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# Unsteady laminar pipe flow of a Carbopol<sup>®</sup> gel.

ANTOINE POUMAERE <sup>a</sup>, MIGUEL MOYERS-GONZÁLEZ <sup>b</sup>, CATHY CASTELAIN <sup>a</sup>, TEODOR BURGHELEA <sup>a</sup>

a. LUNAM Université, Université de Nantes, CNRS, Laboratoire de Thermocinétique, UMR 6607, La Chantrerie, Rue Christian Pauc, B.P. 50609, F-44306 Nantes Cedex 3, France

b. Department of Mathematics and Statistics, University of Canterbury, Private Bag 4800, Christchurch 8041, New Zealand

## Résumé :

*L'écoulement non stationnaire d'un fluide à contrainte seuil (Carbopol 980) à bas nombre de Reynolds a été étudié expérimentalement dans un tube. Tout d'abord, lors de l'étude rhéologique, la transition solide-fluide en présence ou absence de glissement a révélé un couplage entre l'état de déformation irréversible et le phénomène de glissement en paroi. En particulier, la présence de glissement à la paroi supprime presque la dépendance entre les pertes associées à l'hystérésis rhéologique et le taux de déformation du matériau. La transition irréversible solide-fluide et le comportement de glissement à la paroi apparaissent dans la même gamme de contrainte appliquée ce qui induit un couplage fort entre les deux phénomènes. Les mesures du champ de vitesse réalisées durant la phase de montée et de descente de la rampe de pression mettent en évidence trois zones d'écoulement distinctes : solide, solide-fluide et fluide. Les mesures temporelles de ces écoulements, à bas nombre de Reynolds, obtenus par une rampe de pression contrôlée, montante ou descendante, révèlent un scénario irréversible à seuil semblable à celui observé lors des mesures rhéologiques. Les pertes associées à l'hystérésis observée durant la montée et la descente de la rampe de pression montre à quel point les résultats obtenus lors de l'étude de l'écoulement non-stationnaire sont en accord avec ceux observés lors des mesures rhéologiques en présence de glissement. La dépendance de la vitesse de glissement avec la contrainte de cisaillement à la paroi met en évidence un comportement de glissement de type Navier, seulement dans la zone d'écoulement, ce qui indique que le phénomène de glissement est directement couplé avec la transition solide-fluide.*

## Abstract :

*A experimental study of low Reynolds numbers unsteady pipe flows of a yield stress shear thinning fluid (Carbopol<sup>®</sup> - 980) is presented. The investigation of the solid-fluid transition in a rheometric flow in the presence and in the absence of the wall slip reveals a coupling between the irreversible deformation states and the wall slip phenomenon. Particularly, the presence of wall slip nearly suppresses the scaling of the deformation power deficit associated to the rheological hysteresis with the rate at which the material is forced. The irreversible solid-fluid transition and the wall slip behaviour emerge in the same range of the applied stresses and thus, the two phenomena appear to be coupled to each other. In-situ measurements of the flow fields performed during an increasing/decreasing stepped pressure ramp reveal three distinct flow regimes : solid, solid-fluid and fluid. The time resolved measurements of low Reynolds number unsteady flows driven by an increasing/decreasing controlled pressure ramps reveal a irreversible yielding scenario similar to that observed in the rheometric flows. The deformation power deficit associated with the hysteresis observed during the increasing/decreasing branches of the pressure ramps reveals a dependence on the rate at which the unsteady flow is driven consistent with that observed during the rheological measurements in the presence of slip. The dependence of the slip velocity on the wall shear stresses reveals a Navier-type slip behaviour only within the fluid flow regime, which indicates that the wall slip phenomenon is directly coupled to the solid-fluid transition.*

