evaluate the contribution of the extensional flow to dissipation; here, in a Newtonian medium, the local power density reads:  $p_d(r,\theta) = 2\eta(d_{r\theta}^2 + 2d_{rr}^2)$ .



Figure 5: Strain rate profiles  $d_{r\theta}(r,\theta)$  (left) and  $d_{rr}(r,\theta)$  (right) vs. radius r for various angular positions  $\theta$  between two adjacent blades (from  $\theta = 0^{\circ}$  to  $\theta = 60^{\circ}$ ).

In Fig. 4, we first observe that  $V_r(r,\theta) \simeq 0$  for  $\theta = 0^\circ$  and  $\theta = 30^\circ$ ; there is thus no extensional flow in these regions of space, as seen in Fig. 5. This is actually expected from the fore-aft symmetry of the flow around these angular positions.  $V_r$  and its spatial variations (i.e.  $d_{rr}$ ) are maximal at  $\theta \simeq 15^\circ$ . Meanwhile, we observe that  $d_{r\theta}$  is maximal near the blades: at

 $r \simeq R_i$  it is more than 4 times larger at  $\theta = 0^\circ$  than at  $\theta = 30^\circ$ . We then find that  $d_{r\theta}$  (and thus the shear stress  $\tau_{r\theta}$ ) decreases more rapidly from the blades (at  $\theta = 0^\circ$ ) than the  $1/r^2$  scaling of the Couette geometry, whereas it does not vary much with r midway between adjacent blades (it even seems to slightly increase with r as already observed in simulations by Savarmand *et al.* (2007)). It is also worth nothing that at  $r \simeq R_e$ , in contrast with what is observed at  $r \simeq R_i$ , the shear stress value is of order two times lower at  $\theta = 0^\circ$  than at  $\theta = 30^\circ$ .

From the whole  $d_{r\theta}$  and  $d_{rr}$  measurements (Fig. 5), we finally find that in regions where  $d_{rr}$  is maximal, the contribution of the extensional flow to dissipation is of order 25%. Over the whole gap, we then evaluate its average contribution to dissipation to be rather important, of order 5 to 10%. This significant value may be a reason why the torque that has to be exerted to enforce flow is higher than expected from the theory of Atkinson and Sherwood (1992). The confinement effect induced by a close boundary at a radius  $R_e$  likely increases the contribution of the extensional flow to dissipation as compared to the case of an infinite medium.

#### **B** Yield stress fluid

In this section, we study the flows induced by the vane tool on a yield stress fluid (a dense emulsion). We focus on the behavior near the yielding transition, *i.e.* on low rotational velocities  $\Omega$ .



Figure 6: Dimensionless  $\theta$ -averaged velocity profile  $\bar{V}_{\theta}(r, \Omega)/\Omega R_i$  of a yield stress fluid (dense emulsion) in a 6-bladed vane-in-cup geometry for  $\Omega$  ranging from 0.1 to 9 rpm; the dashed line shows the radius of the blades; the dotted line is the theoretical profile for a rigid body rotation (for  $r < R_i$ ).

In Fig. 6, we plot the  $\theta$ -averaged orthoradial velocity profiles  $\bar{V}_{\theta}(r)$  measured at several  $\Omega$  ranging from 0.1 to 9 rpm, corresponding to macroscopic shear rates varying between 0.02 and  $2 \,\mathrm{s}^{-1}$ . We first observe that flow is localized: the material is sheared only up to a radius  $R_c < R_o$ .  $R_c$  is found to decrease when decreasing  $\Omega$ . This is a classical feature of flows of yield stress fluids in heterogeneous stress fields. It has been observed in Couette geometries [Coussot (2005); Ovarlez *et al.* (2008)], where it is attributed to the  $1/r^2$  decrease of the shear stress  $\tau_{r\theta}$ , which passes below  $\tau_y$  at some  $R_c(\Omega) < R_o$  at low  $\Omega$ . In this case, when  $\Omega$  tends to 0,  $R_c$  tends to  $R_i$  and the torque T at the inner cylinder tends to  $\tau_y * 2\pi R_i^2 H$ . In the vane-in-cup geometry, the same argument holds qualitatively thanks to Eq. 8. It implies that the flow has to stop inside the gap at low  $\Omega$ . However, in contrast with the case of the Couette geometry, as the whole

stress field a priori depends on  $\theta$ , this  $\theta$ -averaged equation does not provide the position of the limit between the sheared and the unsheared material (it will determined in the sequel).

