

Viscosity bifurcation in granular materials, foams, and emulsionsF. Da Cruz,¹ F. Chevoir,¹ Daniel Bonn,² and P. Coussot^{1,*}¹*Laboratoire des Matériaux et des Structures du Génie Civil, UMR 113 LCPC-ENPC-CNRS, 2 Allée Kepler, 77420 Champs sur Marne, France*²*Laboratoire de Physique Statistique, UMR 8550 CNRS associated with University P6 and P7, Ecole Normale Supérieure, 24 Rue Lhomond, 75231 Paris cedex 05, France*

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We show that the rheological properties of dry granular materials, as well as foams and emulsions, are similar to typical thixotropic fluids: under a sufficiently strong shear the viscosity decreases in time, leading to a hysteresis in an up-and-down stress ramp. This leads to a viscosity bifurcation around a critical stress: for smaller stresses, the viscosity increases in time and the material eventually stops flowing, whereas for slightly larger stresses the viscosity decreases continuously with time and the flow accelerates. These results show that all jammed systems exhibit strong mechanical similarities around the transition between a “fluid” and a “solid” state, and that the transition between these states is discontinuous. This similarity is further emphasized by the fact that both a simple model for the dynamics of a grain on a sandpile [Quartier *et al.*, Phys. Rev. E **62**, 8299 (2000)] and a simple model for the thixotropic behavior of (colloidal) pastes [Coussot *et al.*, Phys. Rev. Lett. **88**, 175501 (2002)] extrapolated to granular flows qualitatively predict this viscosity bifurcation.

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Jammed systems such as pastes or granular matter are composed of a large number of interacting elements (particles, grains, bubbles, etc.). They can flow like fluids under some conditions, but resist flow under other conditions. For instance, they are capable of supporting their own weight to a certain extent, as happens in a sandpile or in a heap of foam. Such fluids are of great importance in various industries: examples are drilling fluids for the oil industry but also cosmetic gels or creams, cement and concrete, inks, powders, etc., are all systems that can show both solidlike and liquidlike behaviors. There is currently an intense debate about the mechanical behavior of these materials which led Liu and Nagel [1] to propose a conceptual phase diagram for the “jammed” systems. Their proposition is that glassy, pasty, or granular materials show similar mechanical behavior in that these materials are jammed (i.e., they do not flow) at rest, but start flowing (they “unjam”) when the load to which they are submitted increases beyond a critical value.

Generally these systems are thought to exhibit simple yielding behavior, i.e., they do not flow under small applied shear stresses, start to flow for stresses larger than the yield stress and reach a velocity which *continuously* increases from zero with the difference between the applied stress and the yield stress [2,3]. From this (simplified) starting point, attempts have been made to characterize the jamming “transition” with tools using methods from statistical physics [4], drawing an analogy with equilibrium phase transitions. We study here the exact nature of this transition from a mechanical point of view and show that, contrary to what has been hitherto implicitly or explicitly assumed, the mechanical properties show a clear *discontinuity* as a function of the applied stress, which we believe is critical for understanding the underlying general physics connecting these very different systems.

Granular media, foams, and emulsions appear as archetypes of jammed systems, and consequently several attempts have been made to characterize their rheological response [5–7]. Much attention has been paid to determining the velocity profiles in a Couette geometry in which the material is made to flow between two concentric cylinders. These experiments show that for the different systems the flow is localized in the vicinity of the moving cylinder, and the sheared thickness is typically of the order of ten particle (or bubble) diameters. However much less data have been obtained for the apparent viscosity, i.e., the stress as a function of the rotation velocity [3,8]. In the few existing experiments an apparently continuous transition from a jammed (“solid”) to an unjammed (“liquid”) state was observed beyond a critical stress. However, these experiments were carried out under controlled velocity which may preclude observing important behavior characteristics.