**Mots clefs :** yielding ; hysteresis ; wall slip

## 1 Introduction

From a fundamental perspective and even in the case of minimal thixotropic effects, yield stress materials continue triggering intensive debates and posing difficult challenges to both theoreticians and experimentalists. From a historical perspective, the best known discussion is related to the very existence of the yield stress [1]. It was thus suggested that the yield stress is a *engineering* reality rather than a physical one or, in other words, the apparent yield stress behaviour observed during rheological tests is related to a very large Newtonian viscosity rather than to the onset of flow (yielding). In context of the recent developments of the rheometric equipment (now able to resolve torques as small as  $0.1nNm$  and rates of shear as small as  $10^{-7} s^{-1}$ ), a further pursue of this already classical debate on the existence of a yield stress is, in our opinion, trite. Indeed, recent rheological tests performed on various Carbopol<sup>®</sup> gels proved unequivocally the existence of a true yielding behaviour, [8, 3]. The existence of a Hookean solid deformation regime preceding the solid-fluid transition has been experimentally demonstrated in Ref. [8] in the form of a plateau of the rate of deformation measured during increasing/decreasing linear stress ramps.

Whereas there exists a significant number of studies of the yielding of Carbopol<sup>®</sup> gels in rheometric flows, the body of literature concerning experimental studies of the solid-fluid transition of Carbopol<sup>®</sup> gels in industrially relevant flows is, to our best knowledge, more limited. A experimental study of the yielding and flow properties of a 0.2% Carbopol<sup>®</sup> in a capillary tube has been performed by González and his coworkers, [6]. By combining classical rotational rheometry techniques with Digital Particle Image Velocimetry (**DPIV**) measurements of steady flows driven at various pressure drops the authors of this study conclude that a yield stress behaviour does exist. As the driving pressure drop is gradually increased, they observe a smooth solid-fluid transition. To overcome the difficulty of accurately measuring the yield stress triggered by the smoothness of the solid-fluid transition, the authors of the study propose an alternative method of measuring this parameter based on the analysis of the measured velocity profiles, [6]. As the flows studied in Ref. [6] were stationary, these experimental results can neither confirm nor contradict the main features of the solid-fluid transition observed during unsteady rheometric flows in Refs. [8, 9] which motivated us to consider the unsteady pipe flow case in the present study.

The body of existing literature on unsteady pipe flows of Carbopol<sup>®</sup> gels is significantly more limited. Park and his coworkers have studied both experimentally and theoretically oscillatory flows of a 0.075% Carbopol<sup>®</sup> solution in a acrylic made U-shaped tube with a circular cross section, [5]. Consistently with the findings reported in Ref. [8], the authors of this study have provided a clear experimental evidence on the significant role of elasticity on the measured unsteady velocity profiles particularly near the centreline of the flow channel. Due to the elastic effects, strongly nonlinear time histories of the strain are measured. To model their experimental findings, Park and his coworkers propose an elasto-viscoplastic model which fits their data better than the regularised Bingham model. Some discrepancies between theory and experiment still exist and may be due to the fact that their model does not account for the velocity slip near the wall.

The central aim of the present study is to present a detailed experimental characterisation of unsteady flows of a Carbopol<sup>®</sup> gel in the presence of wall slip in both a rheometric flow and a pipe flow. The general question addressed through our study concerns with the dynamics of the solid-fluid transition observed during the unsteady low Reynolds number pipe flow of a Carbopol<sup>®</sup> gel and its connection to both the wall slip phenomenon and the solid-fluid transition previously observed in rheometric flows (with and without slip at the boundaries). The paper closes with a brief discussion of our main findings and their possible impact onto the current understanding of flows of viscoplastic fluids.

## 2 Experimental setup and methods

The experimental setup is schematically illustrated in Fig. 1. It consists of a flow channel **FC** of length  $L = 115\text{ cm}$  with a circular cross-section of inner radius  $R = 1.9\text{ mm}$ . The flow channel is illuminated laterally by an horizontal laser sheet and visualised from the top with a digital camera. Sequences of flow images are passed through a digital particle image velocimetry set of routines implemented

