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We explore the use of magnetic resonance imaging (MRI) velocimetry and pulsed field gradient nuclear magnetic resonance (PFG NMR) data for studying the flow characteristics of yield stress fluids through model pores (a succession of ducts of different diameters) or real porous media (bead packings). We propose different methods for the quantitative analysis of the velocity field, aimed at getting a deep understanding of the different flow regimes (solid and liquid) which typically take place in such fluids and at how the transition from one to the other occurs in space or in time. Our approach exemplifies interdependencies between PFG NMR data and local flow features and how the statistical velocity distribution function obtained by this way can be used and/or processed for extracting quantitative information concerning critical flow characteristics at a local scale. This provides a solid framework of analysis of flows through porous media with pores much smaller than the resolution of MR velocimetry.
Quantitative exploitation of PFG NMR and MRI velocimetry data for the rheological study of yield stress fluid flows at macro- and microscales in complex geometries

T. Chevalier · S. Rodts · C. Chevalier · P. Coussot

Abstract We explore the use of magnetic resonance imaging (MRI) velocimetry and pulsed field gradient nuclear magnetic resonance (PFG NMR) data for studying the flow characteristics of yield stress fluids through model pores (a succession of ducts of different diameters) or real porous media (bead packings). We propose different methods for the quantitative analysis of the velocity field, aimed at getting a deep understanding of the different flow regimes (solid and liquid) which typically take place in such fluids and at how the transition from one to the other occurs in space or in time. Our approach exemplifies the different interdependences between PFG NMR data and local flow features and how the statistical velocity distribution function obtained by this way can be used and/or processed for extracting quantitative information concerning critical flow characteristics at a local scale. This provides a solid framework of analysis of flows through porous media with pores much smaller than the resolution of MR velocimetry.

1 Introduction

Yield stress fluids (such as pastes, concentrated emulsions, foams, clayey suspensions or even some polymeric gels) are in some way the archetype of complex fluids as they exhibit at the same time a solid behavior (below a critical stress, i.e., the yield stress) and a liquid behavior (beyond the yield stress). These fluids are often used to coat solid surfaces (creams or gels on the skin, mortars on walls, sauces on food, etc.) since it is possible to get a deposited layer of large thickness as long as the gravity stress is smaller than the yield stress. However, various applications also concern flows through porous media: penetration of glue in the surface porosity of solid materials, injection of muds, slurries or cement grouts to reinforce soils and, likely the most economically important application, injection of fluids in rocks either for the reinforcement of the wells or for enhancing oil recovery.

In recent years, the detailed characteristics of yield stress fluid flows in simple geometries (Couette, capillary, inclined plane) have been studied with various imaging techniques (PIV, ultrasounds, MRI) (Coussot et al. 2009; Ovarlez et al. 2008, 2010; Divoux et al. 2010, 2011; Rabideau et al. 2010; Poumaere et al. 2014; Chambon et al. 2014), providing the velocity field inside transparent or non-transparent samples. In these cases, it was shown that an unyielded (solid) region coexists with a flowing (liquid) region, with a relative size depending on flow conditions in steady state or flow history in transient conditions. A small number of studies focused on more complex geometries such as the flow around a solid object (Tokpavi et al. 2009; Putz and Frigaard 2010; Boujlel et al. 2012) or extrusion (Götz et al. 1993; Rabideau et al. 2010; Rodts et al. 2010).

In general, unyielded regions were again observed, but in some cases, its extent was found to remain approximately constant, whereas the flow intensity increases (Rabideau et al. 2010). In contrast with the flow through a capillary, the flow of a yield stress fluid through a pore does not necessarily
involve a constant size of the possible volume of fluid transported: As the pressure drop increases a wider region of fluid starts to flow (de Souza Mendes et al. 2007). Such a result was extrapolated to a real porous medium (with a large number of connected pores), leading to conclude that two critical effects could occur: (1) At the pore scale, the flowing volume increases with the pressure gradient (Balhoff and Thompson 2004) and (2) at a macroscopic scale, the flow starts as a percolation effect, i.e., at a critical pressure drop, liquid regions exist only along a specific path throughout the porous medium (Balhoff and Thompson 2004; Sahimi 1993; Roux and Herrmann 1987) and as the pressure drop is increased more flowing paths progressively form within the porous medium. Recent numerical results obtained by lattice-Boltzmann simulations (Talon and Bauer 2013) confirm this last effect in a stochastically reconstructed porous media.

Such an analysis is consistent with the observations concerning the flow of a yield stress fluid through a box (de Souza Mendes et al. 2007). As the flow intensity increases, the liquid regime involves a larger volume in the box. However, it was also shown that a yield stress fluid flowing through a wavy channel tends to develop an unyielded zone along the central axis (Roustaei and Frigaard 2013), despite usual assumptions in that field that a plug can exist only in uniform flows (Lipscomb and Denn 1984). And we can suspect that the flow through a wide set of connected pores of various shapes (i.e., a real porous medium) is more complex. So there is a need for further experimental observations and if possible direct measurements inside model porous systems although it a priori seems more difficult to get data at a scale much smaller than the pore scale, which is itself generally much smaller than the sample scale.

Finally, there is a need to develop not only new techniques, but also new interpretation frames for exploring the flow characteristics of yield stress fluid flows (and more generally of complex fluids) through porous media.

Due to intrinsic heterogeneities of either the fluid or the porous matrix, many systems of interest are non-transparent. Thanks to its ability to assess dynamic information in a non-perturbative way, NMR and/or MRI constitutes a method of choice in this field. Space-resolved imaging of velocity field (MRI velocimetry) produces so-called velocity maps, where one or more components of the velocity fields are measured for each pixel of a 2D slice—virtually cut through the sample by means of space selective methodologies or voxels of a 3D image. They can give full details of the flow field in various setups, provided they are NMR compliant, i.e., they contain no magnetic parts, and do not contain strongly electrically conductive components. The space resolution of the pictures is mainly limited by the signal-to-noise ratio, and typically amounts to one-hundredth of the sample size. Rabideau et al. (2010) and Rodts et al. (2010) showed that velocities as small as few tens of micrometers per seconds can be measured and used this technique to produce detailed maps of the velocity field close to the die of a ram extruder. The work by Sankey et al. (2009) clearly exemplifies the possibility to visualize some details of the velocity inside individual pores of a packing of 5-mm beads packed in a 27 mm diameter and 100 cm high column—in a flow range of few cm/s, however—despite it can hardly be extended to a representative sample containing a wider amount of pores. In more representative samples, the pore size is finally too small to be resolved, but the work of Spindler et al. (2011) shows that pictures of the locally homogenized velocities can still be obtained.

