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PHYSIQUE DE LA MATIÈRE EN GRAINS PHYSICS OF GRANULAR MEDIA

Pastes: lubricated and cohesive granular media

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Abstract We analyze, in the light of experiments, a few typical aspects of the rheological or mechanical behavior of pastes, defined as dense and liquid-saturated assemblies of grains, with or without physical-chemical interactions. Emphasis is placed on the consequences of jamming, which distinguishes pastes from concentrated suspensions, contact lubrication, which distinguishes pastes from dry granular media, and cohesion. The points addressed are the following: the onset of flow, the contribution of friction in the yield stress, solid-liquid segregation and compaction, adhesion (tackiness) and cohesive rupture. *To cite this article: H. Van Damme et al., C. R. Physique 3 (2002) 229–238.* © 2002 Académie des sciences/Éditions scientifiques et médicales Elsevier SAS

pastes / plasticity / rupture / yield stress / dilatancy / vibrocompaction

Les pâtes : des milieux granulaires lubrifiés et cohésifs

Résumé Nous examinons, expériences à l'appui, quelques aspects caractéristiques du comportement d'une pâte, définie comme une assemblée dense de grains, saturée de liquide, en présence ou pas d'interactions physico-chimiques. L'accent est mis sur les conséquences de l'encombrement, qui distingue les pâtes des suspensions concentrées, et de la lubrification des contacts, qui distingue les pâtes des milieux granulaires secs, et de la cohésion. Les points abordés sont, successivement, le mode de mise en écoulement, la contrainteseuil, la densification sous vibrations, l'adhérence et la rupture cohésive. *Pour citer cet article : H. Van Damme et al., C. R. Physique 3 (2002) 229-238.* © 2002 Académie des sciences/Éditions scientifiques et médicales Elsevier SAS

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1. Pastes: jamming and plastic flow

Compared to other forms of soft condensed matter, such as colloidal sols or gels, polymer melts or semidilute solutions, emulsions, foams and granular media, pastes still represent an ill-defined state of matter. In fact, all the previous media may probably be in the 'paste-like state', provided a few conditions are satisfied. If we refer to the archetypes of the paste-like state, putty, fresh mortar, bread dough or mayonnaise sauce for instance, one is led to the conclusion that what gathers these soft solids or complex fluids under the common name of pastes probably boils down to two properties. The first is essentially structural. In spite of their chemical diversity, all those systems are *dense assemblies* of rigid or deformable objects, close to a jamming situation (clay platelets in the putty, sand and cement grains in the fresh mortar, starch and gluten grains in the dough, oil droplets in the mayonnaise) (Fig. 1, showing results of [1]). The packing density is such that no particle can move over a distance of, say, one particle diameter, without deeply perturbing the position of its neighbors, or their shape if they are deformable. The second general property of pastes is more of mechanical nature. Pastes are media which withstand important *plastic deformations*. Beyond some elastic limit or plasticity threshold, they deform continuously at quasi-constant stress. However, any drop of the stress level below this threshold leads to the instantaneous stop of this (visco)plastic flow.

The choice just made for the characteristic properties of pastes has the virtue of emphasizing two features which are usually not considered in the rheological study of suspensions. The first is the potential importance of *contacts*, as a consequence of jamming. Interparticular tribology becomes an important component of mechanical behavior, just like in dry granular media in the quasi-static regime [2]. However, the presence of an interstitial fluid in pastes opens the way to all sorts of surface interactions able to modulate the inter-granular friction. Thus, a paste may be considered as a granular medium, lubricated by hydrodynamic or physical–chemical interactions. The second feature introduced by the definition of pastes adopted here is the use of the stress–strain plane, (σ , ε) or (τ , γ), instead of the usual (τ , $d\gamma/\delta\tau$) plane. In other words, a mechanical approach becomes as relevant—if not more—than the classical rheological approach of suspensions.



