Is microrheometry affected by channel deformation?

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Microrheometry is very important for exploring rheological behaviours of several systems when conventional techniques fail. Microrheometrical measurements are usually carried out in microfluidic devices made of Poly(dimethylsiloxane) (PDMS). Although PDMS is a very cheap material, it is also very easy to deform. In particular, a liquid flowing in a PDMS device, in some circumstances, can effectively deform the microchannel, thus altering the flow conditions. The measure of the fluid relaxation time might be performed through viscoelasticity induced particle migration in microfluidics devices. If the channel walls are deformed by the flow, the resulting measured value of the relaxation time could be not reliable. In this work, we study the effect of channel deformation on particle migration in square-shaped microchannel. Experiments are carried out in several PolyEthylene Oxyde solutions flowing in two devices made of PDMS and Poly(methylmethacrylate) (PMMA). The relevance of wall rigidity on particle migration is investigated, and the corresponding importance of wall rigidity on the determination of the relaxation time of the suspending liquid is examined. © 2016 AIP Publishing LLC. [<http://dx.doi.org/10.1063/1.4945603>]

I. INTRODUCTION

In the last decades, microrheometry, i.e., the measure of rheological properties through microfluidic techniques, received great attention from the scientific community.¹ Indeed, materials properties such as shear viscosity,² elongational viscosity,³ and more recently relaxation times can all be obtained by means of microfluidic devices.^{3–5} The main advantages of microrheometry reside in the very small amount of sample needed for the measurements (a very important issue for precious materials), and in the higher sensitivity possibly achievable with respect to conventional techniques, for some rheological properties at least.⁵

In several instances, the measure of a characteristic relaxation time presents several difficulties. For example, fluids relaxation times down to milliseconds cannot be detected by conventional small angle oscillatory shear techniques simply because values of the storage modulus G' are extremely small ($\sim 10^{-3}$ Pa) in the attainable frequency range.^{5,6} Recently, then, Zilz *et al.*⁴ proposed a microfluidic technique for the measure of the fluid relaxation time based on phenomena of flow-induced instability in a curved geometry, a serpentine microchannel in their work. This method, however, gives data with nonnegligible error bars due to the delicate nature of the flow instability. In our previous work,⁵ we presented an alternative (and more accurate) way for measuring the longest fluid relaxation time in a microfluidic device, based on the viscoelasticity induced cross-flow migration of particles transversally to the main flow direction.⁷ Particles suspended in a non-Newtonian matrix and subjected to a confined Poiseuille flow, indeed, migrate transversally to the flow direction⁷ towards one or more equilibrium positions

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depending on channel geometry^{8,9} and on fluid rheology.¹⁰ In particular, particles suspended in a constant-viscosity but elastic fluid and subjected to a Poiseuille flow in a square-shaped microchannel migrate towards the centreline,^{9,11} while particles suspended in a shear-thinning elastic fluid, in the same geometry, migrate towards the four corners.^{9,11} When particles migrate towards the channel centreline, we showed⁵ that the fraction of particles aligned at the centreline is related to the relaxation time λ : hence, by determining the suspended particle distribution far from the channel inlet, we are in fact able to measure the (longest) relaxation time of the suspending medium.

While our previous analysis^{5,9} has been carried out in Poly(methylmethacrylate) (PMMA) microchannels, most of the microrheometrical measurements available in literature have been carried out in Poly(dimethylsiloxane) (PDMS) channels, bonded on a glass coverslip. In fact, PDMS is the most used material for the fabrication of microfluidic devices.¹² The success of PDMS is related to the versatility of its fabrication process when used in combination with microchannel molds.¹³ Those molds are produced through photolithographic techniques,¹⁴ which allow for spatial resolution of around $0.1\ \mu\text{m}$.¹² Even though PDMS is very cheap, mold fabrication requires expensive instrumentation based on the use of lasers.¹² For this reason, researchers tried to use different channel materials. In the case of channels with very simple geometry, PMMA is often preferred.¹⁵ At this stage, a question naturally arises, namely, whether or not the channel material influences microrheometrical measurements. One expected difference between PDMS and PMMA resides in channel wettability.^{16,17} The velocity profile measured in a microchannel with hydrophilic walls (PMMA¹⁸) is different from that measured in microchannels with hydrophobic walls (PDMS):¹⁷ hydrophobicity enhances the wall slip phenomenon, and, therefore, velocity and stress profiles within the channel cross-section will both be affected. In this regard, Graca *et al.*¹⁹ showed that PDMS hydrophobicity can be finely tuned by adding surfactants: even a small amount of surfactants (1 mM concentration) strongly increases channel wettability, as shown by the pronounced decrease of the measured contact angle.¹⁷