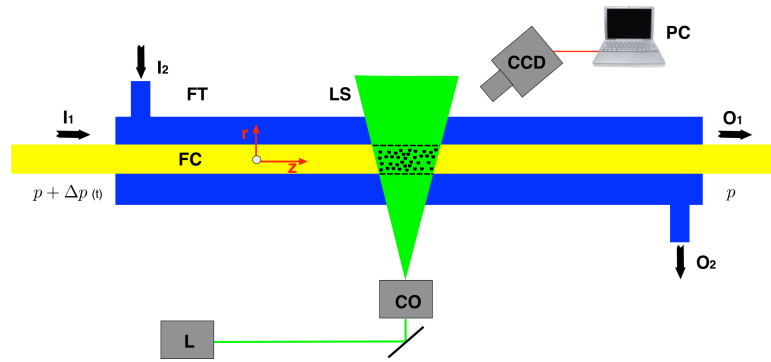


FIGURE 1 – Schematic view of the experimental apparatus : **FT** - fish tank, **I<sub>2</sub>** - water inlet, **O<sub>2</sub>** - water outlet, **FC** - flow channel, **I<sub>1</sub>** - working fluid inlet, **O<sub>1</sub>** - working fluid outlet, **L** - solid state laser, **CO** - cylindrical optics block, **LS** - laser sheet, **CCD** - digital camera, **PC** - personal computer.

in the house which allows one to measure sequences of velocity fields. A 0.08% (by weight) solution of Carbopol<sup>®</sup> 980 has been used as a working fluid. A detailed description of the procedure for the preparation of the solution is given in Ref. [8]. The pipe flow has been driven by controlling the pressure drop  $\Delta p$  between the inlet and the outlet of the channel. To test the reversibility of the flow states linear (in time) increasing/decreasing controlled pressure ramps have performed. The time per each constant pressure step  $t_0$  (to be further referred to as the characteristic forcing time) has been varied in order to probe the sensitivity of the flow states to the steadiness of the forcing.

### 3 Results

To characterise the rheological behaviour of the Carbopol<sup>®</sup> solution increasing/decreasing controlled stress linear ramps have been performed. The stress has been maintained constant for each step of the ramp during a time  $t_0$  which is the rheometric equivalent of the characteristic forcing time at which the pipe flow was driven. The results of such measurements are presented in Fig. 2(a). Below a critical rate of shear  $\dot{\gamma}_c \approx 0.4 \text{ s}^{-1}$ , the presence of slip induces an under-estimation of the measured stresses and, consequently of the viscosity. Beyond this critical shear rate the data acquired with and without slip overlap.

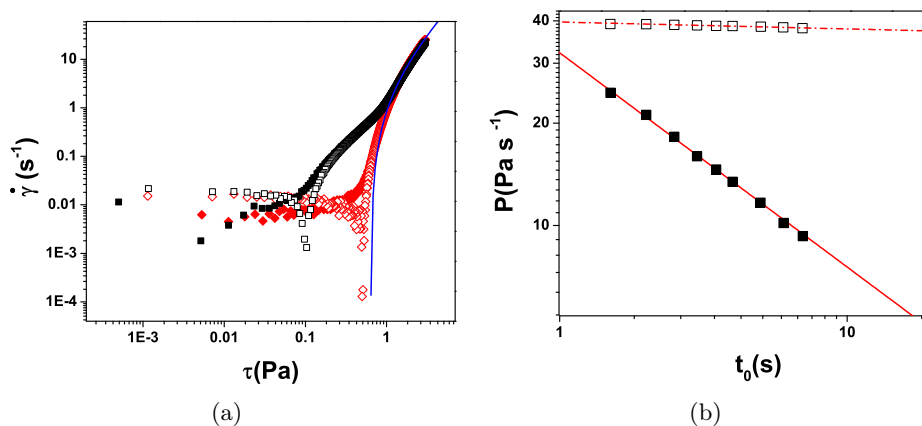


FIGURE 2 – (a) Flow curves measured with a polished geometry ( $\square$ ,  $\blacksquare$ ) and with a rough geometry ( $\diamond$ ,  $\blacklozenge$ ). The full/empty symbols refer to the increasing/decreasing branch of the flow curves. The full line is a Herschel-Bulkley fit that gives  $\tau_y = 0.64 \pm 0.003 \text{ (Pa)}$ ,  $K = 0.4 \pm 0.002 \text{ (Pa s}^n\text{)}$ ,  $n = 0.54 \pm 0.001$ . (b) Dependence of the hysteresis area on the characteristic forcing time  $t_0$  measured with a smooth geometry ( $\square$ ) and a rough one ( $\blacksquare$ ). The full line (—) is a guide for the eye,  $P \propto t_0^{-0.63}$  and the dash dotted line (-.-) is a guide for the eye,  $P \propto t_0^{-0.03}$ .