Figure 1. Typical structure of a paste. In this example (adapted from [1]), colloidal silica particles are dispersed in a apolar organic solvent. The packing is close to the random close packing limit and, in static conditions, the grains interact via a repulsive hard sphere potential, thanks to a grafted hydrocarbon brush on the silica surface. With a 10^3 increase in length scale, the same image might represent a fresh mortar paste.



Figure 2. Stress–strain curves obtained by shearing a clay gel (1% solid volume fraction; [3]) and a mortar paste [4] with a vane tool (a rod with 4 or 6 coaxial blades immersed in the medium). The vane is rotated at constant strain rate (10^{-4} and 10^{-2} s⁻¹ for the gel and the paste, respectively). The initial elastic regime is followed by fragile rupture and viscous flow in the gel and by plastic flow in the paste.

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The choice to link the pasty state of matter to jamming and to plastic flow allows us also to distinguish pastes from the numerous yield stress fluids containing a flocculated network of particles. In those fluids, there is also an elastic limit, corresponding to a yield stress, reflecting the (limited) strength of the highly porous percolating gel network. However, in a constant strain rate mechanical test, once this limit is overcome, the network collapses due to the rupture of critical bonds, and the stress drops to much smaller values (Fig. 2, from [3,4]). The residual stress level may possibly reach a stationary value, related to the size of the network fragments at a given strain rate. In pastes, provided the strain rate remains of the same order as the time scale for grain motion, passing beyond the elastic limit does not deeply modify the structure of the medium. Owing to the high density of particles, the number of nearest neighbors remains essentially constant and so does the stress and the cohesion of the medium (Fig. 2). In terms of stress–strain curves, paste-like behavior is characterized by plastic flow and possibly ductile rupture at large strain and large strain rates, whereas gel-like behavior is characterized by a limited elastic deformation followed by fragile rupture at low strain and, finally, viscous flow or, possibly, slipping.

Paste-like behavior may be observed both with colloidal and non colloidal particles. In colloidal pastes, Brownian motion provides the diffusion mechanism allowing us to keep statistically unchanged the dense structure and to heal the defects, especially in tensile strain [1]. This is analogous to high temperature superplastic strain in nanophase ceramics, where thermally activated atomic diffusion plays the role that Brownian motion plays in colloidal pastes [5]. This spontaneous healing mechanism does not exist in pastes of larger grains. In that case, application of a confinement stress may help to avoid defect growth and to retard failure. This is the basis for the so-called triaxial test in soil mechanics [6]. Flocculated colloidal suspensions provide an interesting example of a more complex situation. Starting from a stable viscous sol, addition a flocculation agent may lead either to a percolating network, i.e. to a gel, or, alternatively, to a collection of finite aggregates, i.e. to a paste-like sediment, depending on the relative gelation and sedimentation rates [7]. In the latter case, the structural units in the paste-like sediment are not individual colloidal particles but flocs, forming an entangled packing of fractal objects.

2. Inter-granular friction and gel strength

Midway between the world of colloids and that of granular media, pastes should exhibit some properties typical of each one of those worlds. One of the basic properties of granular media is dilatancy, discovered by Reynolds [8]. A disordered dense packing of hard grains cannot undergo a shear strain without simultaneously expanding in the direction perpendicular to the shear plane. On the other hand, in the high density and low strain rate quasi-static regime, deformation of granular media is also controlled by the inter-granular friction, which expresses itself in the well-known Coulomb laws which, for a heap of granular material, relate the maximum angle of repose, θ , to a static friction coefficient, μ_s , by $\mu_s = \tan \theta$, and which, for a moving body in sliding contact with a surface, relate the tangential force for initiating the sliding motion, T, to the total normal force, N, by $T = \mu_s N$ [9]. Although extensively studied in dry granular media, these phenomena have given rise to little interest so far in the case of pastes, especially in colloidal pastes [10]. Yet, they should appear, even though we may expect them to be modified by presence of the interstitial fluid.