We then observe that, although this effect is less pronounced than with a Newtonian material, the shear flow still enters the region between the blades, even at the lowest studied  $\Omega$ . Close examination of the profiles shows that the material trapped between the blades rotates as a rigid body only up to  $R_l \simeq 3.65$  cm at  $\Omega = 9$  rpm,  $R_l \simeq 3.75$  cm at  $\Omega = 1$  rpm, and  $R_l \simeq 3.85$  cm at  $\Omega = 0.1$  rpm. We recall that  $R_l \simeq 3.05$  cm with a Newtonian fluid in the same geometry.



Figure 7: Orthoradial velocity profile  $V_{\theta}(r, \theta)$  of a yield stress fluid (dense emulsion) sheared at 0.1 rpm in a 6-bladed vane-in-cup geometry, for various angular positions  $\theta$ , from a blade  $(\theta = 0^{\circ})$  to midway between adjacent blades ( $\theta = 30^{\circ}$ ). The dashed line shows the radius of the blades.

As in Sec. IIIA3, to better characterize the flow field, we have performed experiments in which we have measured 10 orthoradial profiles between two adjacent blades at 0.1 rpm. In Fig. 7, as for a Newtonian fluid, we observe that there is a strong  $\theta$ -dependence of the velocity profiles. The velocity profile that starts from a blade tip (at  $\theta = 0^{\circ}$ ) has a much steeper slope that the profile measured midway between adjacent blades (at  $\theta = 30^{\circ}$ ); again, this shows that the blades tips neighborhood are regions of high shear. Meanwhile the flow stops at a radius  $R_c$  which is larger at  $\theta = 30^{\circ}$  (4.5 cm) than at  $\theta = 0^{\circ}$  (4.3 cm). From these velocity profiles, we have reconstructed a 2D map of the flow field (Fig. 8), with both the limit between rigid motion between the blades and shear, and the limit between shear and rest (i.e. the position where the yield criterion is fulfilled).

Flow is found to occur in a layer of complex shape which is far from being cylindrical even at this very low velocity. These observations are in contradiction with the usually accepted picture for yield stress fluid flows at low rates [Barnes and Nguyen (2001)], namely that the material contained in the virtual cylinder delimited by the blades rotates as a rigid body, and that it flows uniformly in a thin cylindrical layer near the blades. Our results contrast in particular with previous numerical works which showed that the yield surface is cylindrical at low rates for Bingham fluids, Casson fluids, and Herschel-Bulkley materials with n = 0.5 [Keentok *et al.* (1985); Yan and James (1997); Savarmand *et al.* (2007)]. With apparently similar conditions as some of the Yan and James (1997) simulations, we find an important departure from cylindrical symmetry. This means that further investigation on the exact conditions under which this symmetry can be recovered have to be performed. Possible difference between our work and that of Yan and James (1997) is that the blade thickness is naught in this last study.

It is particularly striking and counterintuitive that  $R_c$  is the larger at the angular position  $(\theta = 30^{\circ})$  where shear at  $R_i$  is the less important. As in Sec. IIIA3, this points out the

importance of the extensional flow in this geometry, with  $\theta$ -dependent normal stress differences which have to be taken into account in the yield criterion, and which thus impact the yield surface. It thus seems that the link between the yield stress  $\tau_y$  and the torque T measured at yield with a vane-in-cup geometry is still an open question, although the classical formula probably provides a sufficiently accurate determination of  $\tau_y$  in practice.



Figure 8: Two-dimensional plot of the limit between rigid motion and shear (circles) and between shear and rest (triangles) for a yield stress fluid (dense emulsion) sheared in the 6-bladed vanein-cup geometry at 0.1 rpm (left) and 1 rpm (right). The grey rectangles correspond to the blades.

