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Antoine Soulies, Jeremy Pruvost, Jack Legrand, Cathy Castelain, Teodor Burghilea. Propriétés rhéologiques et écoulement dans des microcanaux de suspensions de microalgues. A detailed characterization of plane micro-channel flows of Chlorella suspensions with various volume fractions in a wide range of driving pressures drops reveals flow. CFM 2013 - 21ème Congrès Français de Mécanique, Aug 2013, Bordeaux, France. hal-03440454

HAL Id: hal-03440454

<https://hal.science/hal-03440454>

Submitted on 22 Nov 2021

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Rheological properties and microscopic flows of suspensions of *Chlorella vulgaris* microalgae

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Résumé :

*Une étude complète des propriétés rhéologiques de suspensions de microalgues non motiles (*Chlorella vulgaris* CCAP 211-19) est présentée pour une large gamme de fraction volumique. Avec l'augmentation progressive de la fraction volumique, plusieurs régimes rhéologiques sont observés. A faible fraction volumique, la suspension montre un comportement rhéologique newtonien. Pour les valeurs intermédiaires des fractions volumiques, un comportement rhéofluidifiant est constaté. L'étude des fractions volumiques les plus hautes montre clairement un comportement viscoélastique.*

La montée et la descente des rampes de contraintes pour cette gamme de fraction volumique mettent en évidence aussi un comportement thixotrope. Afin d'avoir une idée plus précise sur l'origine des régimes rhéofluidifiant et viscoélastique, l'organisation microscopique de cellules individuelles est observée simultanément avec les mesures rhéologiques macroscopiques. Ainsi, l'apparition du comportement rhéofluidifiant est expliquée par la présence de floes de cellules seules et le comportement viscoélastique par la formation d'agrégats de cellules à large échelle qui se comportent comme des bouchons rigides.

*Une caractérisation détaillée de l'écoulement en micro-canaux rectangulaire de suspensions de *Chlorella*, à diverses fractions volumiques, dans une large gamme de pression différentielle contrôlée, révèle un type d'écoulement cohérent avec les régimes rhéologiques observés pendant les mesures rhéologiques macroscopiques.*

Abstract :

*A systematic study of the rheological properties of solutions of non-motile microalgae (*Chlorella vulgaris* CCAP 211-19) in a wide range of volume fractions is presented. As the volume fraction is gradually increased, several rheological regimes are observed. At low volume fraction the suspension display a Newtonian rheological behaviour. For intermediate values of volume fraction a shear thinning behaviour is observed. For the largest values of the volume fraction investigated, an apparent yield stress behaviour is observed.*

Increasing and decreasing stress ramps within this range of volume fraction indicate a thixotropic behaviour as well. To gain a deeper insight into the emergence of the shear thinning and yield stress regimes, the microscopic organisation of individual cells is observed simultaneously with the macroscopic rheological measurements. Thus, the emergence of the shear thinning behaviour is explained by the presence of floes of individual cells and the yield stress behaviour is explained by the formation of large scale cell aggregates which behave like rigid plugs.

*A detailed characterisation of plane micro-channel flows of *Chlorella* suspensions with various volume fractions in a wide range of driving pressures drops reveals flow patterns consistent with the rheological regimes observed during the macroscopic rheological measurements.*

Mots clefs : microalgae ; rheology ; microfluidics

1 Introduction

Microalgae are aquatic photosynthetic unicellular organisms. Each species of micro algae has a specific composition in terms of proteins, lipids and carbohydrates, which explains their large number of applications in food industry, green chemistry, bio fuels and more, [1]. A practical challenge is related to the efficient growth of micro algae under controlled conditions (light, nutrient, pH , temperature) and at industrially relevant scales. Photobioreactor (PBR) technology allows one to significantly increase the efficiency of microalgal production up to biomass concentrations of 10 kg m^{-3} . The modern photobioreactor technology aims increasing the efficiency of the microorganism's growth without necessarily increasing the ratio between the effected illuminated culture area and its volume. This can be typically achieved by generating a controlled flow within the culture which, in turn, requires a good understanding of both the hydrodynamics and rheological properties of solutions of microalgae particularly in a regime of high volume fractions of cells.

The present study is concerned with an experimental study of the rheological and microscopic flow properties of suspensions of the non-motile *Chlorella* microalga in a wide range of volume fractions.