In order to get microscopic flow characteristics, when the pore-to-sample size ratio is very small, the pulsed field gradient (PFG) NMR technique turns out a more adequate alternative (Stejskal and Tanner 1965). It does not perform any imaging, but measures instead statistical information about local molecular movements taking the sample as a whole. In its most common use, it measures so-called flow propagators, that is, the probability density function that a fluid molecule travels a given distance over some given observation time, owing to concomitant convective and self-diffusive motions. The study of flow propagators over various time scales has provided so far an efficient way to study hydrodynamic dispersion in porous systems (Lebon et al. 1996; Tessier et al. 1997). Propagators often probe displacements over several pore sizes and can be sensitive to local rheological properties, as demonstrated by Mertens et al. (2006) in a non-Newtonian case. It has been used to study flows through natural materials such as rocks (Verganelakis et al. 2005; Mitchell et al. 2008) or for the assessment of flow profiles in capillaries (Rassi et al. 2012). A more sophisticated use has been performed to follow simultaneously and separately both continuous and suspended phases in dilute oil-in-water emulsion through glass sphere packing (Creber et al. 2009). On purpose of a more quantitative rheological analysis, we recently found a renewed interest in PFG measurements of the probabilities of instant velocities only, and identified the required experimental conditions to do so in various kinds of fluids (Chevalier et al. 2014b).

Although MR velocimetry and PFG NMR are now well-established techniques, their interpretation frame on purpose of quantitative rheological analysis is still a barely explored area. Yet, the design of quantitative approaches for data analysis is of prime importance to take the step from naive observations of elaborated measurements to a true rheological study of non-Newtonian flows. In the present study, we explore the use of NMR techniques for studying the flow characteristics of yield stress fluids through...
2 Materials and procedures

2.1 Fluid

We used water-in-oil emulsions obtained by dispersing in an oil phase droplets of brine stabilized by a surfactant. Such system was indeed identified as the most suitable for PFG velocity measurements at very low flow rates (Chevalier et al. 2014b). At high volume fraction of the dispersed phase, droplets come into contact and the fluid exhibits a yield stress (Mason et al. 1996). Batches of water-in-oil (81% of water) emulsion were prepared by dispersing a water + CaCl$_2$ (150 g/l) salt solution in a surfactant solution [dodecane (Acros Organics) + 7.5 wt% of Span 80 surfactant] with a Silverson L4RT mixer. The rotation speed was kept at 700 rpm during the addition of brine. It was then increased up to 6,000 rpm for 85 min until a homogeneous emulsion was obtained. A total volume of 10 l of emulsion was finally obtained. MRI experiments hereafter described were directly performed on hydrogen nuclei naturally contained in the sample, so that the fluid was always studied as such, without any further modification. All our measurements rely on the assumption that there is no phase separation in the sample so that NMR or MRI data reflect the motion of a representative elementary volume of sample including a sufficient number of droplets and a sufficient volume of interstitial liquid moving as a whole.

Rheological tests were performed with a Bohlin stress-controlled rheometer equipped with two circular, rough plates (diameter 40 mm). The sample was carefully set up and the gap was set to 2 mm taking care not to entrain any air bubbles. We did not carry the full correction of shear rate, but assumed that the average shear stress can be associated with the average shear rate over the sample volume. A logarithmically increasing then decreasing stress ramp test was then applied over a total time of 4 min. Except for the first part of the increasing curve associated with deformations in the solid regime, the increasing and decreasing shear stress versus shear rate curves almost superimpose. Here, the decreasing part was retained as the flow curve of the material. For similar emulsions, it has been shown that this apparent flow curve obtained from macroscopic observations corresponds to the effective, local constitutive equation observed at a local scale with imaging technique (Ovarlez et al. 2008).

The emulsion exhibits a simple yield stress fluid behavior, and its flow curve can be well fitted by a HB (Herschel–Bulkley) model (Fig. 1):

\[ \tau < \tau_c \Rightarrow \dot{\gamma} = 0 \quad \text{(solid regime)}; \]
\[ \tau > \tau_c \Rightarrow \tau = \tau_c + k\dot{\gamma}^n \quad \text{(liquid regime)} \]

![Fig. 1 Measured flow curve of one emulsion (squares) (maximum error on the shear stress 10%). The continuous line corresponds to the HB model fitted to the data ($\tau_c = 74$ Pa, $k = 13.5$ Pa s$^n$ and $n = 0.4$)](image)

![Fig. 2 Flow geometries: (left) big pore, (right) small pores](image)
Table 1  Average fluid velocities in the entrance ducts of model pores (or inside the pore space of beads packing) and corresponding Bingham numbers for our different tests

<table>
<thead>
<tr>
<th>Big pore (7 cm box)</th>
<th>Small pores (1 cm box)</th>
<th>2 mm beads</th>
<th>0.5 mm beads</th>
</tr>
</thead>
<tbody>
<tr>
<td>V (mm/s)</td>
<td>15</td>
<td>0.12</td>
<td>0.04</td>
</tr>
<tr>
<td>Bi</td>
<td>4.5</td>
<td>4.5</td>
<td>4.5</td>
</tr>
<tr>
<td>V (mm/s)</td>
<td>6.5</td>
<td>7.3</td>
<td>7.3</td>
</tr>
<tr>
<td>Bi</td>
<td>1.8</td>
<td>2.9</td>
<td>4.5</td>
</tr>
</tbody>
</table>

a Experiments where probability density function is also measured for the transverse velocity component

in which τ is the shear stress amplitude, \( \dot{\gamma} \) the shear rate amplitude, \( \tau_c \) the yield stress, \( k \) the consistency factor and \( n \) the power law exponent. Material parameters were found to be \( \tau_c = 74 \text{ Pa}, k = 13.5 \text{ Pa s}^n \) and \( n = 0.4 \) for the emulsion used in model pores and \( \tau_c = 63 \text{ Pa}, k = 13 \text{ Pa s}^n \) and \( n = 0.4 \) for the emulsion used in model porous media. We checked that the rheological behavior of the fluid was not affected by the flow through the geometry by comparing tests made on the fluid before the filling and at the exit.