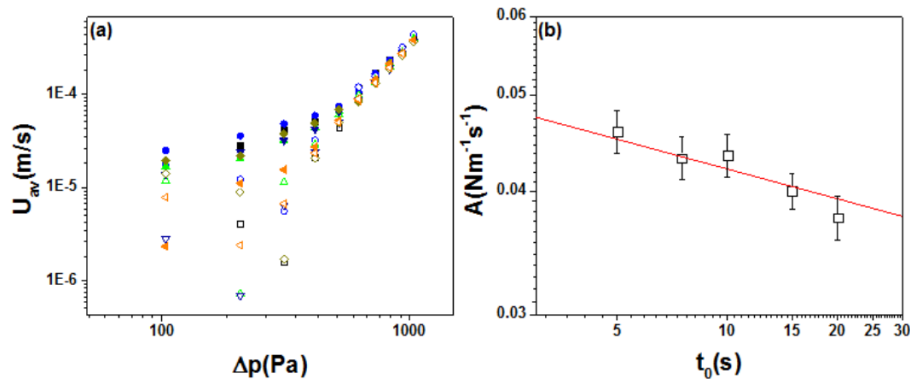


FIGURE 3 – (a) Dependence of the absolute value of the mean flow velocity  $U_{av}$  on the applied pressure drop  $\Delta p$  for various values of the characteristic forcing time : squares -  $t_0 = 4s$ , circles -  $t_0 = 5s$ , up triangles -  $t_0 = 7.5s$ , down triangles -  $t_0 = 10s$ , rhombs -  $t_0 = 15s$ , left triangles  $t_0 = 20s$ . The full/empty symbols refer to the increasing/decreasing branch of the pressure ramp. (b) Dependence of the area of the hysteresis of the dependencies of the mean flow velocity on the driving pressure drop presented in panel (a) on the characteristic forcing time  $t_0$ . The full line is a guide for the eye,  $A \propto t_0^{-0.1}$ .

It is of utmost importance to point out that, unlike predicted within the classical pictures of yielding (Bingham, Herschel-Bulkley etc.) the solid - fluid transition is not direct, i.e. does not occur corresponding to a well and uniquely defined value of the applied stress. Thus, as the applied stresses are gradually increased, three deformation regimes can be observed : solid ( $\dot{\gamma} \approx ct.$ ), fluid ( $\dot{\gamma} > \dot{\gamma}_c$ ) and an intermediate solid-fluid coexistence regime. Within the fluid (fully yielded) regime the data can be well fitted by the Herschel-Bulkley model (the full line in Fig. 2(a)). Thus, consistently with the results previously reported in Ref. [8], the yielding process of the Carbopol<sup>®</sup> is not reversible upon increasing/decreasing applied stresses as (nearly) universally believed. It is also important to note at this point that the yielding transition emerges within the same range of applied stresses where the wall slip phenomenon manifests itself so the two phenomena are intrinsically coupled. The dependence of the area of the hysteresis associated to the irreversibility of the flow curves presented in Fig. 2(a) on the characteristic forcing time  $t_0$  is presented in Fig. 2(b). The important conclusion of these measurements is that the presence of wall slip nearly suppresses the scaling of the power losses associated with the hysteresis with the degree of flow steadiness. A natural question to address at this point is whether the main features of the yielding transition observed in a rheometric flow would still be observed in an industrially relevant flow such as an unsteady laminar pipe flow.