2 Experiment

Artificially concentrated suspensions of microalgae (*Chlorella vulgaris* CCAP 211-19) were used through our study. Microalgae were grown under controlled conditions (nutrient concentration, light, pH and temperature).

The macroscopic rheological measurements have been performed on a Haake Mars *III* rotational rheometer equipped with a nano-torque module and a Rheoscope module that allows the simultaneous microscopic observation of the suspension under shear, Fig. 1 (a).

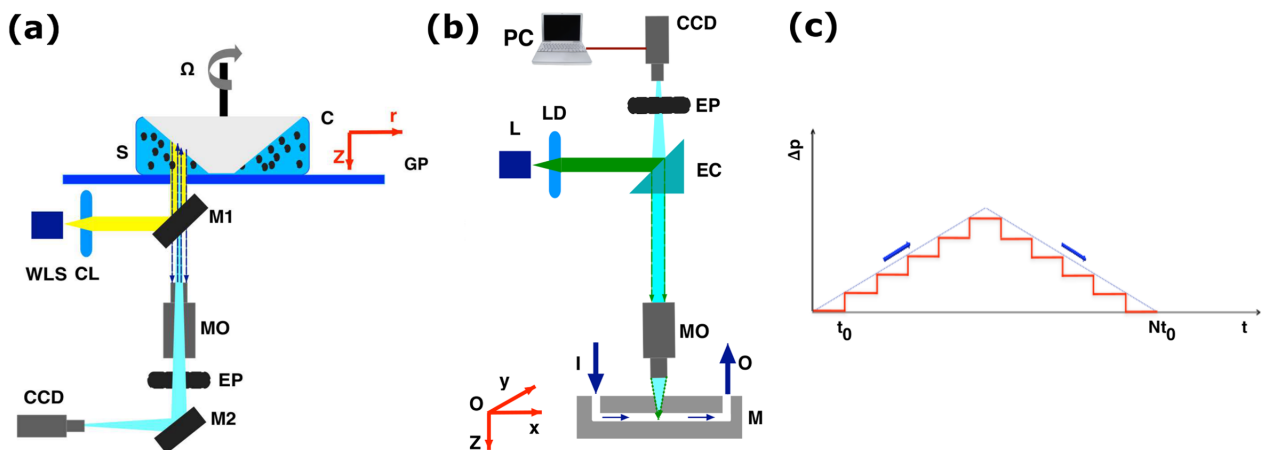


FIGURE 1 – (a) Schematic view of the Rheoscope module : **CCD** - digital camera, M_1 , M_2 - mirrors, **EP** - eye piece, **MO** - microscope objective, **WLS** - white light source, **CL** - collimating lens, **GP** - glass plate, **S** - *Chlorella* sample, **GP** - glass plate, **C** - cone. (b) Schematic view of the microfluidic setup : **M** - microchannel, **I,O** - channel's inlet/outlet **EC** - epifluorescent cube, **LD** - light diffuser, **L** - LED panel, **EP** - eye piece. (c) Schematic representation of the hydrostatic pressure ramp. t_0 stands for the characteristic forcing time (the averaging time per stress point) and N is the total number of steps.

The rheological behaviour of the microalgae suspension has been assessed by controlled stress flow ramps schematically illustrated in Fig. 1 (c). To probe the reversibility of the deformation states, both increasing and decreasing stress ramps have been performed. The second part of our study concerns with the study of flows of *Chlorella* suspensions in a rectangular micro-channel. As illustrated in Fig. 1 (b), a micro-channel (section of $150 \mu\text{m} * 50 \mu\text{m}$) has been placed under a epifluorescent microscope. The flow was seeded with fluorescent latex spheres which allowed measurements of time series of velocity fields via a standard micro-particle image velocimetry technique.

A more detailed description of the experimental system and methods is given in Ref. [3].

3 Results

3.1 Macro-rheological properties of *Chlorella* suspensions

Viscosity measurements of suspensions of *Chlorella* with various volume fractions are presented in Fig. 2(a). Within the dilute regime¹ ($\Phi_v \leq 0.115$) no stress dependence of the viscosity is observed (the down-triangles in Fig. 2(a), (∇)) indicating a Newtonian (**N**) rheological behaviour.