2.2 Experimental setup

The experimental setup was based on that previously described in Rabideau et al. (2010). A flow geometry was plugged at the outlet of an extrusion syringe (8 cm diameter, 1 m long) run by a home-made MRI compliant mechanical press, and inserted vertically in the channel of a vertical proton MRI system (Avance 24/80 DBX by Bruker, 0.5T superconducting magnet by Oxford, 20 cm inner diameter). Two self-similar model porous geometries were used in this work (Fig. 2): a large pore, consisting in a cylindrical box (diameter and length \( D = 7 \text{ cm} \) ) accessed from both top and bottom by a straight conduit of diameter \( D/2 = 35 \text{ mm} \) and a series of four smaller cylindrical boxes (diameter and length \( d = 1 \text{ cm} \) ) connected via a conduit of diameter \( d/2 = 0.5 \text{ mm} \). The four boxes were used in order to increase the signal-to-noise ratio in PFG measurements, but will be supposed to behave in the same way regarding flow characteristics. We also used porous systems made of beads packing. Two types of beads were used with a similar grain size distribution, respectively, in the range 0.8–1.2 times the average diameter \( D_0 = 0.5 \text{ or 2 mm} \). They were covered with a thin layer (\( \approx 0.015 D_0 \) ) of resin which sticks them together, leading to a porosity \( \varepsilon \approx 0.33 \). The bead packing was 7 cm in diameter and 10 cm in height, which is large enough to be considered as representative regarding pore size. In all cases, the length of the inlet conduit was sufficient to get a uniform flow over a significant distance before the entrance in the box (Rabideau et al. 2010). In order to control the flow rate, piston velocity could be set at will in the range 0.3 \( \mu \text{m/s} \)–1.8 \( \mu\text{m/s} \). For a chosen velocity, velocity fluctuations were about \( \pm 1 \% \) of its nominal value.

The initial bubble-free filling of syringe and pore space was performed following procedures previously detailed in Rabideau et al. (2010). Before using a syringe for experiments, its correct saturation was additionally controlled by MRI. No detectable air bubble could be observed. It was found on the contrary that the small pores system often contained more fluid than expected. The pore geometry was indeed made of different pieces which were filled separately, and during assembly, some of this fluid was expelled in interstitial spaces separating constitutive elements of the geometry. This unwanted amount of fluid did not participate to the flow itself, but was nevertheless detected as stagnant fluid in PFG experiments. This problem, hereafter referred to as ‘over filling,’ was not solved during the present work and must be kept in mind in following interpretations.

2.3 Flow conditions

Due to the diameter restriction at the entrance of the geometries, the average velocities (V) in entrance conduits of model pores are accelerated as compared with piston velocity in the syringe. The former could be varied from 0.16 to 9.4 mm/s in the big pore and from 0.2 to 230 mm/s in the small pores (Table 1). Similar acceleration occurs at the entrance of the bead packing due to reduced porosity. The interstitial velocity ranged from 0.12 to 4 mm/s, whatever the bead size. Smaller flow rates could not be investigated in bead packing as we showed that instant velocity PFG distributions are unavoidably impeded by self-diffusive motions in such case (Chevalier et al. 2014b).

The generalized Reynolds number (i.e., \( Re = \rho V^2 / \tau_c \), in which \( \rho \) is the fluid density) was smaller than 1, which means that inertia effects were negligible. Under these
conditions, the flow is mainly characterized by the Bingham number, \( Bi = \tau_c / k \dot{\gamma}^n \), which estimates the ratio of the constant (elastoplastic) to the rate-dependent (viscous) parts of the constitutive equation: \( Bi = \tau_c / k \dot{\gamma}^n \), and finally a priori gives an idea of the relative importance of the solid and liquid regions in the sample. For the flow through a complex geometry, we use a characteristic shear rate \( \dot{\gamma} = V/l \), where \( l \) is the radius of the entrance conduit (\( D/4 \) for big pore and \( d/4 \) for small pores) or a typical pore size radius in the bead packing, here taken to \( l = D/6 \). Corresponding values of the Bingham number for our different tests are shown in Table 1. It is worth noting that explored ranges in small and big pores partly overlap, making it possible to check data consistency between similar flows at different scales and for a large range of Bingham numbers. Finally, in both cases (model pore geometries and bead packing), covered \( Bi \) values ranged from values well above unity, for which plastic behavior is dominant, to values close to 1, where both liquid and plastic behaviors play a major role.

Once the expected velocity had been reached, it was maintained until some material flowed out of the geometry. NMR measurements where then started and could last from few minutes up to several hours. Steady flow conditions were assumed to be reached for each measurement, this latter hypothesis being directly confirmed a posteriori from MRI of PFG NMR results (see below). The waiting time before reaching steady flow condition was also sufficient for possible residual starting creep flows effect in the solid regions to be negligible.

### 2.4 NMR methodology

Proton nuclear magnetic resonance is a non-destructive technique which uses magnetic fields to perform physical measurements on nuclear spins of hydrogen nuclei. In the present case, these are those naturally contained in water and/or oil molecules. The fluid under investigation can then be measured as such, without requiring any additional tracer particle. Measurements basically detect the amplitude and phase of spin magnetization precession around the working magnetic field. Magnetic resonance imaging technique, and more generally the so-called gradient NMR makes use of spatially linearly varying pulsed field components—the field gradients—to make these intensity and phase sensitive to spatial localization and/or motions of the spin system. They propose various imaging and non-imaging protocols to observe a flowing fluid (Callaghan 1999). Sample saturation could then be controlled in each geometry.