Among the various parameters used to describe the rheological behavior of pastes and concentrated suspensions in general, the yield stress, τ_y , is probably the one which is the most able to reveal a possible frictional contribution, in addition to the usual gel strength contribution when the system is flocculated. On the other hand, the appearance of normal forces in shear flow would be direct evidence of dilatancy. This combined influence of dilatancy and inter-particle friction on rheological behavior has recently been studied in the case of flocculated calcium silicate pastes [11,12]. Tricalcium silicate (C₃S in short) is the most important component of ordinary Portland cement. The particle size is of the order of several μ m or tens of μ m, well above the colloidal domain. The particle surface is charged but the large ionic strength of



the aqueous interstitial solution in equilibrium with the solid screens the double layer repulsive forces, so that, if no dispersing polymer is added, C_3S or cement pastes are flocculated.

The evolution of the strain rate and normal force in a shear experiment with a controlled stress rheometer is shown in Fig. 3. One can see that the onset of flow is concomitant with a jump of normal force, clearly revealing the dilatant character of the paste layer. However, as shown in Fig. 4, this jump only appears when the solid volume fraction is above some critical value, which may be considered as a jamming value, ϕ_j . This value is close to 0.37, much below the random close packing or random loose packing values, confirming that the particles which are in a jamming situation are not hard and compact spherical particles but open flocs. Also shown in Fig. 4 is the evolution of the yield stress with paste concentration. If the only contribution to the yield stress were the inter-granular frictional force, one would expect a zero yield stress below ϕ_j . This is not the case. A yield stress is measurable well before, at a volume fraction ϕ_g of the order of 0.28, which may be considered as the threshold value for the formation of a weak gel. As expected for a gelation process, τ_y increases according to a power law of ($\phi - \phi_g$) above ϕ_g . However, above ϕ_j , an additional contribution appears, which may be assigned to an inter-floc friction stress. Remarkably, this frictional contribution is increasing roughly linearly with the normal force (Fig. 5), confirming that Coulomb's law indeed applies to pastes. Thus, the parallel measurement of the yield stress and normal force in shear flow allows us to separate clearly a gel strength contribution to the yield stress



 τ_{int} , generated by DLVO or non-DLVO surface forces, and a frictional Coulomb contribution, τ_{fr} , typical of granular materials in jammed conditions. The coexistence of both aspects is probably a general feature of pastes.

3. Density relaxation and lubrication

In dense granular media, blocking stems from the combined effect of inter-granular friction and vanishing free volume as the density approaches the RCP limit. This so-called jamming transition bears a close resemblance to the glass transition [13,14]. In fact, the simplest model for the glass transition—the free volume model—in which a structural rearrangement probability is calculated by considering the redistribution of free volume in a dense gas of hard spheres [15], might equally well be applied to glasses and to ideally lubricated colloidal pastes. In a dry and static packing of hard grains submitted to its own weight, rearrangements are impossible, unless some free volume is temporarily injected into the system, for instance through taps or sinusoidal vibrations [16,17]. This slow relaxation process leading to denser packing has been shown to follow logarithmic kinetics, according to the following relation for the evolution of the solid volume fraction ϕ :

$$\phi = \phi_{\rm f} - \frac{\Delta \phi_{\infty}}{1 + A \ln(n+1)},\tag{1}$$

in which ϕ_f is the asymptotic reached final solid volume fraction and $\Delta \phi_{\infty}$ the total increase of ϕ . A is a constant and packing dependent parameter and *n* is the number of taps. This logarithmic kinetics has been shown [18] to be consistent with a free volume model, in which the probability for a structural rearrangement event, $W(\phi)$, is given by the original free volume equation:

$$W(\phi) \propto \exp\left(-\frac{v^*}{v_{\rm f}}\right),$$
 (2)

where v^* is a critical free volume for rearrangement and v_f the average free volume per grain in the packing, defined by:

$$v_{\rm f} = v_{\rm p} \left(\frac{1}{\phi} - \frac{1}{\phi_{\rm max}} \right),\tag{3}$$

where v_p is the real volume of one grain and ϕ_{max} the maximum disordered packing density.