Another difference between PDMS and PMMA is due to material stiffness.²⁰ PDMS is an elastomer with a Young modulus $E \sim 1\ \text{MPa}$,¹² while for PMMA $E \sim 1\ \text{GPa}$.²¹ PMMA can then be regarded as essentially rigid. The effect of wall stiffness on fluid-dynamic in microchannels has itself received great attention in the last decade.^{20,22–25} Gervais *et al.*²² and Hardy *et al.*²⁰ theoretically and experimentally showed that PDMS channel walls are deformed under the effect of an imposed pressure-driven flow. These results were also confirmed in more recent experimental studies.^{23,25} In particular, by increasing the flow rate, i.e., by increasing the stress acting on the channel walls, the channel is more and more deformed. This phenomenon acquires then a great importance in several microfluidics applications such as those related with inertial particle migration and focusing.^{26,27} In fact, very high Reynolds numbers, i.e., high flow rates, are required for exciting inertial forces. Notice that all the just mentioned works were conducted on the flow of Newtonian fluids only. Very recently, it has been shown that non-Newtonian fluids in pressure-driven flows can give fluid instability even in straight channels with walls made of soft materials.²⁵

Thus, rheology in a micro channel depends on the properties of the channel walls. As discussed above, microfluidic techniques based on particle migration in microchannel have been proved to be reliable for deriving the fluid relaxation time; those experiments, however, have been only carried out in PMMA devices. In this work, we study the effect of the channel material on particle migration in viscoelastic fluids. We present results for both the migration towards the centreline and the migration towards the corners in a square-shaped microchannel, made of PDMS (soft) and PMMA (stiff) material. Finally, the measure of the fluid relaxation time λ through particle migration is reported for both devices, under the same imposed fluid-dynamic conditions.

II. MATERIALS AND METHODS

A. Suspending fluids and particles

PolyEthylene Oxyde (PEO) with average molecular weight $M_w = 4000\ \text{kDa}$ (Sigma-Aldrich) at several mass concentrations ranging from 0.08 wt. % to 1.6 wt. % in water was used. Glycerol

(25 wt. %) was added to the solutions in order to prevent particle sedimentation.⁹ The viscosity of the glycerol-water solution is $\eta = 2 \times 10^{-3}$ Pa s. The fluid rheological properties are measured by a stress-controlled rheometer (Anton Paar MCR 302 rheometer), with cone and plate geometry with diameters of 50 mm and with a solvent-trap to avoid fluid evaporation. The fluids rheology is reported in Fig. 1.

Fig. 1(a) reports the relative shear viscosity (with respect to the glycerol-water mixture) of the different PEO aqueous solutions as a function of the Deborah number $De = \lambda \dot{\gamma}$, where $\dot{\gamma}$ is the shear rate. The PEO solution at low concentration (green symbols) shows a constant-viscosity behaviour in the whole range of Deborah numbers investigated. As the concentration is increased to 0.5 wt. % (orange symbols), a weak shear-thinning appears for $De \sim 1$. Finally, for the most concentrated PEO solution (blue symbols), a remarkable viscosity-thinning is observed beyond $De \sim 0.1$ with the viscosity reducing by one order of magnitude over two decades of Deborah numbers. The flow curves at the other concentrations are not reported because the solutions behave similarly to the PEO 0.1%.⁵