In this paper, we investigate the macroscopic rheological response of model granular media, a foam, and an emulsion in detail, using accurate rheometrical tests under controlled stress. We show that a strong common feature of their behavior lies in that these systems are thixotropic, i.e., they become more fluid after a rapid shear and restructure more or less rapidly under a small applied stress. Tests at different stress levels show that this leads to a viscosity bifurcation for a critical value of the stress: for small stresses the viscosity increases continuously in time, and the material eventually stops flowing. For slightly larger stresses, the viscosity decreases with time and the flow accelerates to reach a steady state with a low viscosity after a long time. This immediately leads to the conclusion that stable flows are possible only for shear rates larger than a critical value. This shows that the jamming transition is associated with a discontinuous jump of the viscosity from a low value to infinity, and provides a natural explanation of the observed shear localization. This implies that the “jamming” transition is more complicated than was hitherto assumed, i.e., these materials do not exhibit “simple” yielding behavior; this in particular means that the

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suggested analogy between jamming and the glass transition [1] is no longer straightforward, since the viscosity increases continuously through the glass transition point [2].

Rheometrical tests were carried out with a Bohlin C-VOR rheometer equipped with a (roughened) cone and plate geometry (angle, 4° ; diameter, 4 cm) for the foam and the emulsion. A vane geometry (six blades, diameter, 3.6 cm; height, 4.5 cm) in a roughened cylinder (diameter, 4.7 cm) was used for the granular materials. The vane geometry is used to avoid any perturbing effects due to slip along the inner cylinder with granular materials. In rheology this tool is considered equivalent to the conventional Couette geometry [9]. As is the case for coaxial cylinders, the stress distribution in this setup is heterogeneous so that *a priori* the shear rate cannot be determined directly from the relative velocity of the tools [10]. In addition since the granular materials were not confined in the height direction they could (and did) dilate a little under shear. Previous experiments [7] have shown that this does not change the velocity profile significantly. However this dilatancy, which is geometry and flow dependent, may affect the measured, apparent viscosity. As a consequence, for the granular systems we simply present here the macroscopically measured parameters: the torque on the rotating inner cylinder as a function of its rotation velocity. This reflects the corresponding, effective stress-shear rate curve (i.e., the apparent viscosity under these flow conditions) and does not affect our qualitative interpretations of the results. The materials were polystyrene beads (diameter: 200–300 μm) and two types of glass beads (200 and 500 μm) for which the results were qualitatively similar. The same qualitative effects can be observed with sand but a complete series of data could not be obtained because the velocity after the solid-liquid transition was beyond the range covered by our apparatus. We also studied a foam (*Gillette Foamy regular*) and an emulsion [*mayonnaise (Amora)*].

The process of putting the samples into the measurement cell signifies that some arbitrary residual stress or anisotropy may be present. In order to erase this “memory” before starting the tests, we imposed a constant high shear (high stress or velocity) until reaching a steady state, i.e., a constant stress under imposed shear rate, or the inverse. In this regime it is highly probable that the entire sample flowed, as shown by the fact that all the particles at the free surface were seen to move. This ensured that reproducible results were obtained during subsequent tests. A typical test consisted in imposing a stress ramp increase followed by a reverse stress decrease. The stress was increased logarithmically from its initial value, with typically a few seconds between subsequent stress increases or decreases: we measured the instantaneous apparent viscosity and do not necessarily reach a steady state in this first series of experiments. After each test it was checked that the same preshear as the initial one provided the same stress or shear rate levels, which ensures that strong perturbing effects (segregation, edge effects, coarsening of the foam or the emulsion) do not influence our results. The detailed mechanical responses of the materials show some subtle differences, which are undoubtedly related to their specific microscopic structure and its evolution under

flow. However, the qualitative responses are remarkably similar between these very different materials; we therefore focus on these.

Figure 1 shows that for the three systems, different regimes can be distinguished in the stress ramps.

(1) For very low stresses a continuous increase of the apparent deformation rate is observed (regime 1); this region corresponds to successive, very small deformations; these might be associated to small, irreversible rearrangements [11], and reflect an elastoplastic behavior of the material.