The same 2D map as above is plotted for  $\Omega = 1$  rpm in Fig. 8; the same phenomena are observed, with enhanced departure from cylindrical symmetry, consistently with the observation that  $R_l$  decreases when increasing  $\Omega$ . This result was also unexpected, as simulations find uniform flows for shear-thinning material of index  $n \leq 0.5$  [Barnes and Carnali (1990); Savarmand *et al.* (2007)]; we would have expected the same phenomenology in a Herschel-Bulkley material of index n = 0.5 (and thus  $R_l$  to tend to  $R_i$  when increasing  $\Omega$ ). This observation shows that a Couette analogy can hardly be defined for studying the flow properties of such materials in a vane-in-cup geometry because the equivalent Couette geometry radius  $R_{i,eq}$  would probably depend also on  $\Omega$ .

Let us finally note that this departure from cylindrical symmetry has important impact on the migration of particles in a yield stress fluid (see below).

#### C Dense suspension

In this section, we investigate the behavior of a dense suspension of noncolloidal particles in a yield stress fluid (at a 40% volume fraction).

A detailed study of their velocity profiles would *a priori* present here limited interest: such materials present the same nonlinear macroscopic behavior as the interstitial yield stress fluid, and their rheological properties (yield stress, consistency) depend moderately on the particle volume fraction [Mahaut *et al.* (2008a); Chateau *et al.* (2008)].

On the other hand, noncolloidal particles in suspensions are known to be prone to shearinduced migration, which leads to volume fraction heterogeneities. This phenomenon is well documented in the case of suspensions in Newtonian fluids [Leighton and Acrivos (1987b); Abbott *et al.* (1991); Phillips *et al.* (1992); Corbett *et al.* (1995); Shapley *et al.* (2004); Ovarlez *et al.* (2006)] but is still badly known in yield stress fluids (some studies exist however in viscoelastic fluids [Tehrani (1996); Huang and Joseph (2000); Lormand and Phillips (2004)]). In the model of Leighton and Acrivos (1987b) and Phillips *et al.* (1992), migration is related to shear-induced diffusion of the particles [Leighton and Acrivos (1987a); Acrivos (1995)]. In a wide gap Couette geometry, the shear stress heterogeneity is important (Eq. 4); the shear rate gradients then generate a particle flux towards the low shear zones, which is counterbalanced by a particle flux due to viscosity gradients. A steady state, which results from competition between both fluxes, may then be reached, and is characterized by an excess of particles in the low shear zones of the flow geometry (near the outer cylinder in a wide-gap Couette geometry [Phillips *et al.* (1992); Corbett *et al.* (1995); Ovarlez *et al.* (2006)]). Note that there are other models [Nott and Brady (1994); Mills and Snabre (1995); Morris and Boulay (1999); Lhuillier (2009)] in which particle fluxes counterbalance the gradients in the particle normal stresses, and which can be used directly for non-Newtonian media.

As the development of migration depends on the spatial variations of shear, one may wonder how the orthoradial heterogeneities of shear introduced by the vane tool effect migration; a related question is that of the relevance of the Couette analogy for this phenomenon. In the following, we thus focus on the particle volume fraction distribution evolution when the material is sheared.

#### Behavior at high shear rate

We first study the behavior at high shear rate, in the absence of shear localization. We shear the suspension in both the standard Couette geometry and the vane-in-cup geometry at a rotational velocity  $\Omega = 100$  rpm. In this first set of experiments, we only study the steady-state of migration. At  $\Omega = 100$  rpm, this steady-state is reached in less than 30 min (which corresponds to a macroscopic strain of order 50000, consistently with strainscale evaluations from data of the literature [Ovarlez *et al.* (2006)]). In Fig. 9 we plot the steady state volume fraction profiles observed after shearing the suspension at  $\Omega = 100$  rpm during 1h.