As the volume fraction is increased, an entirely different rheological behaviour is observed (the rhumbs (\diamond) in Fig. 2(a)). Corresponding to stress values smaller than a critical value ($\tau \leq \tau_0$) a Newtonian behaviour characterised by a viscosity plateau which defines the zero shear viscosity η_0 is observed. Beyond this critical stress value ($\tau > \tau_0$) a shear thinning rheological behaviour **ST** is observed, $\eta \propto K\dot{\gamma}^{n-1}$ where K , n stand for the consistency and the power law index, respectively. A second viscosity plateau which defines the infinite shear viscosity η_{inf} is observed at large applied stresses. The viscosity data acquired within this regime may be well described by the Cross model, [4].

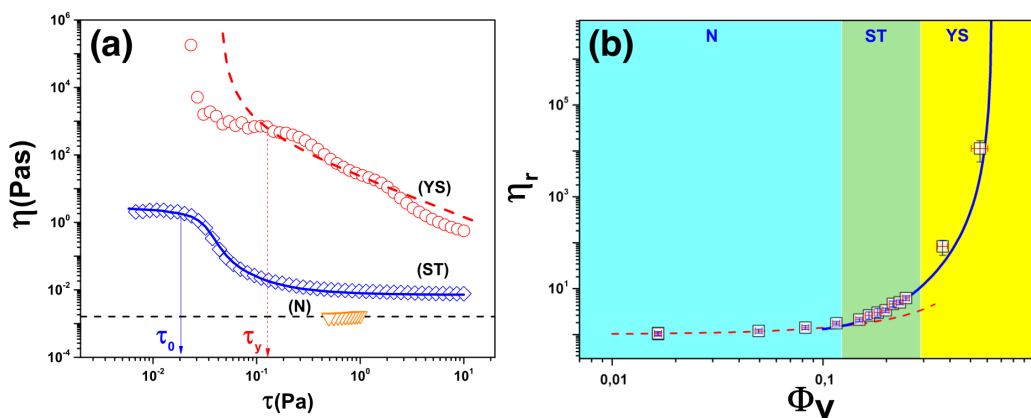


FIGURE 2 – (a) Stress dependence of the viscosity of the microalgal suspension measured in a controlled stress mode for several volume fractions : (∇) - $\Phi_v = 0.082$, (\diamond) - $\Phi_v = 0.165$, (\circ) - $\Phi_v = 0.556$. For each stress value the viscosity data has been averaged for a time $t_0 = 15$ s. The full line (—) is a fit according to the Cross model, the dashed line (---) is a fit according to the Herschel-Bulkley model. (b) Dependence of the relative viscosity of the microalgal suspension η_r on the volume fraction of the microalgae Φ_v . The dashed line is a fit according to the Quemada model [5] and the full line is a fit according to the Simha model, [6]. The labels indicate the experimentally observed rheological regimes : **N** - Newtonian, **ST** - shear thinning, **YS** - yield stress. Within the **ST** and **YS** regimes, the relative viscosity has been measured at a fixed stress within the reversible fluid regime, $\tau = 1$ Pa.

The dependence of the relative effective viscosity η_r of *Chlorella* microalgae suspensions measured at a constant applied stress $\tau = 1$ Pa (which corresponds to the reversible fluid regime) on the volume fraction Φ_v is presented Fig. 2 (b). Within the Newtonian volume fraction regime observed for dilute *Chlorella* suspensions (**N**) the volume fraction ($\Phi_v \leq 0.115$) dependence of the relative viscosity η_r is often modelled by the Quemada model, [5] (the dotted line in Fig. 2 (b)). For high volume fractions ($\Phi_v \geq 0.115$), the Simha's cellular model [6] can reliably describe the rheological behaviour of the highly concentrated suspensions (the full line in Fig. 2 (b)). As already illustrated and discussed in Fig. 2, within the highly concentrated regime **YS** a viscosity divergence is observed in a range of small stresses on both the increasing branch of the stress ramp (the squares (\square) in Fig. 3) and the decreasing one (the circles (\circ) in Fig. 3). The viscosity divergence can be understood in terms of a jammed particle network extended over the entire sample. The cell network responsible for the yield stress behaviour depicted in Fig. 3 breaks down into a suspension of flocs of cells (mesoscopic cell

1. We note that this regime is yet beyond the Einstein's ultra-dilute limit which was of no particular interest to the present study.