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Figure 6. Densification of dry (closed circles) and oil-saturated (open symbols) packings of 0.5 mm glass beads under periodic taps. The Newtonian viscosities of the oils are 5 (circles), 100 (triangles) and 1000 (diamonds) mPa·s, respectively. The presence of the interstitial fluid slows the densification down but, by lubricating the contacts, allows higher final densities to be reached.

In pastes, the presence of the interstitial fluid is expected to introduce two significant differences with respect to dry granular media. The first is the slowing down of the grain motion under a given force, due to viscous drag. The second is the lubrication of contacts, i.e. the reduction of the inter-granular friction coefficient. As shown by the results in Fig. 6, both effects are clearly shown by the density relaxation curves of columns of glass beads in air or in oil submitted to periodic taps [19]. Dry and oil-saturated packings made of 500 μ m glass beads were prepared in gauged test tubes and submitted to periodic taps. The density value was known with a ± 0.005 accuracy. Three silicon oils having different viscosities (0.005, 0.1 and 1 Pa·s) but the same density were used in order to explore the effect of the fluid viscosity only on the kinetic law of densification.

The densification curves figuring the solid volume fraction ϕ plotted against the number *n* of taps show that densification goes qualitatively the same way, whatever the fluid in the packing (air or silicon oils) (Fig. 6) [19]. This suggests that the mechanism underlying the densification process is the same in dry and pasty packings. Two regimes may be identified: a 'fast' initial regime and a 'slow' asymptotic regime. The initial regime is hardly noticeable in the dry packings (it concerns only the first tap applied) but is clearly visible in the paste-like packings. As expected, the densification rate in this 'fast' regime is slower in the paste-like packings, and the more viscous the interstitial fluid, the slower the initial densification rate. This may be due to the smaller kinetic energy imparted to the particles at each tap, due to viscous drag, and suggests that the particle motion in this initial regime is essentially ballistic. The final regime of the densification process is of a different nature. The key result here is not related to kinetics but to the final equilibrium state. As visible in Fig. 6, the more viscous the interstitial fluid, the higher the final density. In this case, it is clearly the increasing lubricating power of the fluid which is involved, which suggests that the final regime is controlled by inter-granular friction.

Actually, lubrication is probably involved at all stages of the densification process. Owing to the high initial density in pastes, the average inter-particle distance is always smaller than the particle diameter. Each particle is enclosed in the cage formed by its neighbors and lubrication is involved both in the center-to-center approach of the particles and in the sliding motion of particles in contact or, more precisely, separated by a lubricating film. Lubrication in the later case may be accounted for by taking into account a smaller friction coefficient μ_s in Coulomb's law. In the former case, the problem is more complex. The force we need to apply for approaching two equal spheres at constant velocity increases with this velocity and diverges as (interparticle distance)⁻¹ [20]. Thus, qualitatively, the denser the packing, the slower further densification is expected at constant force, independently of free volume considerations. In low viscosity fluids such as air, this lubrication effect may probably be neglected, but in liquids, it has to be considered.

It is therefore not surprising that Eq. (1), initially established for dry granular media [16] and derived from simple free volume considerations [18], is in good agreement with the data obtained for the dry

Figure 7. Densification curve of an oil-saturated pasty packing of 0.5 mm glass beads in a 5 mPa·s viscosity silicone oil, under sinusoidal periodic vibration. Contrary to what is observed with taps, a decompaction regime is observed before the onset of densification. The duration of the decompaction regime increases with the depth into the vibrated column (circles: bottom; squares: middle). The parameter $\Theta(n)$ is defined as $[\Phi(n) - \Phi_{in}]/[\Phi_{max} - \Phi_{in}]$, where Φ_{in} and Φ_{max} are the initial and maximum solid volume fraction in the packing, respectively.



