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Flow and blockage of highly concentrated granular suspensions in non-Newtonian fluid

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ABSTRACT

Studying the flow of highly concentrated granular suspensions represents a great challenge since they are characterized by a rather complex rheological behavior. In addition, macroscopic heterogeneities may be induced by the flow during rheological measurements due to the eventual relative motion between the liquid and the granular phases that may take place under certain conditions. Solid–liquid separation may ultimately lead to flow blockage. In the present investigation we consider experimentally the influence of the rheological properties of the suspending fluid on the transition between the flow and blockage of a concentrated suspension in a squeeze set-up geometry. The suspending fluid consists of an aqueous Xanthan solution for which rheological properties can be tuned by changing the polymer concentration. For each polymer concentration, it is shown that there exist flow parameters (squeeze velocity and gap thickness) for which one has a transition between homogeneous flow of the suspension and its blockage diagrams, delimiting flowability and blockage zones, are then determined. Physical arguments are given to relate the evolution of the blockage diagrams to the flow parameters and rheological properties of suspending fluid.

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1. Introduction

Some of industrial materials are composites made-up of a continuous phase (matrix) in which particles of different forms are dispersed in order to improve the effective properties of the matrix. This includes polymer and metallic short fiber composites, cementitious materials, etc. In order to take full advantage of the effect of the inclusions on the effective composite properties, such as mechanical, thermal, electrical properties, etc., the particles have to be homogeneously distributed throughout the matrix. During the forming process of the composite, flow-induced heterogeneous distribution of the inclusions may take place. Taking into account flow-induced heterogeneities when dealing with the problem of composite materials processing is then a crucial issue.

Several studies have been reported in the literature pointing out that concentrated suspensions or pastes (which represent an approximation of a real composite material in a fresh state) become heterogeneous in complex flows. For example, it is well known that non-uniform shear flows induce particle migrations

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in concentrated suspensions [1]. The origin of this phenomenon is well understood and attributed to irreversible interactions (collisions) among the particles [2,3]. In the present study flow-induced heterogeneities are considered from the point of view of continuum mechanics, regardless of the origin of the solid-liquid separation. We focus on the problem of flow-induced heterogeneities in the squeeze flow geometry, in which flowgradients are in any case heterogeneous. Squeeze flow fixtures are often used to determine rheological properties of highly viscous fluids, including cementitious materials [4,5], molten polymers [6], electrorheological suspensions [7], etc. Similarly to other types of rheometers, undesirable effects may appear to be complicating the interpretation of the rheological measurements. This is the case for example when we have presence of wall-slip. This issue has been extensively investigated in the literature [8-10]. In our experiments we used rough surfaces and assume that we do not have wall-slip.

More recently it has been shown that solid–liquid relative motion may take place in squeeze flow experiments further complicating the interpretation of rheological measurements. Moreover, the presence of solid–liquid relative motion may lead to the blockage of the flow. The problem of squeeze flow in the presence of solid–liquid relative motion has been considered theoretically by Sherwood [11,12]. There is also a number of experimental results reported in the literature, concerning different materials, dealing with this issue. Solid–liquid separation

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has been observed (or suspected) for example when considering the squeeze flow of ceramic pastes [13], sewage sludge [14], cellulose pastes [15], clay pastes [16] and model suspensions [17–19].

In the above studies, squeeze-flow-induced heterogeneities have been attributed to the filtration of the fluid phase through the porous media made up by the solid particles. In particular, this has been interpreted [13,14,17] in terms of the competition between the flow of the suspension as a whole and the Darcy filtration of the fluid phase. The two phenomena take place at different time scales depending upon the properties of the suspension, including its rheological properties, those of the fluid phase and the permeability of the granular skeleton. In the present study we focus on the influence of the rheology of the interstitial fluid on the transition between flow and blockage of a concentrated suspension.

2. Experimental

2.1. Experimental procedure and materials

2.1.1. Materials

The suspensions are composed of Polymethyl methacrylate (PMMA) spherical particles dispersed in aqueous solutions of Xanthan. The particle size distribution is approximately Gaussian with an average value of 0.2 mm. To vary the rheological properties of the suspending fluid, different Xanthan concentrations (by weight) were used: 1000, 3000, 5000 and 10 000 ppm.