(2) For a stress larger than a critical value there is a dramatic increase of the shear rate (several decades over a very limited stress range): there is an apparent plateau. Imposing the stress, a high shear rate is reached, which may be interpreted as the “unjamming” of the material. This will be referred to as regime 2.

(3) Just after this apparent plateau there is an abrupt change in the slope of the stress-rate curve, the stress and the shear rate increasing together (regime 3). Note that for the foam this slope change is so rapid that the curve, measured with increasing stress, initially presenting a plateau, immediately coincides with the curve obtained upon decreasing the stress. For the glass beads we were unable to reach this regime in this first test because of the very rapid increase of the shear rate with stress, but it was reached in tests under fixed stress values (see below).

(4) Upon subsequently decreasing the stress, it follows that this last regime is the only one for which no time-dependent effects are obtained, i.e., the curves corresponding to the stress increase and decrease exactly superpose in this region; this branch is independent of the flow history of the material.

(5) However, a spectacular effect occurs when further decreasing the stress below the critical value for the unjamming upon increasing the stress: the shear rate decreases continuously with decreasing stress. This branch corresponds to apparent viscosities that are lower than those measured upon increasing the stress.

(6) For even lower values of the stress a new critical value is reached for which the shear drops very rapidly from a relatively high value to almost zero: the system jams abruptly, but this happens at a different stress than the unjamming. This is associated with a new stress plateau, but is even clearer from the corresponding data for the total deformation ($\int_0^t \dot{\gamma} dt$) as a function of time: for the foam and the emulsion, the strain abruptly stops increasing when the system jams, and subsequently even decreases upon further decreasing the stress.

These results consequently show that, unexpectedly, all these materials exhibit an apparent thixotropy: their apparent viscosity decreases with time during shear; a hallmark of this behavior [12] is a hysteresis loop as is found here; indeed in the absence of any thixotropic effect the steady state associated to each stress level would be reached immediately leading to superimposed increasing and decreasing curves. This is unexpected, as thixotropy is usually associated with a (reversible) destruction of the microstructure under flow and a long-term restructuring at rest; for colloidal gels for instance, thixotropy is due to the breaking of bonds under the