Measurements of the average (during the characteristic forcing time  $t_0$  and across the flow direction) flow speed are presented in Fig. 3 for several values of the characteristic forcing time indicated in the figure caption. As in the case of the rheological measurements illustrated in Fig. 2(a), the dependence of the mean flow speed  $U_{av}$  on the driving pressure drop  $\Delta p$  is not fully reversible upon increasing/decreasing  $\Delta p$  unless within a range of large pressures where the material is fully yielded. Thus, these measurements confirm once again that the yielding process occurs within a band of externally applied forcing rather than a given value associated to yield stress. The consistency between the assessment of the yielding process in a rheometric configuration and in a non-rheometric one should, in our opinion, pass as a reason to reconsider picture of Carbopol<sup>®</sup> gels as "ideal yield stress fluids" in future studies. Measurements of the dependence of the slip velocity  $U_s$  on the velocity gradient measured at the pipe's wall for several values of the characteristic forcing time  $t_0$  are presented in Fig. 4(a). Quite remarkably, regardless the value of the characteristic forcing time  $t_0$ , the data overlap indicating a universal feature of the slip behaviour and the slip velocity scales linearly with the wall velocity gradients. By interpreting the velocity gradients near the wall as an effective rate of shear, one can correlate these measurements to the rheological flow curves (see Fig. 2(a)) and produce the dependence of the slip velocity on the wall shear stress  $\tau_w$ , Fig. 4(b). Within this representation the

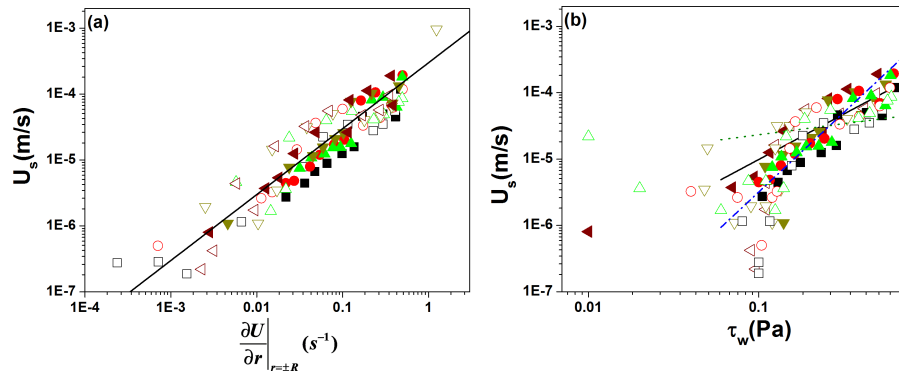


FIGURE 4 – (a) Dependence of the slip velocity  $U_s$  on the wall velocity gradients. The full line is a guide for the eye,  $U_s \approx 3 \cdot 10^{-4} \frac{\partial U}{\partial r} \Big|_{r=\pm R}$ . (b) Dependence of the slip velocity  $U_s$  on the wall shear stresses  $\tau_w$ . The full line is a power law fit,  $U_s = 2.3 \cdot 10^{-4} (\pm 5.4 \cdot 10^{-5}) \cdot \tau_w^{1.32 \pm 0.26}$ . The dotted and the dash-dotted lines are the theoretical predictions by Piau (see Ref. [7]) :  $U_s \propto \tau_w^{1/3}$ ,  $U_s \propto \tau_w^2$ . In both panels the symbols are :  $(\triangle, \blacktriangle)$  -  $t_0 = 4$  (s),  $(\circ, \bullet)$  -  $t_0 = 7.5$  (s),  $(\square, \blacksquare)$  -  $t_0 = 10$  (s),  $(\nabla, \blacktriangledown)$  -  $t_0 = 15$  (s),  $(\triangleleft, \blacktriangleleft)$  -  $t_0 = 20$  (s).

universality of the slip velocity data with respect to the degree of flow steadiness  $t_0$  is preserved only within the fluid regime. The scaling properties within this regime equally deserve a brief discussion.

Using a Hertzian framework to model the contacts between neighbouring micro-gel elastic particles (a detailed description of the theoretical framework can be found in Ref. [4]), Piau predicts two scaling laws of the slip velocity with the wall shear stress depending on the packing of the soft particles :  $U_s \propto \tau_w^2$  for a closely packed system and  $U_s \propto \tau_w^{1/3}$  in the case of a loosely packed system. The scaling exponent obtained by fitting the data presented in Fig. 4 corresponding to the fully yielded regime differs from these theoretical predictions (see the dotted and the dash-dotted lines in Fig. 4 (b)).