Figure 9: a) Steady-state volume fraction vs. radius at  $\Omega = 100$  rpm in both the Couette geometry (empty circles) and the vane-in-cup geometry (squares). In the vane-in-cup geometry, the profile is performed in a 1 cm thick slice situated exactly between two adjacent blades (see Fig. 10b). The inset is a zoom; the line is a fit of the data measured in the Couette geometry to the Phillips *et al.* (1992) model with  $K_c/K_{\mu} = 0.42$ . b)  $\theta$ -averaged orthoradial velocity profile  $\bar{V}_{\theta}(r)$ ; the dotted line is the theoretical rigid motion induced by the rotation of the vane tool; the dashed line shows the radius of the blades.

As expected, we first observe that the material is strongly heterogeneous in the Couette geometry: the volume fraction varies between 37% near the inner cylinder and 43% near the outer cylinder (note that the NMR technique we use do not allow for quantitative measurements

near the walls). This heterogeneity is quantitatively similar to that observed in Couette flows of Newtonian suspensions at a same 40% particle volume fraction [Corbett *et al.* (1995)]; the profiles are actually well fitted to the Phillips *et al.* (1992) model (see Eq. 16 of Ovarlez *et al.* (2006)) with a dimensionless diffusion constant  $K_c/K_{\mu} = 0.42$  which is close to that found by Corbett *et al.* (1995) ( $K_c/K_{\mu} = 0.36$ ), although this model is not expected to hold in non-Newtonian suspensions.

In the vane-in-cup geometry, the volume fraction profile shows very different features; note that the profile is performed in a 1 cm thick slice situated exactly between two adjacent blades (see Fig. 10). In Fig. 9, we first observe that there is a strong particle depletion in a wide zone between the blades. A homogeneous volume fraction of 40% is observed for radii inferior to 3.1 cm. At a radius  $R_l = 3.1$  cm, there is a strong drop in the volume fraction down to 5% within 1 mm (corresponding to 4 particle diameters). Close inspection of the velocity profile Fig. 9 shows that this radius  $R_l$  corresponds to the transition between rigid motion and shear between the blades. The volume fraction then increases basically linearly with the radius up to a 40.5% volume fraction at a radius r = 3.85 cm which is close to the blades radius. The volume fraction then increases only slightly (between 40.5% and 42.5%) in the gap of the geometry: the heterogeneity is here much less important than in a standard Couette geometry.

To get further insight in the new strong depletion phenomenon we have evidenced, we have performed 2D magnetic resonance images of the material. Such images provide a qualitative view of the spatial variations of the particle volume fraction as only the liquid phase is imaged. Images are coded in grey scales; a brighter zone contains less particles. In Fig. 10b, we first see an image of the homogeneous material. Before any shear, as expected, the light intensity is homogeneous in the sample (intensity variations correspond to noise). After a 1h shear at  $\Omega = 100$  rpm, we observe very bright and thin curves on the image: they correspond to zones where the volume fraction suddenly drops down to a value close to zero. These curves are not cylindrical. More precisely, between two adjacent blades, a depleted zone goes from the edge of one blade (at  $\theta = 0^{\circ}$ , r = 4.02 cm) to the edge of another blade (at  $\theta = 30^{\circ}$ . Note that as the volume fraction profile is averaged over a slice which is 1 cm thick in the orthoradial direction (see Fig. 10b), the fact that we measure a minimum of 5% at r = 3.1 cm in the slice probably means that the volume fraction minimum is actually equal to zero in the depletion zone.

As pointed out above, this curve also likely marks the transition between the unsheared material (which rotates as a rigid body) and the sheared material. Note in particular the similarity with Fig. 3b, the data of which are reported in Fig. 10a for illustration. A first interpretation of the phenomenon would then simply be that migration is caused by shear and naturally stops at this transition zone. Indeed, as shear is maximum near the blades, particles tends to migrate out of this zone; moreover, there is no source of particle flux from the unsheared zone between the blades to balance the migration towards the outer cylinder. However, this does not explain why the volume fraction drops down to zero: heterogeneities observed at steadystate in the literature are usually moderate and do not lead to zones free of particles. A better understanding of the phenomenon can be gained by zooming on the previous image (Fig. 10c). We now see that while the depletion phenomenon seems symmetric around both sides of the blades at a macroscopic scale, it is clearly asymmetric at a local scale near the blades and depends on the direction of rotation: depletion is more pronounced at the back of the blade (note that the vane tool rotates counterclockwise). This would mean that the noncolloidal particles trajectories are asymmetric around the blade: a particle that is found at a radius  $r \simeq 4.02$  cm just before the blade is necessarily found at a radius slightly higher than 4.02 cm after the blade as there are no particles at r = 4.02 cm. This feature is reminiscent of the fore-aft asymmetry that is observed in the bulk of noncolloidal suspensions [Parsi and Gadala-Maria (1987)] and that leads to their non-Newtonian properties [Brady and Morris (1997)]. It thus seems that, in addition to the shear-induced migration mechanism intrinsic to suspensions, the vane tool induces a specific