#### 2.4.2 Velocity imaging

2D MRI maps of the velocity vectors were measured in the big pore, in a 10-mm-thick vertical slice including the central axis of the geometry (Fig. 3). Creep flows in cylindrical geometries are expected to obey cylindrical symmetry and so only in-plane components were observed. The slowest flow rates to be considered in Table 1 are very small for the standard use of MRI. Signal-to-noise ratio (SNR) becomes critical, and fast flow imaging techniques such as those recently developed by Tayler et al. (2010) and Shiko et al. (2012) were then regarded as unsuitable in the present case. The present work relied instead on an optimization of more classical MRI approaches (Stapf and Han 2006). The MRI sequence (Fig. 3) was a 2D spin-echo Fourier imaging with a slice selection. In order to avoid flow-induced image distortions, the imaging process was made robust against fluid motions by means of velocity compensating gradient pulses. The read-out imaging gradient was applied parallel to syringe axis so as to spread imaging artefacts owing to intermittent flow at the very exit of the geometries away from
interesting parts of the picture. Velocity was encoded in magnetization phase by an additional pair of pulsed field gradients. Signal intensity in a pixel was then a complex number the argument of which was proportional—within some fixed unknown additive constant—to the velocity component in a chosen direction. Three images were finally acquired quasi-simultaneously: a reference and two pictures encoded along each of the two in-plane directions (Rabideau et al. 2010).

Local velocity vectors were deduced from a pixel by pixel comparison of the phases of the three images.

In order to cope with insufficient SNR, parameters of the NMR experiment (field of view, number of pixels, timing and strength of imaging and velocity encoding gradients, …) were first optimized numerically, taking into account the disturbing effect of local fluid diffusivities and NMR relaxation times. The velocity-overcoding concept (Rodts et al. 2004) was used extensively. It consists in pushing the sensitivity of velocity encoding far above the limit dictated by phase folding problems, phase unfolding and velocity reconstruction being performed after measurements by means of a dedicated home-made software. Such approach was found in the past to produce a 10- to 100-fold decrease of noise level on 2D velocity maps (Moller et al. 2008; Rabideau et al. 2010; Rodts et al. 2010). Additionally, prior to data processing, the extra amount of non-Gaussian noise emitted by the motor of the extrusion setup was cleaned out by means of a dedicated numerical filter (Bytchenkoff et al. 2010).

Depending on flow characteristics to be observed, space resolution was either 4 mm (longitudinal) × 1 mm (radial) or 2 mm (longitudinal) × 2 mm (radial). Steady flow conditions during the typical 10-min recording time were verified a posteriori through the absence of ripple-like artefacts on resulting pictures (Rabideau et al. 2010). The tuning of the MRI system was adjusted for each flow rate, and obtained absolute uncertainty on velocity components was ±3% of the maximal velocity in the duct. In our concentrated emulsion, water and oil were finally supposed to move together and to share the same local flow velocity. 2D velocity maps were then measured on the two phases as a whole.

Due to limited space resolution, 2D velocity mapping was only performed in the large pore geometry.

2.4.3 Direct measurements of velocity probabilities

PFG NMR (Stejskal and Tanner 1965) uses two self-compensating magnetic field gradient pulses to encode the initial and final position of a moving molecule over a given time delay \( \Delta + \delta \) (Fig. 4). The resulting NMR sequence induces a phase shift \( \varphi \) in molecular spin precession following

\[
\varphi = \gamma \tilde{G} \left( \int_{\Delta}^{\Delta+\delta} \tilde{r}(t)dt - \int_{0}^{\delta} \tilde{r}(t)dt \right)
\]

where \( \delta \) is the pulse duration, \( \gamma \) is the gyromagnetic ratio, \( \tilde{G} \) the vectorial intensity and direction of the applied magnetic field gradient, and \( \tilde{r}(t) \) the molecular trajectory. Had molecular diffusion been frozen, \( \varphi \) can be expressed as a series of motion derivatives:

\[
\varphi = \gamma \delta \Delta \tilde{G} \cdot \tilde{v} \left( \frac{\delta-\Delta}{2} \right) + \frac{1}{12} \gamma \delta \Delta (\Delta^2 + \delta^2) \tilde{G} \cdot \frac{\partial^2 \tilde{v}}{\partial v^2} \left( \frac{\delta-\Delta}{2} \right) + \cdots
\]

High-order terms can be neglected when molecular displacements during the sequence remain smaller than pore size, and the collected complex NMR signal on the whole sample then reads:

\[
S(\tilde{G}) \propto \int p(\tilde{v}) \exp(i \gamma \delta \Delta \tilde{G} \cdot \tilde{v}) d\tilde{v}
\]

The probability density function \( p(\tilde{v}) \) (pdf) on local velocity values is retrieved by sampling \( S(\tilde{G}) \) for different \( \tilde{G} \) values following well-known Shannon rules, and numerically inverting the Fourier transform (2). In this work, \( \tilde{G} \) was varied along one direction only (longitudinal of transverse to the geometry axis), so as to get one-dimensional statistics about solely the longitudinal or transverse (in y direction) velocity component.

The sequence used in this work is depicted in Fig. 4. It relies on a multiple echo train with alternating gradient pulses in order to compensate inhomogeneities of the magnetic field owing to the magnetic susceptibility of the sample itself, which are known to be a major source of bias otherwise (Williams et al. 1978; Latour et al. 1993). Due to imperfections of the gradient system inherent to any MRI facility, the sequence was terminated by a space selective module to restrict measurements to the central area of the flow geometry. Its design is robust against intermitent flow at the exit of the geometry. Depending on flow rate, the number of repetition in the alternated gradient train ranges from \( n = 0 \) to \( n = 3 \). Coherence pathway selection is performed by means of a cogwheel phase cycling which never exceeds 32 steps.
10-cm-high central area where field gradients exhibit the best linearity.