Fig. 1(b) reports the linear viscoelastic response for PEO 1.6 wt. %, for an imposed deformation $\gamma = 5\%$ and over three frequency decades, respectively. The PEO 1.6% shows the typical trends of the moduli at low frequencies, i.e., $G' \propto \omega^2$ and $G'' \propto \omega$. Then, we estimate the fluid relaxation time by the intersection of the straight lines at low frequencies giving $\lambda \sim 0.18$ s. By using our own microrheometer,⁵ we measured the fluid relaxation time of the PEO 0.5%, obtaining $\lambda \sim 6$ ms. We use these values for the study of particle migration reported in Section III. The relaxation time of the others PEO solutions is also reported in Section III.

Polystyrene (PS) particles (Polysciences) with diameter $D_p = 10 \mu\text{m}$ and density $\rho_p = 1.05 \text{ g/l}$ are used. A dilute suspension with a volume fraction $\phi = 0.01\%$ is prepared. Particles are added to the matrix that is put first in a mixer (Vortex, Falc Instruments) to guarantee a good dispersion and then in an ultrasonic bath (Falc Instruments) to remove air bubbles. This procedure is repeated before each experiment.

B. Microfluidic device

Experiments are carried out in two square-shaped cross-section channels made of PMMA (substrate thickness 1 mm, Goodfellow) bonded on PMMA and of PDMS bonded on glass, fabricated by following our previous works.^{9,28} The channel height H for both the devices is $H = 100 \mu\text{m}$. Some clarifications need to be done for the PDMS device bonded on a glass coverslip (see Fig. 2(b)). The thickness H_{Th} of the whole PDMS device is $H_{Th} \sim 4 \text{ mm}$ and the lateral thickness L_{Th} is $L_{Th} \sim 25 \text{ mm}$ (see Fig. 2(b)). In addition, small amount of Tween 20 (Sigma Aldrich) was added in order to change the surface wettability of the PDMS.¹⁹

FIG. 1. (a) Measured steady relative shear viscosity η_r for the aqueous PEO solutions at different concentrations as a function of the Deborah number De . The relative viscosity η_r is calculated with respect to the viscosity of the solvent (a glycerol-water mixture with $\eta = 2 \times 10^{-3}$ Pa s). (b) Measured elastic modulus G' (green triangles) and loss modulus G'' (red circles) for the aqueous PEO 1.6 wt. %. The slopes of the straight lines are 2 and 1 for G' and G'' , respectively, and indicate the expected frequency dependence in the “terminal region” of a viscoelastic fluid.

FIG. 2. (a) Schematic representation of the channel used in the experiments. A smooth entrance at the channel ends is fabricated in order to guarantee an uniform particle distributions. In the figure, the relevant dimensions are reported. The dimensions along X and Z are not in scale. (b) Frontal view of the PDMS-glass device. The lateral thickness L_{Th} and the thickness H_{Th} of the PDMS device are reported.

The Young modulus of PDMS is $E \sim 1$ MPa,²² while that for the PMMA is $E \sim 3$ GPa (from the material data sheet).

A schematic picture of the channel geometry is reported in Fig. 2(a). The entrance (triangular shape from the top view, left side of Fig. 2(a)) usually assures a smooth velocity field from the inlet to the main channel.⁹ When dealing with PEO dilute solutions, however, the fluid can recirculate within the device inlet giving rise to vortex formation.²⁹ Nevertheless, due to the relatively small designed angle at the inlet $\alpha = 20^\circ$, and in the range of Deborah number investigated, we can safely assume that no recirculation phenomena are present. In addition, we did not observe an anomalous particle distribution at the channel entrance. The relevant dimensions of the device are shown in Fig. 2(a).

The flow rate is controlled by a syringe pump (Nemesys) using glass syringes. This machine is able to discriminate different flow rates with a precision of 1 nl/min. The applied flow rates vary in the range of $0.1 \mu\text{l/min} < Q < 3 \mu\text{l/min}$.