Figure 10: 2D magnetic resonance image of a suspension of particles in a yield stress fluid in a vane-in-cup geometry: (a) after a 1h shear at  $\Omega = 100$  rpm (corresponding to a macroscopic strain of order 75000), and (b) before any shear. The crosses in Fig. 10a correspond to the limit between rigid motion and shear for the Newtonian oil of Fig. 3b. The white rectangle in Fig. 10b shows the slice in which the volume fraction profiles of Figs. 9a and 11 are performed. (c) is a zoom of image (a) near the edges of a blade. The images are taken in the horizontal plane of the geometry, at middle height of the vane tool, and correspond to vertical averages over 2 cm. The vane tool rotates counterclockwise.

migration mechanism which has its origin in the direct interactions between the particles and the blades; this effects leads to the full depletion that is observed at the transition between the sheared and the unsheared material. Such direct effect of a flow geometry on migration have also been observed in microchannel flows of colloidal suspensions [Wyss *et al.* (2006)], and also led to full particle depletion. The kinetics of the phenomenon will be briefly discussed below.

The rest of the volume fraction profile results from a complex interplay between shear-induced migration and the fore aft asymmetry around the blades; this leads to the rapid increase of the volume fraction between 3.1 cm and 4.02 cm. After 4.02 cm the flow lines do not meet the blade edges, and the phenomenon evidenced above should have basically no effect on the heterogeneity that develops in the gap of the geometry. On the other hand, the mean volume fraction should be slightly higher due to mass conservation; it is indeed observed to be equal to around 42%. Nevertheless, as the mean radial shear rate heterogeneity is basically similar to that observed in a standard Couette geometry (see previous sections), we would *apriori* expect the heterogeneity to be somehow similar. However, we observe that the volume fraction profile is only slightly heterogeneous: there is less than 5% variation of the volume fraction in the gap, to be compared to the 15% variation observed in the Couette geometry. Clearly, this means that the Couette analogy is irrelevant as regards this phenomenon, and that the details of shear matter. Here, the extensional flow that adds to shear may be at the origin of this diminution of migration. A more detailed analysis is out of the scope of this paper.

#### Behavior at low shear rate

Let us now study the behavior at low shear rate. Low shear rates are typically imposed in the end of measuring the yield stress of such materials. Starting from a homogeneous suspension at rest, we apply a rotational velocity  $\Omega = 1$  rpm (without any preshear), and we measure the evolution of the particle volume fraction in time. The corresponding volume fraction profiles are depicted in Fig. 11.



Figure 11: Volume fraction vs. radius at  $\Omega = 1$  rpm measured in the vane-in-cup geometry after different times of shear: 5min, 1h, 14h. The material is homogeneous at the beginning of shear. The inset presents the  $\theta$ -averaged orthoradial velocity profile  $\bar{V}_{\theta}(r)$  measured in the first stages of shear; the dotted line is the theoretical rigid motion induced by the rotation of the vane tool; the dashed line shows the radius of the blades.

In Fig. 11, we observe that, although shear is much less important than in the previous experiments, particle depletion also appears between the blades. Comparison of the velocity profile and the volume fraction profile shows that depletion also appears between the blades at the transition zone between the sheared and the unsheared materials. This phenomenon appears

with a very fast kinetics: the lower volume fraction value in the measurement zone is 36% after only a 5 minutes shear (corresponding to a macroscopic strain of order 50). Afterwards, it continues evolving slowly: the minimum observed volume fraction is of order 33% after a 1h shear and of order 32% after a 14h shear (corresponding to a 10000 macroscopic strain). Note that the radial position of the minimum value of the volume fraction slightly decreases in time; it likely corresponds to progressive erosion of the material between the blades (we did not measure the velocity profiles to check this hypothesis).