packing densification in Fig. 6, but fails to describe correctly the densification of the pasty packings. The final density ϕ_f is overestimated and the density variations are not accurately reproduced. These results led us to generalize Eq. (1) in order to describe both dry and saturated packings [19]. The constant parameter *A* was replaced by a function of *n* in order to take into account the increasing importance of lubrication in the center-to-center relative motion of particles. An exponential form, $A(n) = \alpha \exp(n/n_t)$, allows at the same time for a more satisfying prediction of the final density value and for a satisfying description of the whole density evolution. The best fit characteristic number of taps, n_t , in the previous equation corresponds approximately to the inflexion point of each curve, which occur roughly at the same density for all the packings, around 0.62. This is well within the density range where jamming is important.

Shortening the rest time between taps strongly modifies the course of compaction, confirming that sluggish lubrication-controlled motions are acting. The general trend is that increasing the frequency of taps introduces a latency time before the usual densification process. During this latency time, no significant densification is observed. Remarkably, when truly sinusoidal vibrations are applied instead of taps, an initial *decompaction* phase is observed before the start of the densification process (Fig. 7) [21]. The onset of densification is then particularly rapid. The whole process looks like a slow swelling phase, followed by a sudden collapse. What gives the start signal of this collapse is still unknown.

4. Cohesion, tackiness and rupture

Where does the cohesion of pastes come from? This deceptively simple question has several facets. On simple thermodynamic grounds, if we stick to the usual definition, $W_{ad} = 2\gamma$, where γ is the surface energy, a paste should have a vanishing cohesion energy. Indeed, since the surface of a paste is always covered by a liquid film, the cohesion energy should be of the order of $0.1 \text{ J} \cdot \text{m}^{-2}$. This is way below the energy which has to be spent to actually separate in two pieces a lump of paste. Thus, other sources of cohesion have to be looked for. Capillary forces are a first possible important source of cohesion. They are totally responsible for the cohesion of wet sand, where they are acting in all the liquid bridges between particles, but they vanish in liquid-saturated media at rest, when the curvature of the air–liquid boundary at the paste surface is close to zero. However, in dry conditions, i.e. when evaporation takes place, the liquid–air menisci tend to enter deeper in the inter-particle voids. Their curvature increases, increasing the Laplace pressure drop, $\Delta P = \gamma \kappa$, where κ is the mean curvature of the meniscus, and the cohesion of the medium. Similarly, when a dense paste is sheared, dilatancy forces the liquid–air menisci to enter deeper into the medium, increasing the cohesion.

Actually, the major part of the energy required to overcome the cohesion of a lump of paste in a real experiment performed at finite strain rate is lost in dissipation. A convenient setup to measure this is the so-called probe tack test, in which a thin layer of paste is confined between two parallel plates which are pulled

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Figure 8. Patterns or profiles obtained after separation of two square $(0.25 \times 0.25 \text{ m}^2)$ glass plates in between which a thin layer of colloidal suspension or granular paste was placed. Top: a non-Newtonian shear-thinning bentonite suspension (5%, w/w in water). Middle: a Laponite gel (2% w/w in water). Bottom: a mortar paste ([cement + sand]/water = 0.28). The dominant features of the fracture mechanism are viscous flow with Saffman–Taylor-like fingers in the suspension, slippage and interfacial crack propagation in the gel, cavitation, followed by plastic flow and finally ductile rupture in the paste [4,25].

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apart. This geometry is classically used to probe the stickiness or 'tack' of pressure-sensitive adhesives [22]. In this strongly confined geometry, a large negative hydrostatic pressure builds up as the plates are pulled apart. This hydrostatic component of the stress field is responsible for the extensive cavitation observed during debonding of soft adhesives. These materials are weakly cross-linked or non-cross-linked polymer melts. Within the framework of a purely elastic model, a cavity is predicted to grow in a medium of Young's modulus *E* if $\sigma > 5E/6$, regardless of its size [23]. This condition can be readily extended to a viscoelastic medium, such as a polymer melt, by replacing *E* by a dynamic quantity, $G'(\omega)$, the storage shear modulus at a given frequency [22].