structure). Within this intermediate state, a viscosity plateau is observed, similar to the low stress Newtonian plateau observed at intermediate volume fractions within the **ST** rheological regime. As the applied stress is increased even further, the flocs of cells break down and a shear thinning fluid state is observed. Upon a decrease of the applied stresses (the "down" branch of the stress ramp) the

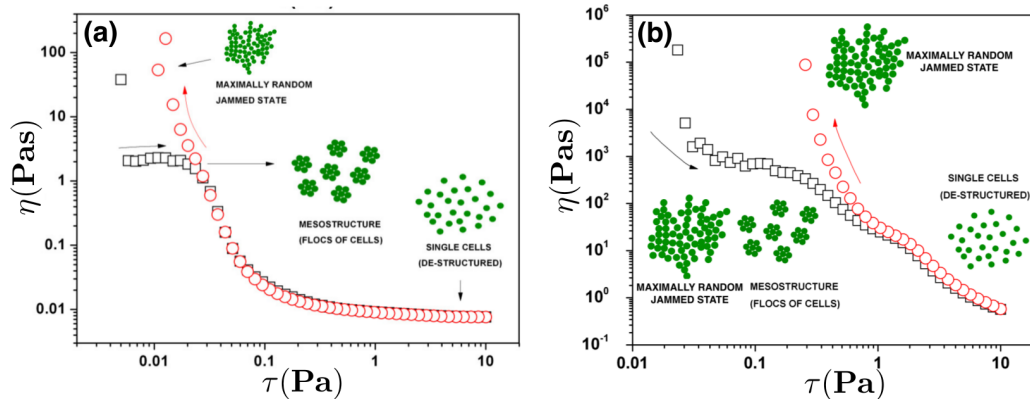


FIGURE 3 – Viscosity measurements within : (a) the **ST** regime ($\Phi_V = 0.148$) (b) the **YS** regime ($\Phi_V = 0.556$) for both increasing (\square) and decreasing (\circ) values of the applied stresses. The characteristic forcing time was $t_0 = 15$ s.

deformation states are recovered only within the fluid regime. We note that no intermediate viscosity plateau is observed on the decreasing stress branch : the system passes directly from a de-structured state to a cell network state characterised by a divergent viscosity.

Individual inspection of various increasing/decreasing flow ramps measured within both the **ST** and **YS** regimes indicates that there exists an onset of irreversibility of the deformation states which, most probably, corresponds to the complete breakdown of cell structures (flocs). Unfortunately, for the case of *Chlorella* microalgae, we are not aware of a theoretical framework able to describe this by accounting not only for the hydrodynamic interactions but for the electrostatic repulsions as well.

The irreversibility of deformation states is usually associated with thixotropic or time dependent effects in the rheological behaviour, [7]. To gain a deeper insight into the behaviour highlighted above, we have

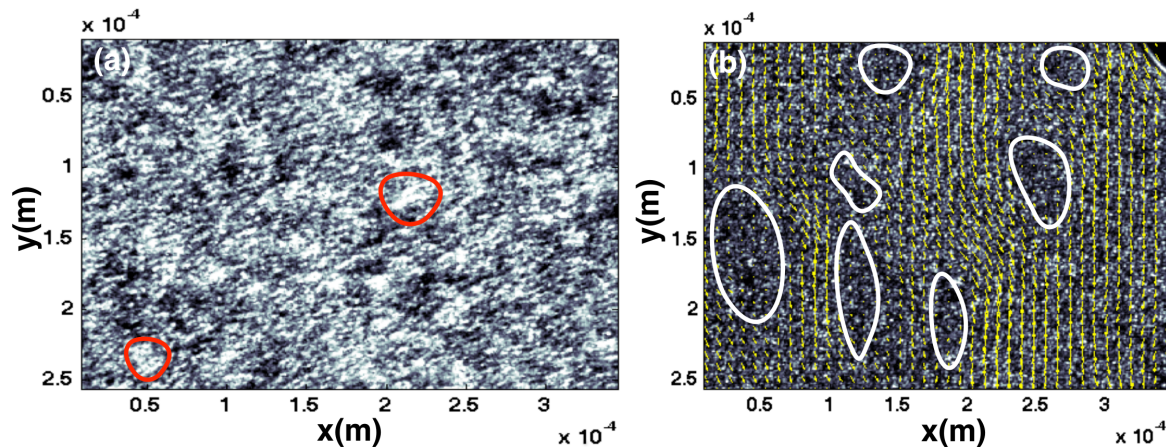


FIGURE 4 – Micrograph of a *Chlorella* suspension with a volume fraction, a), $\Phi_V = 0.15$ and, b), $\Phi_V = 0.556$ at a stress $\tau = 0.02$ Pa. The closed curves overlapped to each panel highlight the solid-like agglomerations of cells.