C. Particle positions

Particles flowing in the channel are observed using a straight microscope (Olympus BX-53) with a $4\times$ objective. Image sequences are collected with a fast camera (IGV-B0620M, Imperx) at a frame rate variable between 30 and 400 fps depending on the flow rate. The observations are all made at a fixed distance of $L = 8$ cm from channel inlet. All the experiments are performed at room temperature.

The velocity distributions are measured by particle tracking.⁹ Particle positions within the channel cross-section are derived by combining particle tracking visualisation and numerical simulations of the fluid velocity profile to locate a particle in a specific position of the channel cross-section. Denoting with Z the direction of the flow and with X and Y the directions parallel to the sides of the cross-section, the visualisation from the top of the device allows to measure the particle velocity $V_{p,Z}$ as well as the coordinate of the particle centre X_p .

By simulating the velocity profile $V_{sim,Z}(X_p, Y_p)$ of the unloaded fluid as reported in our previous work,¹⁰ the following equation can be solved:

$$V_{sim,Z}(X_p, Y_p) = V_{p,Z}, \quad (1)$$

and the value Y_p is thus computed. This methodology is strictly valid when the symmetry of the channel is respected. For the PDMS device, indeed, the procedure could not be used because of the asymmetry given by different channel wall materials (three walls of PDMS and one made of glass). However, particle positions reconstruction has been carried out by locating all the particles in one square only, corresponding to their absolute values of X and Y coordinates.

III. EXPERIMENTAL RESULTS AND DISCUSSION

We first present experimental results on the normalised particle velocity as a function of the dimensionless coordinate $|X|$ in Fig. 3. Notice that the abscissa in the figure is in fact the absolute value of the X -coordinate, thus negative and positive X -positions are all included. Fig. 3(a) shows that the majority of particles suspended in a PEO solution 0.5% flows with the highest possible velocity, i.e., $v/v_{max} \sim 1$, and is located close to $|X| = 0$. In other words, by looking at the device from an upper view, most particles are fast, hence they are performed distributed around the Z -axis. Notice that particles are located close to $|X| = 0$ in both the PMMA and PDMS devices. Even at this low flow rate, a hint on a difference in the migration behaviour in the two devices can be caught. Particles suspended in a PEO 0.5% solution flowing in the PMMA device are in a isotropic cloud around the axis of the channel, whereas particles in the PDMS device are distributed on a line in Fig. 3(a), thus indicating some preference towards the XZ -plane. On this point, see also the comments on Fig. 4 below.

Figs. 3(b) and 3(c) report again the measured normalised velocity of the particles as a function of the dimensionless coordinate $|X|$, for the more concentrated (1.6%) PEO solution, at two De -values. In both those figures, at $De = 1.5$ and 3, respectively, the effect of the nature of the wall on particle distribution becomes apparent, as data obtained with different devices form different clouds. Indeed, in Fig. 3(b), most of the particles flowing in the PMMA device have positions $|X|_p$ located in a range of $0.15 < |X|_p < 0.25$; an average normalised particle velocity

FIG. 3. Normalised particle velocity as a function of the dimensionless coordinate $|X|$ for particles in both PMMA and PDMS devices for (a) PEO 0.5% at $De = 0.15$, (b) PEO 1.6% at $De = 1.5$, and (c) PEO 1.6% at $De = 3$.

FIG. 4. Experimental results on particle migration in PEO 0.5%. (a) Particle positions at $De = 0.15$ in PMMA device. The symbols denote the position of the particle centres. (b) Particle positions, at $De = 0.15$ in PDMS-glass device. The dashed lines delimit the region accessible to the particles.

around 0.75 can be estimated. Particles flowing in the PDMS device, instead, are mainly located around $|X| \sim 0.3$, with a normalised average velocity around 0.5. Thus, particles flowing in the PMMA and the PDMS devices migrate differently. In Fig. 3(c), at $De = 3$, particles flowing in the PMMA device are still located around $|X| = 0.2$ with $v/v_{max} = 0.75$, whereas particle positions in the PDMS device are found to shift from $|X| \sim 0.3$ (at $De = 1.5$) to $|X| \sim 0.25$ (at $De = 3$), with a normalised velocity shifting to around 0.6.