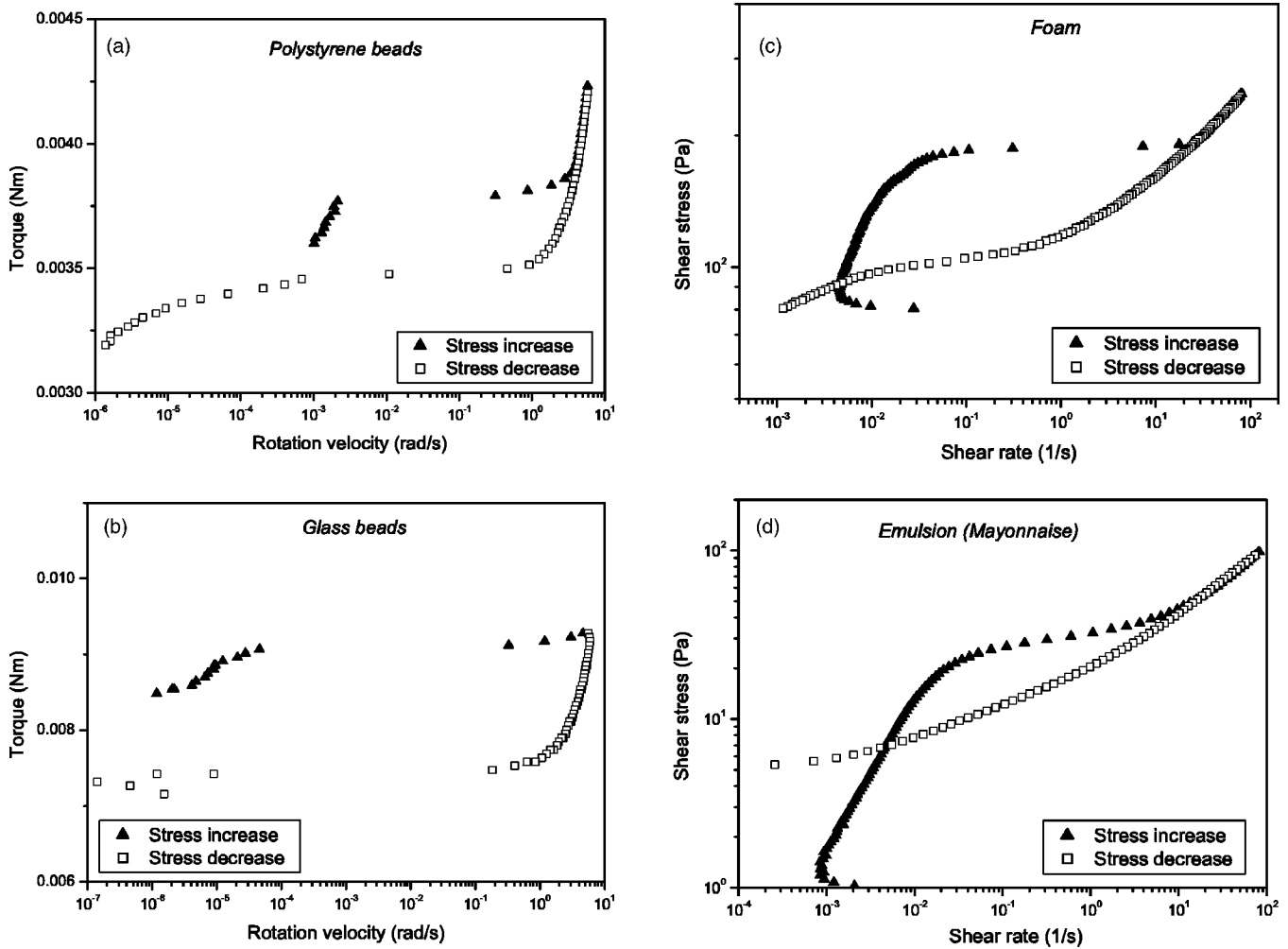


FIG. 1. Torque vs velocity (for granular materials) or stress vs rate of shear curves (for foam and emulsion) during up-and-down stress ramps in a rheometer (preparation procedure in brackets): (a) Polystyrene beads (50 s^{-1} during 10 s, no rest), from 3.2 to 4.24 mN m in 60 (up) + 60 s (down); (b) Glass beads (50 s^{-1} during 10 s, no rest), from 7.16 to 9.28 mN m in 30+30 s; (c) Foam (200 s^{-1} during 10 s, 20 s rest), from 80 to 250 Pa in 120+120 s; (d) Emulsion (150 Pa during 100 s, 120 s rest), from 1 to 100 Pa in 240+240 s.

shear. The microscopic structure of our materials, however, is unaltered after the stress ramp, simply because the initial structure is, within our time scale of observation, immediately recovered at rest. Thus thixotropy is present but with a much shorter characteristic time than for colloidal suspensions. In this light it is worth noting that, as for usual thixotropic fluids, the hysteresis we observe varies with the procedure: if we start, for example, from a more compact assembly of grains, we obtain a higher start-up plateau, while a slower stress decrease leads to a jamming plateau at a higher stress. It follows that at rest or under slow flow these materials restructure themselves leading to a more rigid “structure,” which is destroyed by rapid shear. This explains why the stress associated to the unjamming of the system is larger than that for the jamming of a sheared system, which leads to the existence of the abrupt stress plateaus associated to these transitions.