resorted to the in-situ microscopic visualisation of the rheometric flow using the Rheoscope module. A flow micrograph acquired within the **ST** regime at low applied stresses is presented in Fig. 4 (a). According to this image, the low shear viscosity plateau visible in Figs. 2(a), 3 can be associated to the presence of flocs of individual cells (see the highlighted regions). A typical flow micrograph together with a velocity field acquired within the **YS** regime is presented in Fig. 4 (b). Note in this plot the smallness of the flow speeds (typically smaller $1\mu\text{m/s}$) which is consistent with the extremely large

values of the measured viscosity (the circles (\circ) in Fig. 2). The viscosity divergence observed during the rheological measurements in a range of low applied stresses can be explained by the formation of large scale cell aggregates which act as rigid plugs (see the highlighted regions in Fig. 4 (b)).

It is important to point out the rheological flows are not relevant with the flows met in a practical case (photobioreactors) in terms of applied stress and velocity. In connection with the rheological behaviour studied in this part, we focus in the next section on micro channel flows of *Chlorella* suspensions at various volume fractions. For a more detailed discussion of the rheological regimes briefly discussed through this section the reader is referred to Ref. [3].

3.2 Plane micro-channel flow of *Chlorella Vulgaris* suspensions

In the context of intensified photobioreactors, the rheometric flows such as the ones studied in the first part of the paper are perhaps not the most representative from a practical point of view. For this reason, we focus in the following on the study of plane micro channel flows of *Chlorella* suspensions. As already described in Sec. 2 and illustrated in Fig. 1 (b), the flow of *Chlorella* suspensions in a glass micro-channel with a rectangular cross section has been visualised under an upright microscope. As in the case of the rheoscope visualisation during the rheological measurements, the acquisition of an image sequence allowed one to measure the velocity fields using the digital particle image velocimetry (DPIV) technique. Such measurements were performed for three different concentrations of microalgae corresponding to each of the rheological regimes discussed above and within a wide range of driving pressure drops.