An elaboration of the data appearing in Fig. 3 can show more clearly the influence of the device on migration. Figs. 4(a) and 4(b) show the particle positions plotted in a square $|X| - |Y|$ at $L = 8$ cm from the channel inlet at $De = 0.15$ for the PMMA and the PDMS devices, respectively. These data refer to the PEO solution at 0.5%, and the fluid behaves as a constant-viscosity liquid (see Fig. 1(a)). Within this type of representation, the particle distribution within the cross section becomes self-evident. Most of the particles are aligned on the channel centreline, but distribution tail is isotropic for the symmetric PMMA device and anisotropic for the asymmetric PDMS-glass device. Indeed, in the latter case, particles are all close to the XZ-plane.

Figs. 5(a) and 5(b) show the particle positions plotted in the square $|X| - |Y|$ at $L = 8$ cm from the channel inlet at $De = 1.5$ for the PMMA and the PDMS devices, respectively. As general illustration, it should be noted that at this De -value, the channel axis is no more reached by the particles, at least at this distance from the inlet. By comparing *a* and *b* panels, the different particle distribution in the two devices is apparent. For the PMMA device, the particles are closer to the corners, while for the PDMS-glass device many particles also crowd the lateral region, i.e., close to the side walls. Figs. 5(c) and 5(d) show the same representation of the particle distribution for the case at $De = 3$. A similar situation for the PMMA device is encountered, while a more pronounced lateral wall attraction is found for the PDMS-glass asymmetric device.

The experimental observations just reported clearly indicate a qualitative effect of the device wall nature on the viscoelasticity induced migration of the suspended particles. A possible explanation of this behaviour might derive from the possible deformation of soft walls. Indeed, PMMA and PDMS are very different in terms of rigidity. As mentioned in Section I, the relevance of channel stiffness on fluid-dynamics in microchannels has already been addressed. Indeed, Gervais *et al.*,²² through simulations and experiments, studied the deformation of the PDMS ceiling in a rectangular-shaped PDMS-glass microchannel as a function of the volumetric flow rate Q induced by flow of a Newtonian liquid (water). They proposed a scaling argument to qualitatively describe the channel walls deformation due to flow. For a Newtonian liquid in Poiseuille flow with a pressure drop Δp in a microchannel made of a material with a Young modulus E , Gervais *et al.* propose the following scaling law:

$$\frac{\Delta H_{max}}{H} \sim \frac{\Delta p}{E}, \quad (2)$$

FIG. 5. Experimental results on particle migration in PEO 1.6%. (a) Particle positions as in Fig. 4 at $De = 1.5$ in PMMA device. The symbols denote the position of the particle centers. (b) Particle positions as in (a), at $De = 1.5$ in PDMS-glass device. (c) Particle positions as in (a), at $De = 3$ in PMMA device. (d) Particle positions as in (b), at $De = 3$. The dashed lines delimit the region accessible to the particles.

where ΔH_{max} is the maximum variation of the channel height, possibly near to the channel entrance,²² and H is the channel height. In what follows, we will refer to PDMS only, as in this case the wall softness is important (finite Young modulus).

The pressure drop Δp can be evaluated from the Hagen-Poiseuille equation, which for square section channels and Newtonian liquids⁶ reads

$$\Delta p = \frac{3.56\eta LQ}{H^4}. \quad (3)$$

Thus, by combining the two latter equations, an estimate of the channel enlargement is easily obtained.

When particles are suspended in a non-Newtonian power-law liquid, with m being the consistency and n being the flow index, the pressure drop is³⁰

$$\Delta p = \left[\frac{\left(0.68 + \frac{0.21}{n}\right)Q}{H^3} \right]^n \frac{4mL}{H}. \quad (4)$$

In our experiments, data taken at $De = 0.15$ and PEO 0.5% might be envisaged as pertaining to a Newtonian liquid, since the rheological flow curve reported in Fig. 1 clearly indicates that the system is well within the Newtonian plateau of the viscosity. On the contrary, data taken at $De = 3$ and PEO 1.6% are well within the shear thinning region of the corresponding flow curve. Thus, for the two cases, the two different Δp equations indicated above apply.