In order to further study this behavior we determined the steady-state flow behavior. To this aim, after the initial pre-shear we imposed different stress values and followed the

resulting shear rate in time (cf. Fig. 2). Note that the apparent viscosity, which is simply the ratio of the stress to the shear rate, can easily be deduced from these data. These measurements reveal the existence of a critical stress value around which the viscosity bifurcates: for stresses just below the critical value the shear rate decreases continuously towards low values; the system jams. In addition, data not reported here reveal stick-slip motions after longer durations for such stress levels. For a stress just above the critical value the shear rate increases continuously with time to saturate at a large, stable value (larger than a finite value): the system flows. These results are consistent with the hysteresis loops presented above. Indeed these data again show, similar to what was observed recently for “normal” thixotropic systems [12], that there is a critical stress beyond which the material tends to liquefy abruptly and below which the material cannot flow. The direct consequence of this is that there is a large range of shear rates that cannot be reached in the steady state: for a stress smaller than the critical value a small shear rate can be transiently reached but it will then

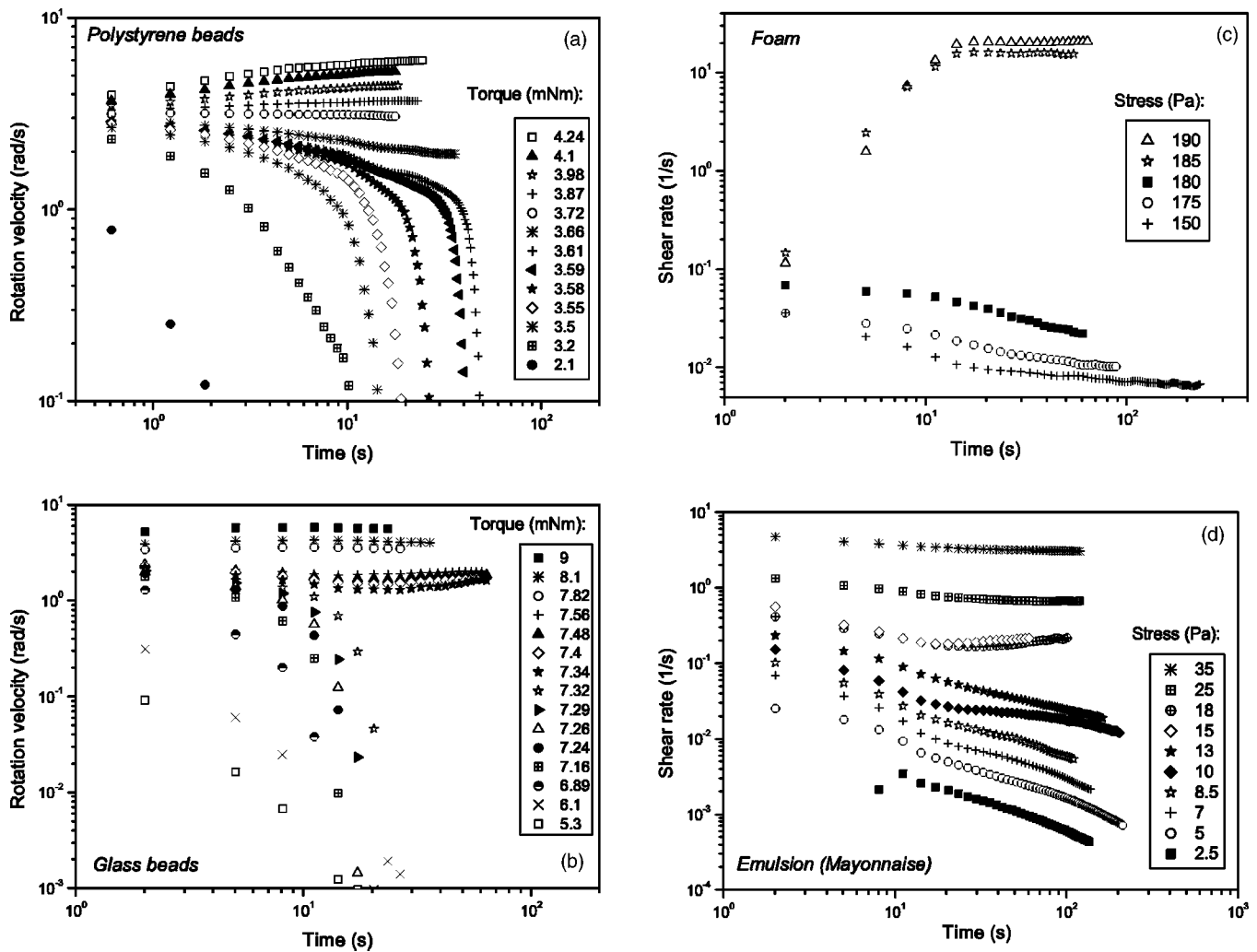


FIG. 2. Velocity (for granular materials) or shear rate (for foam and emulsion) as a function of time for different applied stress levels, after the same preparation of the material: (a) Polystyrene beads (50 s^{-1} during 10 s, no rest); (b) Glass beads (50 s^{-1} during 10 s, no rest); (c) Foam (200 s^{-1} during 10 s, 20 s rest); (d) Emulsion (150 Pa during 20 s, 20 s rest).

progressively decrease until reaching a very low value. For a stress larger than the critical one the shear rate remains larger than a critical value, which obviously is the shear rate associated to the critical stress.

The range of shear rates for which this happens corresponds to the stress plateau in our ramp tests: at the plateau, for a slight stress increase the (apparent) shear rate abruptly varies from a low to a high value. Similar plateau can be obtained from tests under controlled rate with soft-glassy materials, which is typically the signature of a shear banding transition [13]. Although we have no definite proof, here it appears likely that regime 2 is the regime for