Molecular diffusion usually results in a blurring on measured pdf. We showed in the present case that this blurring can be kept under some undetectable level provided specific values of δ, Δ and $\hat{G}$ are used, and measurements are made only on the droplet phase (Chevalier et al. 2014b). Phase selectivity can be performed in a number of ways (Voda and Duynhoven 2009) and was achieved here by isolating the water component in acquired time-domain signal.

Although PFG is of wide use for the study of molecular diffusion, mono- and multiphase transport and/or hydrodynamic dispersion in porous systems, this technique has not been frequently used so far for true rheological purposes. Let us exemplify the specific interest of velocity pdf within the frame of the study of yield stress fluid flows. We consider the simple case of a steady state uniform flow through a straight cylindrical conduit. For a Herschel–Bulkley fluid, the momentum equation can be solved to obtain the statistical velocity distribution, from which we can deduce the theoretical expression of the velocity pdf $P(v)$ (Appendix 1). $P(v)$ is represented in Fig. 5 for two different values of $Bi$: one much smaller and the other much larger than 1. In the former case, the sheared region is wide and no significant plug flow appears; in the latter case, the sheared region is very thin and plug flow occupies most of the conduit (inset of Fig. 5). These characteristics can be observed on pdf curves (Fig. 5): $P(v)$ at small Bingham number exhibits a peak associated with the maximum velocity around the central axis of the conduit; the area under the peak is rather small, whereas the rest of the curve shows a large plateau-like region reminiscent of Newtonian behavior in cylindrical geometries. On the contrary, at high Bingham number, $P(v)$ more distinctly differs from the Newtonian case, with very low values at low velocities, and most of the distribution weight located under a very high peak close to $v/V = 1$. This example shows that with yield stress fluids, unsheared regions can be easily identified as they produce peaks in the statistical velocity distribution.

A direct measurement of velocity pdf gathers statistical information from every possible place in the pore space. It is not subjected to limited space resolution and so can provide valuable information concerning velocity inside geometries with very small pores, especially when direct imaging is not possible. In this context, the main source of artifacts in our study was the excess of immobile fluid due to slight over filling of the small pores setup.

2.4.4 Velocity statistics from MRI pictures

Since both MRI and PFG NMR can be performed on the big pore geometry, the latter sample was used for a quantitative comparison of both experiments. To do so, velocity statistics are computed from the 2D MRI velocity maps. Such approach was already reported in (Kutsovsky et al. 1996; Sederman et al. 1997), although none of these authors gave any detail about their processing procedure. From our own experience, we found that naïve approaches consisting in arranging pixel-wise data in a velocity histogram lead to very bad, scattered and unusable statistics. Only the exact probability density function of an interpolated velocity field over the whole picture permitted to restore satisfactory results and to avoid most problems related to the discrete nature of raw MRI data. In this work, a linear interpolation between neighboring pixels was found sufficient. On purpose of calculating the probability density function, each velocity was weighted by its radial coordinate so as to render velocity statistics in the whole 3D sample and not just in the imaged slice. The spatial windowing of PFG measurements was well rendered by processing only the 10-cm-high central zone of the pictures.

2.4.5 Integrated mechanical deformations from MRI pictures

This last post-processing approach, specifically devoted to the study of yield stress fluid flows, aims at identifying, from a velocity picture, the material regions which are in the solid regime and those which are in the liquid regime. It was recently developed to study the boundary layer around an object moving through a yield stress fluid (Boujlel et al. 2012). It consists in following the successive mechanical deformations undergone by a fluid element when it travels through the flow geometry.
In a first step, we reconstruct the various stream lines. For a given line, a starting point is taken at the entrance of the geometry, and the total deformation given by the second invariant of the Green–Lagrange tensor is then integrated as a function of the curvilinear coordinate. Ramps and plateaus in the resulting curve can then be interpreted in terms of local rheological behavior. As compared with the previous work, the use of the linear interpolation of velocity field turned out a major improvement. Details on processing steps are given in Appendix 2.

3 Flow through a model pore: general characteristics

Unless otherwise specified, this section focusses on flow characteristics when the mean velocity in the entrance duct is set to $V = 0.16$ mm/s. The corresponding Bingham number is very large and implies that yielding properties of the fluid a priori play a major role in flow characteristics. However, it will be shown later that this is actually not the case, and that flow characteristics are very similar in the whole range of $Bi$ tested. Data presented below are then representative of all observed flow characteristics in our range of parameters.

3.1 Velocity map

A typical 2D map of the norm of velocity vectors (i.e., $\sqrt{v_r^2 + v_z^2}$ where $v_r$ and $v_z$ are the radial and longitudinal components of the velocity field, respectively) in the big pore is shown in Fig. 6. Three regions can be distinguished as (1) a region (blue) around the central axis where the velocity field takes relatively high values and is approximately uniform, (2) an outer region (red) along the box’s walls where the fluid is apparently at rest and (3) an intermediate sheared layer (green) between the two latter regions.

From the complete quantitative velocity map (not shown here), profiles of the longitudinal velocity component can be extracted versus the radial coordinate for any given longitudinal position. In particular, such profile can be observed at various positions in both entrance and exit ducts. Figure 7 shows averaged profiles over the last 0.5 cm before entering and the first 0.5 cm after exiting the cylindrical box, respectively. They appear to be in close agreement with the theoretical profile (Appendix 1) expected for a steady uniform flow in an infinite straight conduit. In addition, no significant evolution of the velocity profile is observed upstream from the entrance or downstream beyond the exit of the box. Thus, at a first approximate flow characteristics in the entrance and exit ducts are not affected by the presence of the central cavity.

We now focus on the evolution of flow characteristics along the box. First of all, an expansion and a contraction can be noticed just after the entrance and just before the exit of the box, respectively (Fig. 6): The radial width of the moving region indeed first increases (entrance) and then decreases (exit). This effect is confirmed through the observation of velocity profiles along the central axis. Let us focus on the radial width of the flowing region (Fig. 8a) that starts from 37.5 mm at the entrance duct and reaches a value around 43 mm in the center of the box. At the same time, the maximum velocity at $r = 0$ decreases from about 0.16 to 0.14 mm/s. The exit process behaves the opposite way: The radial width of the flowing region decreases, while the maximum velocity at $r = 0$ increases (Fig. 8b).
The expansion and contraction of the flowing region develop over a longitudinal length of about 2 cm. It is worth noting that flow characteristics are on the contrary quite independent on the longitudinal position in a 3-cm-high center area: All velocity profiles in this region superimpose (Fig. 8c).