When particles are suspended in PEO 0.5% at $De = 0.15$, the pressure drop is estimated as $\Delta p \sim 23$ kPa, thus $\Delta H_{max}/H \sim 2.3\%$: the channel deformation is therefore small.

The rheological properties of the PEO solution at 1.6% at large shear rates are well described with a power-law model with $n = 0.54$ and $m = 1.5$ (with these values the viscosity, $\eta = m\dot{\gamma}^{n-1}$, is given in Pa s). At $De = 3$, we can estimate $\Delta p \sim 1.2 \times 10^5$ Pa, thus $\Delta H_{max}/H \sim 12\%$. It is then plausible that channel deformation might play an important role and can effectively affect

FIG. 6. Comparison between experimental results on the fluid relaxation time λ derived with our “microrheometer” in the PMMA (grey circles) and PDMS channel (white inverted triangles), for PEO 4 MDa at different concentrations. Dashed line is the theoretical prediction for the semi-dilute unentangled regime.⁵ Error bars are evaluated by dividing the statistical samples in three sets and calculating the standard deviation between the three λ -values. Error bars smaller than the symbol size are not shown.

migration dynamics. In our experiments, an additional source of differentiation in particle migration arises, of course, from the asymmetric wall properties for the PDMS-glass device.

Two considerations seem now at order. (i) Migration towards the channel centreline, i.e., the behaviour expected at low De -values, is only slightly affected by the channel walls material. (ii) Outward migration, i.e., typical at large De -values, is prone to be affected by the channel walls material.

From a microrheometrical perspective, the just stated considerations suggest that some care should be taken when designing a microrheometer application. Indeed, particle migration phenomenon has been used in our previous work⁵ for measuring the fluid relaxation time λ . Consideration (i) suggests that the measure of the fluid relaxation time should not be affected by channel material, if the flow rate is within a *low* De condition. To confirm this, we present here the comparison of the fluid relaxation time measured through our home made microrheometer⁵ in both the PMMA and the PDMS devices, in order to assess the possible influence of the microchannel material on the estimated relaxation time following the protocol reported in our previous work.⁵ Fig. 6 shows the comparison between experimental results on the fluid relaxation time λ derived through our “microrheometer” in the PMMA (grey circles) and PDMS-glass channel (white triangles), for PEO 4 MDa at different concentrations, all of them below 0.5%. Indeed, as we demonstrated above, channel deformation is definitely very minor at these concentrations. As a matter of fact, the results from both devices in Fig. 6 almost superimpose and are in good agreement with theoretical predictions.³¹ Thus, a proper rheo-engineered device can be safely used to estimate the suspending fluid relaxation time, regardless of the wall properties.

IV. CONCLUSIONS

In this work, we investigate on the role of channel walls stiffness on viscoelasticity induced particle crossflow migration in a square-shaped microchannel. Experiments are performed in devices made of two different materials, the PMMA (stiff) and the PDMS (soft), respectively. On a general basis, softer walls and asymmetric wall properties do affect particle migration. Indeed, different particle distributions have been detected depending on the walls nature. More specifically, however, the fate of particle is less influenced, as focusing at the channel axis is observed at low De -values, and outwardly directed migration is found at larger De .

The observed effect of wall rigidity has been rationalised in terms of a scaling argument.²² This effect manifests itself through a possible gradient in channel deformation, and

consequently in a variation along the axis of the confinement ratio. We speculate that particle migration should be slower at the entrance, where the channel enlargement is expected to be large. Finally, we prove that the measure of the fluid relaxation time λ through particle migration in microfluidic devices might be unaffected by wall deformability if care is taken when selecting the flow conditions. Specifically, the dimensionless parameter $\Delta p/E$ must be small.

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