which recent observations of the velocity profile revealed that the shear rate was stress independent, accompanied by a shear localization in a small band close to the turning cylinder [6,7]. Indeed the apparent shear rates of these previous experiments always fall in the range of apparent shear rates in the second regime in our tests, the maximum value being of the order of 10 s^{-1} , precisely before the value (between 10 and 20 s^{-1}) associated to the transition to the third regime for our mate-

rials. The transition between the second (plateau) and the third regime for high shear rates might indicate that there is an abrupt change in the qualitative aspect of the flow. For example, the material might be rapidly sheared over a distance significantly larger than in the regime 2. At this stage we have no precise measurements but only direct observations of the free surface at the top of the granular samples: almost all the particles in the gap appear to move, thus suggesting that a layer of the order of 25 particle diameters for the smallest glass and polystyrene beads is sheared at a rate significantly larger than that observed in the tail of the velocity profiles in the regime 2. The value 25 is significantly larger than that (around 8) found for the typical shear band in the regime 2 [6,7]. However the exact nature of this transition and the corresponding changes in physical characteristics at this transition remain to be determined.

Previous rheometrical experiments carried out under controlled shear rate also indicate a similar transition from a shear rate independent regime to a regime for which the stress varies as a function of a certain power of the shear rate

[8,14]. From these experiments and simulations [15] the power was found to vary between 0 and 2. Here, above the plateau, the shear stress depends on the shear rate according to a power law with an exponent around 0.5 for glass beads and 0.2 for polystyrene beads. This apparent shear-thinning behavior might be due to the fact that the system is free to dilate vertically. Anyway most of these interpretations (influence of dilatancy, localization of shear, etc.) remain to be proved by further studies using specific techniques (nuclear magnetic resonance imaging, flow visualization, etc.). Here we mainly intend to show the nontrivial transition of these systems from solid to liquid from a mechanical point of view and its similarity with the thixotropic behavior of gels and colloidal glasses [12,16].