We also note that migration is negligible in the rest of the sheared material as expected from the theory of migration briefly described above (a larger strain would be needed to observe significant migration). Nevertheless, we note some particle accumulation (with a volume fraction value of 43%) at  $R_c$ =4.7 cm after a very long time. This corresponds to the yield surface as flow is localized at low velocity (see velocity profile Fig. 11). Migration profiles usually result from an equilibrium between various sources of fluxes. On the other hand, the unsheared material does not produce any particle flux while it receives particles from the sheared region. This particle accumulation is thus the signature that the migration phenomenon is indeed active, although not observable on the profile measured in the sheared zone. It is probable that this accumulation process would stop only (after a very long time) when there are no more particles in the sheared region.



Figure 12: 2D magnetic resonance image of a suspension of particles in a yield stress fluid in the horizontal plane of a vane-in-cup geometry after a 14h shear at  $\Omega = 1$  rpm (a). (b) is a zoom of image (a) between two adjacent blades. The image is taken in the horizontal plane of the geometry, at middle height of the vane tool, and corresponds to a vertical average over 2 cm. The vane tool rotates counterclockwise.

As above, 2D magnetic resonance images of the material provide an insight in the phenomenon. In Fig. 12, we observe again that particle depletion is enhanced at the rear of the blades; this confirms that this phenomenon is likely due to direct interactions between the blades and the particles, leading to the asymmetry of the particles trajectory around the blades. This *a priori* occurs with any particle whose trajectory is close to the blades, explaining why particle depletion appears so rapidly. There is probably no way to avoid it. Note that the images are here much brighter very close to the blades than midway between two adjacent blades; this would mean that the particle volume fraction is probably close to 0 near the blades, although we observe volume fraction of order 32% between two blades. Finally, let us note that the bright line provides a good idea of what the limit between the sheared material and the material that moves as a rigid body. We see as in Sec. IIIB that this is far from being cylindrical even at this low velocity.

#### Consequences

We finally present some consequences of this phenomenon. From the above observations, our conclusion is that depletion sets up quickly and is probably unavoidable. Then two situations have to be distinguished. If viscoelastic properties of a material are measured at rest on the homogeneous material in its solid regime, without any preshear, then these measurements pose no other problem than that of the relevant Couette analogy to be used (see Sec. IIIA). If a yield stress measurement is performed at low imposed rotational velocity, starting from the homogeneous material at rest, then this measurement is likely valid as long as only the peak value or the plateau value at low strain (of order 1) is recorded. On the other hand, any subsequent analysis of the material behavior will a priori be misleading: irreversible changes have occurred and the material cannot be studied anymore. More generally, any measurement performed after a preshear will be uncorrect and any flow curve measurement will lead to wrong evaluation of the materials properties. In these last cases, the consequence of the new particle depletion phenomenon we have evidenced is a kind of wall slip near the blades, whereas there are no walls. Here the "slip layer" is made of the (pure) interstitial yield stress fluid in a zone close to the blades, as would be observed near a smooth inner cylinder. This contrasts with the common belief that the vane tool prevents from slippage.

In order to illustrate this feature, we present some results of Mahaut *et al.* (2008a): Mahaut *et al.* (2008a) have performed classical upward/downward shear rate sweeps with a vane-in-cup geometry in a pure dense emulsion, and in the same emulsion filled with 20% of 140  $\mu$ m PS beads. In these experiments, constant macroscopic shear rates increasing from 0.01 to 10 s<sup>-1</sup> and then decreasing from 10 to 0.01 s<sup>-1</sup> were applied during 30s, and the stationary shear stress was measured for each shear rate value. The results are shown in Fig. 13.