Cavitation competes with a simpler mechanism which is simply flow of the layer towards the center of the plates. This flow is essentially shear flow in the beginning and becomes increasingly extensional as the separation increases. The driving force for the flow is the pressure gradient within the layer. When a probe tack experiment is performed with a low viscosity and incompressible Newtonian fluid, flow towards the center is the debonding mechanism which is dominating, unless very high separation rates are applied. However, the air–liquid interface at the boundary of the layer is unstable and Saffman–Taylor-like fingers form readily, growing inwards. When a non-Newtonian shear-thinning colloidal suspension is used, the mechanism remains basically unchanged. The formation of fingers is even easier. In addition, the yield stress of these fluids prevents the walls between fingers from flattening after total separation of the plates, allowing the visualization of the geometry of finger growth (Fig. 8). With gels or non-Newtonian fluids with a high yield stress, the interface between the plates and the gel becomes the weak part of the system and debonding occurs by the combined action of crack propagation at the interface and slippage (Fig. 8).

Once more, pastes show a marked departure from those non jammed situations. Cavitation becomes again the main failure mechanism, as in pressure sensitive adhesives (PSAs), and the rupture profile is typical of ductile rupture (Fig. 8), in agreement with the plastic character of deformation (Section 1). No significant fingering is observed. This may stem from the small compressibility that most pastes possess, owing to the almost unavoidable inclusion of air during preparation. Indeed, it has been shown that even a very small compressibility is enough to flatten the pressure profile within the layer and to suppress the driving force for fingering [22,24]. One important difference with PSAs, however, is the absence of fibrillation. In PSAs, the lateral growth of the cavities leads to thin walls which can withstand large extensional strain (think of molten cheese). Colloidal pastes and even less granular pastes cannot withstand such large extensional strain and rupture occurs at moderate plate separation. There is no direct (i.e. visual) evidence for the mechanism which, from localized cavitation, leads to rupture of the paste layer, but it might probably be viewed as a generalized damage process, resulting from the increase in the number of cavities. Contrary to what happens in PSAs, where there is no intrinsic characteristic length scale and where the size of the cavities is essentially determined by the plate separation, in pastes, there is a natural length scale for the cavities, which is the size of the inter-granular voids.

5. Conclusion and open questions

In this overview, we have tried to show that pastes, defined as dense suspensions of colloidal or granular particles in a liquid, exhibit distinct features with respect to both concentrated suspensions and dry granular media. Major questions remain to be solved. Perhaps the most basic is the microscopic description of flow close to this jamming limit. Plastic flow in crystals is made possible by extended defects such as dislocations. This can hardly be extended to amorphous materials and to pastes in particular. Much more realistic on physical grounds would be a mechanism analogous to the superplastic deformation mechanism in nanophase polycrystalline metals and ceramics, as already mentioned in Section 1 [5]. This implies that deformations occur by the sliding of kinematically coherent domains along slip planes, analogous to grain boundaries. The relative positions of particles within each domain remains unchanged during flow, but fast diffusion of a small number of boundary particles is necessary to accomodate and to fill in the voids opened

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by the sliding [26,27]. Interestingly enough, the same type of mechanism is also considered for flow of inorganic glasses in the vicinity of the glass transition [28].

A second open question to be addressed is the role of particle size distribution. While most theoretical efforts devoted to the physical understanding of suspensions and granular media are focusing on morphologically (spheres) and statistically (monodisperse) simple systems, real media are almost invariably more complex. This should not be considered as a second-order perturbation. An extreme example is that of fresh high strength concrete, in which up to six populations of grains are used, spanning more than five decades in particle size. This allows remarkably high solid densities and strength to be reached in the hardened material but, at the same time, decreases dramatically the viscosity and yield stress of the fresh paste. How this extremely broad particle size distribution modifies the flow mechanism in the fresh paste and the stress propagation in the hardened paste remains to be established.

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