Looking at different longitudinal velocity profiles along the box, there is at first sight an unsheared region in the central part of the profile. The fact that this situation persists throughout the box suggests that this corresponds to a solid region which remains intact while it moves through the box. However, the situation is not as simple as for a uniform flow through a straight conduit: It has been said that the velocity and width of this solid region evolve during the penetration and exit of the box, which means that it is deformed. As a consequence, it is not clear whether this is effectively a solid region undergoing finite and transient deformations or a slowly flowing region in the liquid regime. In order to clarify that point, we need to follow the state of material elements all along their trajectory through the box. In that aim, we follow the approach described in Sect. 2.4.5.

A fluid element situated in the plug area of the entrance duct is supposed to be in the solid regime and undeformed before entering the box. Then, as it moves through the box, such a fluid element becomes liquid if the total deformation it has undergone is larger than the critical deformation of the material ($\gamma_c$). It is found that except in an outer layer of a few millimeters, the fluid in the upstream plug region (dotted lines in Fig. 9) undergoes a total deformation smaller than 0.1 before reaching the central plug region (Fig. 9). Only the elements initially very close to the wall of the entrance duct undergo much larger deformation (Fig. 9). Since in simple shear, the critical deformation beyond which our fluid turns from a solid to a liquid is about 0.2; this means that most of the central fluid part remains in its solid regime during the motion through the box. Since we do not have a precise view of the limit between unsheared and sheared regions in the entrance duct, it is difficult to elaborate more about the outcome of fluid elements in this zone. It can only be said that the plug may be very slightly eroded during its motion through the box, but it essentially remains in a solid state. That means despite the presence of the large volume of surrounding fluid in the box, the flow takes the form of a solid body going through the box with a thin sheared layer lubricating this motion.

### 3.2 Velocity probability density functions

Figure 10 shows velocity pdf obtained from either direct PFG measurements or image analysis. The two curves
Flow of a yield stress fluid through the big pore geometry \((V = 0.16\,\text{mm/s}, \; Bi = 36)\). Total deformation undergone by fluid elements along streamlines starting at different radial positions. From bottom to top: 0, 5, 10, 15, 16, 16.5 and 17.25 mm. The inset represents associated streamlines. Dotted lines are related to fluid elements starting in the plug area of the entrance duct.

![Probability density function of the longitudinal velocity component in the big pore geometry at \(V = 0.16\,\text{mm/s} (Bi = 36)\) computed from the velocity map of Fig. 6 (red line) and directly measured by the PFG NMR sequence (blue line).](image)

The velocity pdf thus provides an excellent appreciation of the existence of the main flowing regions, despite lost information on their respective locations.

On a more quantitative ground, the two curves nevertheless do not perfectly superimpose. In particular, a significant difference appears in the intensity of peak 2. Although both curves are supposed to concern only the part of the sample contained in a 10-cm-long zone around the pore center, the physical spatial selection performed during the PFG experiment does not have such sharp edges as the numerical windowing performed on the MRI picture. Thus, we believe that the actual fluid volume taken into account in ducts at the entrance and exit of the pore cavity for PFG measurements is larger in the latter case.

A second difference concerns the plateau between peaks 1 and 3—mainly corresponding to the green sheared region in Fig. 6—which looks more intense in PFG data. This cannot just be attributed to flow and/or sample variability between the two experiments. We believe instead in a more subtle effect, owing to possible chaotic dynamics of emulsion droplets in highly sheared zones. Since PFG is sensitive to any kind of motion, such effect may enhance locally the apparent random mobility of water molecule—and so the apparent diffusion coefficient—and would result in the PFG experiment in an enhanced blurring of the velocity probability between peaks 1 and 3, far beyond the capabilities of the ‘anti-blurring’ sequence tuning. On the contrary, the MRI picture is quite insensitive to this effect. The imaging process indeed performs a pixel-by-pixel averaging of fluid motion, so as to retain only the average fluid velocity, thus canceling to some extent the influence of diffusive and/or random molecular motions.

In the present work, this effect was regarded as minor and not further investigated. Our interpretations of velocity probabilities will instead focus on the positions of peaks 1, 2 and 3, which are largely insensitive to the measurement technique.
4 Flow characteristics through a model pore versus the Bingham number

Velocity maps obtained for different average velocities in the big pore (7-cm box) are shown in Fig. 11. The color scale on each picture was adjusted according to the maximum velocity. Except for the highest velocity, all pictures look quite similar. This means that the velocity maps rescaled by the average velocity are similar: There is no significant evolution of flow characteristics in the explored range of Bingham numbers.

Figure 12 compares radial profiles of the longitudinal velocity component extracted from the uniform region (i.e., between \( z = -1.5 \) cm and \( z = 1.5 \) cm) for the different Bingham numbers. At first sight, they all look similar provided the velocity is rescaled by the average velocity; they consist of a central plug surrounded by a homogeneously sheared region (i.e., the slope of the velocity profile is approximately constant, so that the shear rate looks uniform); the thickness of the central plug is almost independent of the Bingham numbers. A precise look at the data shows that the sheared thickness \( \delta \) increases from 5 to 8.2 mm (±10 %) when \( Bi \) decreases from 36 to 7.0. This thickness is well above the space resolution of MRI pictures, so that velocities in the sheared zone can be trusted.

Fig. 11 Flow of a yield stress fluid through a model pore (7 cm diameter). Velocity map in a longitudinal cross-section for \( Bi = 36, 22, 14.5, 12, 8.9 \) and 7.0. Color correspondence to velocity amplitudes (in micrometers per second) are given in bottom right insets. The fluid moves downward (white arrow)

Fig. 12 Radial profile of the longitudinal velocity component averaged over 3 cm in the center of the box (scaled by the average velocity in the entrance conduit) for the flow of yield stress fluid through a box for \( Bi = 36 \) (open circles), 22 (stars), 14.5 (filled circles), 12 (open squares), 8.9 (cross squares) and 7.0 (dotted line)
the big pore, this relative difference slowly decreases with 
its origin in the fact that the flow starts to involve a wider range of velocities, in particular around the plug region.