A possible reason of this discrepancy might originate in the fact that the interstitial lubricating fluid considered in Ref. [7] was non-Newtonian and, to get a deeper insight into the slip behaviour of Carbopol<sup>®</sup> gels a additional discussion of this issue is in order. A physical rationale for the choice of a non-Newtonian (shear thinning) interstitial fluid in Ref. [7] might be to decouple at a micro scale the shear thinning behaviour (by attributing it to the interstitial fluid) from the yield stress behaviour by implicitly attributing it to the elastic-solid Carbopol<sup>®</sup> micro particles.

The disagreement we pointed out above seems to indicate that this decoupling is not as straightforward and should be more cautiously considered in future attempts to theoretically describe the relation between flow properties, wall slip and microstructure. Future theoretical approaches should definitely reconsider this problem.

## 4 Conclusions, outlook

An experimental investigation of unsteady pipe flow of a Carbopol<sup>®</sup> solution is presented. Depending on the value of the applied pressure several flow regimes are observed.

Corresponding to the lowest values of the driving pressure  $\Delta p$  a solid like flow regime is observed. This regime is characterised by a an elastic solid plug which never reaches a steady flow state. The largest level of fluctuations of the solid plug is observed near the centre line of the flow channel which we interpret as the signature of an unsteady solid (elastic) plug.

For the largest values of the driving pressure  $\Delta p$  a fluid regime is observed. Within this regime the velocity reaches a steady state and the flow is reversible upon increasing/decreasing pressures. As in the case of the rheometric flow initially studied in Ref. [8], the transition between the solid-like regime to the fully yielded regime is not direct but mediated by an intermediate solid-fluid coexistence regime.

For each value of the characteristic forcing time  $t_0$  we have explored, this intermediate phase coexistence regime manifests itself by a clear hysteresis (observed upon increasing/decreasing the driving pressure drop) which is qualitatively similar to the hysteresis observed in the rheological flow curves. The experimentally observed hysteresis of the flow curves is associated with a deformation power deficit and measurements of this quantity for various values of  $t_0$  reveal no sensitive dependence on the frequency of forcing,  $A \propto t_0^{-0.1}$ . This fact is fully consistent with the measurements of the deformation power deficit performed in a rheometric (plate-plate) setup in the presence of slip,  $P \propto t_0^{-0.03}$ , Fig. 2(b). As a conclusion for this part, the main features of the solid-fluid transition observed in a rheometric flow in the presence of wall slip are equally observed in a unsteady pipe flow. In spite of its ubiquitous nature and tremendous importance, the phenomenon of wall slip in pasty materials and dispersions is still under-looked by many researchers, [2]. As Carbopol<sup>®</sup> gels are concerned, a detail characterisation of their slip properties in slow pipe flows was, to our best knowledge, insufficiently documented. The time and space resolved measurements of the flow fields based on the **DPIV** technique allowed us to attempt to fill this gap and provide a detailed experimental characterisation of the wall slip phenomenon. Systematic measurements of the slip velocity during controlled pressure ramps with various degrees of steadiness (i.e. for several values of the characterising forcing time  $t_0$ ) reveal a clear coupling between the irreversible solid-fluid transition and the slip phenomenon. A linear scaling of the slip velocity with the wall velocity gradients is found, Fig. 4(a). This scaling law is universal in the sense that it does not depend on the flow steadiness and invariantly holds for both the increasing and the decreasing branches of the controlled pressure ramp. A clear picture of the coupling between the irreversible solid-fluid transition and the slip phenomenon can be obtained by plotting the slip velocity against the wall shear stress, Fig. 4(b). It is only within the fully yielded regime that a Navier-type nearly linear scaling of the slip velocity can be observed. The measurement of the slip coefficient allowed one to estimate the thickness of the lubrication layer responsible for the slip behaviour. The result of this estimate seems to be consistent with previous experimental findings. It is equally interesting to note that, within the fully yielded regime, the scaling of the slip velocity is practically independent on the flow steadiness (described by the value of the characteristic forcing time  $t_0$ ) and whether the driving pressure is increasing or decreasing.

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