Figure 13: Shear stress vs. shear rate for upward/downward shear rate sweeps in a pure dense emulsion (open squares) and for the same emulsion filled with 20% of 140  $\mu$ m PS beads (filled/open circles). Figure from Mahaut *et al.* (2008a).

While the same curve is observed for the upward/downward shear rate sweeps in the case of the pure emulsion (as expected for a simple non-thixotropic yield stress fluid), the shear stress during the upward shear rate sweep differs from the shear stress during the downward shear rate sweep in the case of the suspension. Moreover, any measurement performed on the suspension after this experiment gives a static yield stress equal to the dynamic yield stress observed during the downward sweep. This means that there has been some irreversible change. This irreversible change is actually the particle depletion near the blades we have observed in this paper. As the flow of the suspension is localized near the inner tool at low shear rate, it means that after the first upward sweep that has induced the particle depletion, during the downward shear rate sweep, only the pure emulsion created by migration near the blades remains in the sheared layer at sufficiently low rotational velocity. This explains why the same apparent value of the yield stress is found in the suspension during the downward sweep as in the pure emulsion with this experiment. On the other hand, the yield stress at the beginning of the very first upward sweep is that of the suspension as migration has not occurred yet.

The conclusion is that the vane tool is probably not suitable to the study of flows of suspensions.

## IV Conclusion

As a conclusion, let us summarize our main findings:

- In the case of Newtonian fluid flows, our experimental measurements of the velocity field support the Couette equivalence approach: the  $\theta$ -averaged strain rate component  $d_{r\theta}$  decreases as the inverse squared radius in the gap. However, the value of the Couette equivalent radius we find with our display is higher than the theoretical prediction of Atkinson and Sherwood (1992) (it is here 6% higher). Equivalently, for a given rotational velocity, the torque with our vane tool is significantly higher than that predicted by the theoretical approach (there is a 12% difference). It is thus much closer to that of a Couette geometry of same radius as the vane blades than expected. A key observation may be that there is a significant flow between the blades which adds an important extensional component to shear, thus increasing dissipation. The main shortcomings of the theoretical approach for its use with common rheometers are that the blades are supposed to be infinitely thin and that the outer cylinder radius is infinitely large, in contrast with our (and most) display. These two features may be at the origin of this discrepancy; however, the conditions (as regards the blade thickness and the gap size) in which the theoretical predictions can be used still have to be found.
- In the case of yield stress fluid flows, we find that the thin layer of material which flows around the vane tool at low velocity is not cylindrical, in contrast with what is usually supposed in the literature from simulation results. Consequently, a non negligible extensional component of shear has probably to be taken into account in the analysis. At this stage, there are too few experimental and simulation data to understand the origin of this discrepancy. It thus seems that progress still has to be performed, in particular through simulations, which allow studying a wide range of parameters. This may help understanding how the torque is linked to the yield stress of a material at low velocity, depending in particular on the geometry parameters.
- An important and surprising result is the observation of particle depletion near the blades when the yield stress fluid contains noncolloidal particles. This phenomenon is thus likely to occur when studying polydisperse pastes like coal slurries, mortars and fresh concrete. It has to be noted that the phenomenon is very rapid, irreversible, and thus probably unavoidable when studying flows of suspensions. It results in the creation of a pure interstitial yield stress fluid layer and thus in a kind of wall slip near the blades. It contrasts with the classical assumption that is made in the field of dense suspensions rheology where the vane tool is mainly used to avoid this phenomenon.

Consequently, we would say that, in the case of pasty materials, if accurate measurements are needed, the vane tool may finally be suitable only for the study of the solid (elastic) properties of materials and for the static yield stress measurements; as the yield stress measurement may induce irreversible particle depletion near the blades, any new measurement then requires a new sample preparation. Furthermore, the vane can be used as a very accurate tool without any hypothesis nor any calibration to measure the relative increase of the elastic modulus of materials as a function of their composition [Alderman *et al.* (1991); Mahaut *et al.* (2008a)]. In order to study accurately the flows of pasty materials, our results suggest that a coaxial cylinders geometry with properly roughened surfaces is preferable when possible. If the use of a vane tool cannot be avoided, one should keep in mind our observations in order to carefully interpret any result.

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