A way to quantitatively study pdf evolutions versus the Bingham number consists in following evolutions of the relative shift between the two peaks on the velocity axis, namely $(v_2 - v_1)/V$. In our range of Bingham numbers in the big pore, this relative difference slowly decreases with $Bi$ (Fig. 14), in agreement with the trends observed concerning plug velocities. Figure 14 also shows similar evolution at high Bingham numbers between $(v_2 - v_1)/V$ and the—rescaled—sheared layer thickness in the center of the box in the big pore. This may suggest the existence of an independence between the two information, which can, however, not be further investigated here due to insufficient data.

Further information can be obtained concerning the structure of the flow by looking at velocity pdf in the small pores (Fig. 15). Peak 2 now appears more intense than in the previous case: Indeed, in the small pores geometry, the 10-cm-long PFG window contains several replicas of pore cavities and connecting (small) ducts, which tends to better render the actual relative volumes associated with each region.

At low Bingham number values, peaks 1 and 2 are visible, but still close to each other. The relative velocity difference evolves in a way similar to that observed in the big pore in the same range of Bingham values (Fig. 14), thus supporting the hypothesis of data similitude between the two geometries. Indeed for such creep flows, flow characteristics in geometries reproducing the same shape at different sizes are expected to be similar for similar $Bi$ values.

When the Bingham number is decreased below six, the distance between the two peaks increases significantly (Fig. 15a,b), thus leading to a strong increase in $(v_2 - v_1)/V$ and connecting ducts in the same range of $Bi$, which further confirms the consistency of PFG measurements.

The above analysis of relative peak positions in velocity pdf left apart information related to amounts of fluid associated with different flow regions. To do so, an alternative way to observe the data is provided by the so-called statistical velocity distributions $D(v)$. It is nothing but the integral function of $P(v)$, so that $D(v)$ represents the probability to have a velocity below some given value. $D(v)$ is null for negative velocities. It then exhibits a steep increase around zero due to the first peak in $P(v)$ associated with the fluid at rest.

Two humps appear later in the curve, the second one leading to the ending plateau (Fig. 16), and correspond to peaks 1 and 2. There are not much marked for $Bi > 10$ (inset in Fig. 16) and start to clearly appear under approximately $Bi = 7$. The first bump progressively extends over a wider range (down to the end of the first increasing part.
of the curve for $Bi \approx 1$ as the Bingham number decreases, suggesting that the flowing region extends in the radial direction inside the box or more precisely that the sheared region is much thicker inside the moving region.

At last, it is worth noting that this curve tends to saturate for large $Bi$ (>10) (inset in Fig. 16), which contrasts with the situation for smaller $Bi$ (Fig. 16) and illustrates again the tendency of the fluid to form a kind of shear-band of finite thickness at high $Bi$.

5 Flow through bead packing

PFG measurements in bead packing are shown in Fig. 17 in terms of both velocity pdf $P(v)$ and distribution $D(v)$.

Fig. 15 Rescaled pdf of the longitudinal velocity component in small pores for different values of the Bingham number: a 15 (orange), 13.6 (dark blue), 8.8 (red) and 5.4 (light blue); b 3.5 (green), 2.2 (black), 1.4 (pink) and 0.9 (gray)

Fig. 16 Distribution of the longitudinal velocity component in small (main graphic) and big (inset) pores as a function of rescaled velocity for data of Fig. 15 (same color correspondence). Labels A and B mark the end of the two humps (see text)

Fig. 17 Probability density functions (a) and associated distributions (b) of the longitudinal velocity component as a function of velocity, scaled by the mean velocity $V$ in the pore space, for $D = 2$ mm (dot lines) and $D = 0.5$ mm (continuous lines) bead packing at different flow rates: $V = 0.04$ mm/s (light blue), $V = 0.12$ mm/s (dark blue), $V = 0.4$ mm/s (green), $V = 1.2$ mm/s (red) and $V = 5$ mm/s (black). Explored Bingham numbers range between 1 and 7.3
For the lowest flow rate (light blue curves in Figs. 15, 16), experimental conditions lie at the very limit of the parameter space which allow PFG measurements of instant velocities to be performed (Chevalier et al. 2014b). Observed oscillations stem from the strongly depreciated SNR.

$P(v)$ curves in Fig. 17 strikingly differ from those in model pores (Fig. 15a): Whatever the flow rate, no sharp peak is found exactly centred on zero velocity. $P(v)$ instead rapidly increases at some strictly positive velocity value and then progressively decreases toward zero. There is no isolated peak for any specific velocity. Instead, the statistical velocity distribution well spreads over some range and does exhibit neither any well-defined plug flow nor any stopped region occupying a significant volume as compared to the whole fluid volume in the sample. These observations are confirmed by statistical velocity distribution functions (Fig. 17b): They do not show any step at zero or at any finite velocity value as was observed for model pores (Fig. 16).

The absence of sharp step is not so surprising: When flowing through a non-periodic real porous medium, the fluid encounters pores of various sizes so that its local flow rate is prone to widely vary from one pore to another. It then seems impossible that a plug flow at a given velocity—as that found in a single straight model pore—could subsist over a significant distance. The absence of yielding region (at zero velocity) is much more surprising.

Another surprising result concerns data similarity when rescaled by the mean velocity: Except for the lowest flow rate, all distributions of the longitudinal velocity component look similar (Figs. 15a, b). A similar result is obtained for transverse velocity (Fig. 18). This result would not be so surprising for a Newtonian fluid at low Reynolds number since it is expected that the fluid flows in the whole medium (even in so-called dead zones where vortices take place), whatever the flow rate, and that flow similarity between different flow rates and samples of different sizes should be maintained due to linear behavior of the Stokes regime. For viscoplastic fluids at Bingham number of the order or larger than 1, however, constitutive rheological equations of the fluid are strongly nonlinear and then flow characteristics should significantly evolve with the Bingham number. For example, a significant evolution of statistical velocity distribution was found in model pores when Bingham number decreases from 10 to 1 (Fig. 16).