Features similar to the ones reported here have been in fact already observed for free surface flows of granular systems under the action of gravity [17]. The solid-liquid transition above a critical shear stress is simply evidenced by the angle of maximum stability on a pile, associated with the internal friction [18]: other studies rather emphasize a critical shear rate separating stick-slip from continuous flow [19,20]. A hysteresis results naturally from the lower value of the angle of repose as compared to the angle of maximum stability, or the lower value of the dynamic friction as compared to the static friction. This is true for a single grain [21], and for a layer of grains flowing down a rough inclined plane [22]: two limiting curves for the solid-liquid transition, whether the flow starts or stops, are found. Similar observations were made for granular flows in a rotating drum as a function of the rotation rate [23]. Our results, obtained in controlled, viscometric flows, show that these features (the hysteresis in solid-liquid transition) are not a result of the specific boundary conditions: at least from a qualitative point of view they constitute intrinsic properties of granular materials. Finally, the slow restructuration of the granular material associated to the divergence of the viscosity recalls the slow granular rheology observed in the compaction of a granular piling under tapping [24] or sinusoidal shearing [25].

A simple phenomenological model proposed for colloidal systems [16] qualitatively reproduces all of the features found here, except for the elastoplastic regime 1. In this model, one assumes that the viscosity is given by the instantaneous structure of the material, which results in the difference between a restructuration rate (mainly depending on the material) and a destructuration by the flow (mainly depending on the instantaneous shear rate). The competition between these two phenomena leads to a continuously increasing viscosity for low applied stresses, where the restructuration wins over the destruction of the structure by the flow: the system eventually jams and stops flowing. At higher stress, the destructuration is dominant, leading to accelerating flows, and a steady state with a low viscosity: the model reproduces the viscosity bifurcation (see Ref. [16] for details). This phenomenon is associated to a (theoretical) steady-state flow curve which decreases at low shear rates: this implies that no stable flows under controlled velocity can be obtained in this range, which accounts for the shear banding. At high shear rates, a well-behaved flow is retrieved from the model, as is indeed observed in the experiments.

The microscopic origins of this competition are obviously different from those for colloidal systems. For granular systems it is the change of the particle configuration in space that may induce viscosity variations. This happens in confined systems but, in other geometries, dilatancy (and compaction for slow flows) surely contributes also. The detailed microscopic understanding of the liquid-solid transition lies in the dynamics of the contact network and the evolution of the texture (coordination number, mobilization of friction) near the onset of flow [26] and near jamming [4]. Conceptually this may still be considered as a destruction or rebuilding of the structure so that the qualitative predictions of the model are not affected. In the case of foams and emulsions, the microscopic interpretation is more difficult; the relaxation time of a deformed bubble or droplet is very small, and it is more likely that, since the system is intrinsically under tension, local and collective rearrangements at rest are responsible for the observed viscosity variations with time. The flow on the other hand, will deform the bubbles or droplets, which will lead to the observed viscosity decrease.

It does not seem useful to compare these observations with the existing models of the dynamics of granular systems since they mainly concern the steady-state flow properties or are incapable to predict start-up flow as well as developed flow and stoppage. There nevertheless exists one model which exhibits these characteristics, although it aims at describing the dynamics of a single grain on a rough plane [21]. Here we will assume that the behavior of one grain can be extrapolated to that of a granular flow as a whole, in an approach somewhat similar to that of Jaeger *et al.* [27], since the basic ingredients of motion (collisions, trapping, gravity effect) are similar. Here we do not discuss the validity of this model but simply show its ability to predict our experimental results, thus suggesting a physical explanation for these. The principles of the model are as follows: the force exerted on the grain is the sum of two terms, one (F_0) tending to “trap” it in the potential wells (corresponding to the bed roughness), the other (F_∞) related to the steady motion involving a balance between collisional dissipations and gravity. The complete dynamics is described by a “matched” sum of these forces,

$$F = (1 - h)F_\infty + hF_0, \quad (1)$$

where $h(V)$ is a matching function which tends towards zero when the velocity $V \geq 1$ and tends towards 1 when $V \rightarrow 0$, and may be interpreted as a proportion of trap inside the flow. This matching procedure attempts to represent reality with respect to the asymptotic conditions: Equation (1) predicts that the flow is either mainly governed by F_∞ ($V \geq 1$) or by F_0 ($V \rightarrow 0$) and provides an expression for the force in the intermediate cases.