The volume fraction of the solid particles in the suspensions (ϕ) was fixed to 0.5.

Since we are mainly interested in the phenomenon of *flow-induced* liquid-particulate separation, we have to check whether other sources of such a phenomenon may be present. This is the case of sedimentation that must be estimated. The average settling velocity of the suspension (V) may be estimated using for instance the Richardson–Zaki phenomenological equation [20]:

$$V = V_0 (1 - \phi)^{5.5} \tag{1}$$

where V_0 is the settling velocity of an isolated particle in the suspending fluid:

$$V_0 = \frac{2}{9} \frac{(\rho_s - \rho_f)ga^2}{\mu}$$
(2)

where ρ_s is the density of the particle (1160 kg/m³), ρ_f the density of the suspending fluid (1000 kg/m³), *a* the particle's radius (0.1 mm), *g* the acceleration of gravity and μ the viscosity of the suspending fluid (assumed to be Newtonian). For the less viscous suspending fluid considered in our study (1000 ppm), the estimation of the hindered settling velocity of the suspension gives: $V \approx 0.02$ mm/min, which is 5 times smaller than the smallest squeeze velocity used in our experiments (note that we used $\mu = A_f$, the consistency of the polymer solution, see below). Sedimentation effects can be then ignored.

2.1.2. Squeezing set-up

The suspensions were squeezed out between two parallel plates mounted on a compression-traction machine (from MTS). In order to minimize wall-slip, the plate surfaces were covered with a glass paper with a roughness of 0.2 mm (equal to the average size of the particles). Although wall-slip is an important parameter in squeeze flow experiments [8–10], we do not address this issue in the present study.

The upper plate was displaced at controlled velocities, while the lower one was maintained stationary. The normal force exerting



Fig. 1. Flow curves of the suspending fluid for different Xanthan concentrations. (●) 1000 ppm; (◇) 3000 ppm; (□) 5000 ppm; (◇) 10 000 ppm.

on the former was recorded as a function of time for each fixed velocity. In all the experiments, the maximum initial plate's separation was taken to be 6 mm, which was much smaller than the diameter of the smallest disc (40 mm), insuring lubrication-type flow conditions.

2.1.3. Rheological measurements

The rheological properties of the suspensions and the suspending fluids were determined using a stress-controlled shear rheometer (AR2000 from TA Instruments). The coaxial cylinder fixture was used in the case of the suspending fluid, while the vane geometry was preferred for the suspensions. In the later geometry, the tested material is not subjected to a uniform shear rate. This condition is usually required in rheological experiments in order to measure actual material properties, and to get a simple relationship between the measured torque/rotational velocity and the shear-stress/shear-rate. Vane geometry is nevertheless recognized to be appropriate for highly concentrated suspensions [21,22] since slippage can be avoided and the material is sheared in volume. The gap (distance between the periphery of the Vane and the outer cylinder) was 5.5 mm, which was more than an order of magnitude larger that the maximum size of the particles (about 0.35 mm). The shear-rate and the shear-stress were inferred from the torque and the rotational velocity of the vane by calibrating with a Newtonian fluid. The temperature was regulated at 25 °C (to within 0.1 °C) thanks to a circulating water system.

2.2. Experimental results

2.2.1. Rheological properties

Fig. 1 represents the flow curves of the suspending fluids (Xanthan solutions) for different polymer concentrations. As already reported in numerous publications [23–26] the rheological behavior of a Xanthan solution can be fairly well described using a power-law model (Oswald de Waele fluid). That is:

$$\sigma = A_f \dot{\gamma}^{m_f} \tag{3}$$

where σ is the shear-stress, $\dot{\gamma}$ the shear-rate, A_f the consistency and m_f the fluidity index. The rheological parameters of the polymer solutions, including the consistency and fluidity index, are reported in Table 1.

The polymer solutions are shear-thinning (fluidity index smaller than 1). The shear-thinning index decreases (the fluid

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