In a recent work, it was shown that these distributions functions are also similar to those of both a Newtonian and a shear-thinning fluid (Chevalier et al. 2014a). This result along with the similarity of rescaled distributions was further analyzed suggesting that velocity field for a yield stress fluid in such a complex geometry is very close to that of a Newtonian fluid. This effect would result from the fact that the fluid is continuously and significantly deformed in various directions as it moves through a complex network, so that all occurs as if its deformation were imposed, leading to a velocity field essentially independent of its constitutive equation, contrary to what would occur if the flow characteristics were controlled by stress distribution. This original result needs to be further studied, but this is out of the scope of our present work which focuses on the interpretation frame of MR data for studying the specificities of yield stress fluid flows.

6 Conclusions

In this work, we have investigated ways to analyze MRI velocimetry and velocity PFG data so as to study in-depth properties of confined yield stress fluid flows. In large geometries, MRI velocity fields can be imaged sufficiently precisely to determine the location of solid and liquid regions, and get a quantitative insight of sheared areas. Tracking fluid deformations along stream lines also brings a further analysis of these data and can show how fluid elements possibly evolve from the solid to the liquid state. Measuring velocity statistics with the PFG technique provides data consistent with these observations. On purpose of a quantitative analysis, we found that the use of the integrated statistical velocity distribution can be more convenient than the more traditional $pdf$ representation. In particular, it permits to determine the fluid fraction at rest and to estimate the fluid fraction moving as a plug. We nevertheless observed some discrepancy between PFG and imaging data in highly sheared regions, suggesting that some progress is still possible in this field.
In the case of a disordered porous system, velocity PFG can still be used to extract critical information concerning flow characteristics. Statistical distributions provide a fingerprint of the flow at microscale, and permitted here to uncover significant similarities between confined flows of yield stress and Newtonian fluids. Granted the complex geometry of such systems, pure velocity PFG may constitute an easy way to get a quantitative and meaningful rheological characterization of complex fluids under restricted conditions.

Appendix 1: Flow characteristics of a yield stress fluid in a circular tube

Flow profile

We consider the laminar flow of a fluid obeying Herschel–Bulkley model Eq. (1), through a circular tube of radius $R$, length $L$ and submitted to a pressure gradient $\Delta p$. At each radial position $r$, the momentum equation applies:

$$\tau = (\Delta p/2L)r.$$  

Incorporating this expression in the constitutive equation, we deduce the following expression for the velocity profile:

$$v(r) = v_{\text{max}} \left(1 - \frac{(r - r_c)}{r_c}ight)^{1+1/n}$$

where $v_{\text{max}}$ is the maximal velocity and the critical radius, $r_c = 2LT/\Delta p$, delimits the frontier between the flow and the plug zones. In the circular tube, the Bingham number $Bi = \frac{\tau_c}{V^2}$, with $V$ the average velocity, is as follows:

$$Bi = \frac{\tau_c}{V^2} = \left(\frac{r_c}{2L} v_{\text{max}} \left(\frac{r_c}{2L} + 1/n\right)^{1+1/n} \right)$$

Probability density function and distribution

The statistical velocity distribution $D(v_0)$ is defined as the volume fraction of the fluid flowing with a velocity $v < v_0$. It actually corresponds to the fluid fraction contained between the conduit surface and the radial position $r_0$ such as $v_0 = v(r_0)$. One then gets:

$$D(v_0) = \int_{v_0}^{v_{\text{max}}} \frac{v^{n-1}}{\Gamma(n)} \exp(-v) \, dv$$

where $r_0$ is regarded as an implicit function of $v_0$ and can be determined through the inversion of Eq. (3).

The velocity pdf is deduced from Eq. (4) following its definition

$$P(v_0) = \frac{\partial D}{\partial v_0} = -\frac{2n r_0}{R^2} \frac{\partial r_0}{\partial v_0}$$

$$P(v) = \chi(0 < v < v_{\text{max}}) \left(\frac{r_c + (R - r_c) \left(1 - \frac{v}{v_{\text{max}}}\right)^n}{n+1}\right)$$

with $\chi = 1$ if $0 < v < v_{\text{max}}$ and zero elsewhere and $\delta(v-v_{\text{max}})$ is a Dirac delta function respect to the velocity. Let us emphasize that in the Newtonian case, $r_c = 0$ and $n = 1$, such that all velocities share the same probability $P(v) = 1/v_{\text{max}}$.

Appendix 2: Deformation integration along streamlines

Starting from an initial position, trajectories are built as a series of successive elementary steps of fixed length $dl$ performed in the direction of the local interpolated velocity field $(V_{x}(r, z), V_{y}(r, z))$. Due to interpolation, radial position $r$ and longitudinal position $z$ are considered as continuous parameters. $dl$ is chosen small regarding image resolution. The duration of each step, is evaluated following

$$dr = dl/\sqrt{V_{x}^2 + V_{y}^2},$$

and the displacement gradient $\nabla u$ during this step is computed in cylindrical coordinates following Boujlel et al. (2012)

$$\nabla u = dr \begin{pmatrix} V_{r} & 0 & V_{z} \\ 0 & V_{r} & 0 \\ V_{z} & 0 & V_{zz} \end{pmatrix}$$

The Green–Lagrange strain tensor related to each step $e$ is evaluated following

$$e_{ij} = \frac{1}{2} (u_{ij} + u_{ji})$$

Within the hypothesis of small deformations, the collected strain $e_{\text{total}}$ along the trajectory is obtained as the simple sum of successive $e$ operators at each step. The second invariant $e_{II} = \sqrt{1/2 \left( (\text{tr}(e_{\text{total}}^2) - \text{tr}(e_{\text{total}})^2) \right)}$ of thus integrated deformation is finally calculated for each intermediate point of the trajectory.

References


References


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