The force F_∞ was determined experimentally [21]. For our purpose it is sufficient to retain the simplified dimensionless form

$$F_\infty = (\theta - \theta_1) - V^2. \quad (2)$$

The trapping force is supposed to take the (simplified and dimensionless) form

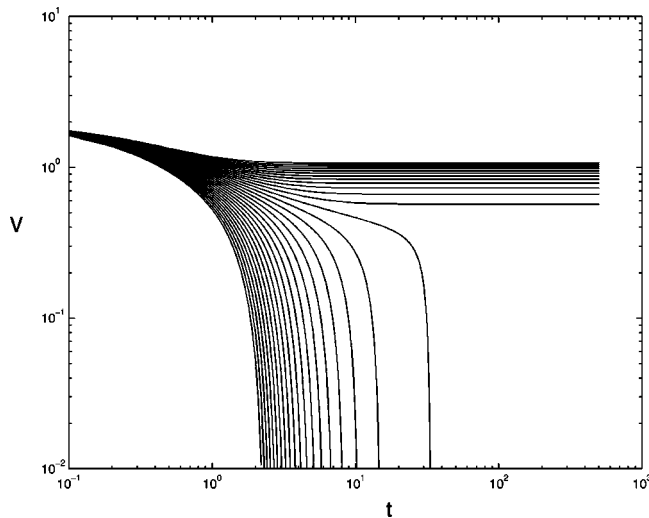


FIG. 3. Velocity as a function of time as predicted by the model (1–3). Here the values of the model parameters are $\theta_1=1$, $\theta_2=2$, $V_i=2$. The curves correspond to different values of bed slope (θ_f) between 1.9 and 1.1 by steps of $\frac{1}{30}$ (from top to bottom).

$$F_0 = V^{1/2}(\theta - \theta_2). \quad (3)$$

These forces depend on the difference between the bed slope (θ) and two reference angles (θ_1 and θ_2 such that $\theta_1 < \theta_2$) related to the two critical angles of stability of a sandpile. The model becomes complete after writing the momentum equation ($F = dV/dt$) and by assuming a reasonable form for the matching function, $h(V) = \exp(-V)$. In these equations the force, velocity, and time were scaled, respectively, by the gravity force acting on one grain (mg), the typical velocity

of a grain falling in a trap under the action of gravity only, and the time of this fall.

The equivalent of the imposed stress in our experiments is the bed slope in this model. Let us consider the case of a steady-state flow for a slope $\theta_i > \theta_2$: the granular material at a fixed initial velocity (V_i). Now the bed slope is suddenly changed to a given value θ_f , in the range $[\theta_1; \theta_2]$. It appears that the evolution of the velocity with time for different angles is similar to that observed for our granular material under different stresses in our rheometrical tests (cf. Fig. 3): depending on the relative values of the imposed angle and a critical value the velocity evolves either towards zero rather abruptly after a finite time (decreasing as the difference between θ_f and the critical angle) or tends towards a value larger than a finite, critical velocity. Although this model is mainly qualitative it shows that the viscosity bifurcation for granular materials may find a natural explanation on the basis of the usual, physical ingredients in this field.

In conclusion, our results show that there is a remarkable similarity in the mechanical behavior of granular materials, emulsions, and foams with that observed for polymer or colloidal gels and even colloidal glasses. Although the microscopic origins and the characteristic times are very different, all these systems show an apparent thixotropic behavior, leading to a viscosity bifurcation around a critical stress. This tends to show that the common denominator for jammed systems as proposed by Liu and Nagel [1] are their mechanical properties. However, our measurements also clearly show that the jamming and unjamming of these systems are discontinuous rather than continuous, and that the situation is more complicated than the simple presence or absence of flow in response to an applied stress.

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