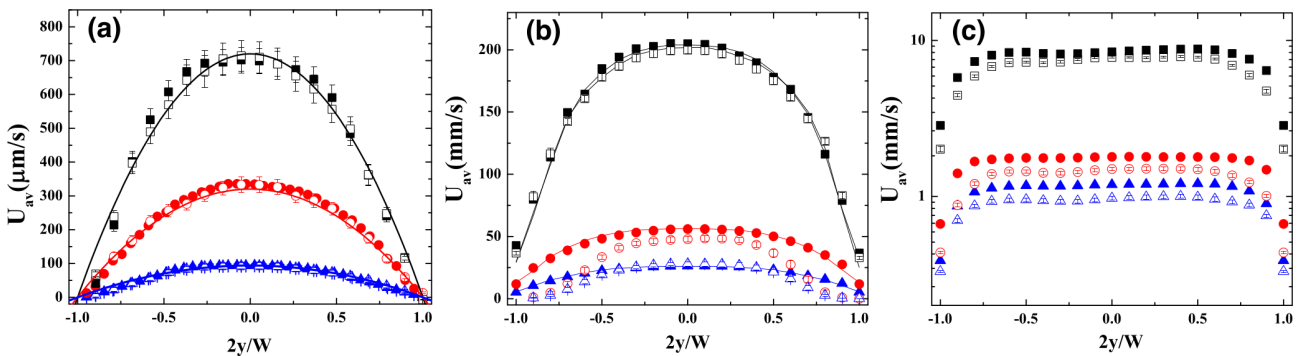


FIGURE 5 – Velocity profiles measured for various volume fractions Φ_v and at various driving pressure drops Δp : (a) $\Phi_v =$ and $\Delta p = 8.3 Pa$ ($\blacktriangle, \triangle$), $\Delta p = 25 Pa$ (\bullet, \circ), $\Delta p = 50 Pa$ (\blacksquare, \square). (b) $\Phi_v =$ and $\Delta p = 10 Pa$ ($\blacktriangle, \triangle$), $\Delta p = 16.7 Pa$ (\bullet, \circ), $\Delta p = 46.7 Pa$ (\blacksquare, \square). (c) $\Phi_v =$ and $\Delta p = 25 Pa$ ($\blacktriangle, \triangle$), $\Delta p = 41.7 Pa$ (\bullet, \circ), $\Delta p = 116.8 Pa$ (\blacksquare, \square).

In Fig. 5 we present transversal velocity profiles measured with suspensions of various volume fractions and for three values of the driving pressure drop Δp . Fig. 5 (a) illustrates a Poiseuille flow for each driving pressure drop investigated. The flow is reversible upon increasing/decreasing (full/empty symbols) pressure drops. This flow behaviour can be clearly identified as Newtonian. Also, at this volume fraction, no wall slip phenomenon is noted. The rest of the panels correspond to the shear thinning (ST) regime (Fig. 5 (b)) and the yield stress (YS) regimes (Fig. 5 (c)). As the volume fraction is increased beyond the Newtonian regime, the wall slip phenomenon can be observed within both the ST and the YS regimes. Within both volume fraction regimes, at low values of the driving pressures a clear and systematic difference between the transversal velocity profiles measured on the up/down branches of the ramp. This is observed which indicates that the flow is irreversible. The flow irreversibility (hysteresis) can equally be observed in the Fig. 6 which presents the dependence of the averaged (across the microchannel) velocity versus the driving pressure drop Δp . Within the Newtonian regime, the dependence is linear as one would expect for a Poiseuille flow (the full line in Fig. 6). For the shear thinning and the yield Stress regimes, the microscopic flow irreversibility is consistent with the observed rheological hysteresis. The main features of the plane micro channel

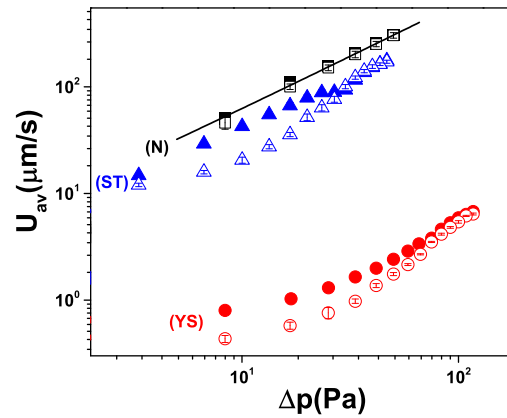


FIGURE 6 – The dependence of the average flow speed U_{av} on the driving pressure drop Δp measured for *Chlorella* suspensions at various volume fractions : (■, □) - $\Phi_v = 0.02$, (▲, △) - $\Phi_v = 0.12$, (●, ○) - $\Phi_v = 0.57$. The labels (N), (ST), (YS) stand for Newtonian, shear thinning and yield stress, respectively. The full/empty symbols distinguish between the data acquired on the increasing/decreasing branch of the stepped pressure ramp, respectively. The full line is a linear fit.

flows of the *Chlorella* suspensions at various volume fractions are fully consistent with the rheological measurement presented in Sec. 3.1.

4 Conclusions

A systematic study of the rheological properties and microscopic flows of suspensions of *Chlorella* microalgae within a wide range of volume fractions is presented. Macroscopic rheological measurements reveal three distinct rheological regimes. At low volume fractions a Newtonian regime is observed. Upon an increase of the volume fraction a shear thinning regime develops. This regime characterised by a low and a high shear viscosity plateau originates in the microscopic scale aggregation of individual cells in flocs. Corresponding to largest volume fractions investigated an apparent yield stress regime is observed. Both the shear thinning and the yield stress regimes are irreversible upon increasing/decreasing stress ramp. This regime originates in a microscopic jamming of individual cells.

The macro-rheological study is complemented by investigations of plane micro-channel flows of suspensions at various volume fractions. Measurements of the transverse velocity profiles at various pressure drops and within a wide range of volume fractions are fully consistent with the rheological regimes identified by means of the macro-rheological techniques.

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