

# Ageing of the avalanche angle in immersed granular matter

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**Abstract.** The stability of packings of glass beads immersed in various fluids is studied experimentally in a rotating drum. The avalanche angle depends on the time during which the system remains at rest before measurement only when the grains are immersed in water. The experimental results prove that ageing of immersed granular materials, under low applied stress and in the absence of external sollicitations, only originates from chemical reactions that occur at the surface of the grains.

**PACS.** 45.70.Cc Static sandpiles; granular compaction – 81.40.Cd Solid solution hardening, precipitation hardening, and dispersion hardening; aging – 45.70.Ht Avalanches – 81.40.Lm Deformation, plasticity, and creep

## 1 Introduction

The physical properties of dense packings of solid grains are the subject of numerous both experimental and theoretical studies [1–3]. These so-called *granular materials* are of practical interest as they appear in many industrial or geophysical situations. From the fundamental point of view, the physicist is interested in establishing the relation between the physical properties of the individual grains (*mesoscopic* scale) and their resulting collective behavior (*macroscopic* scale).

Among the puzzling properties of granular materials, the mechanics of a dense packing of solid grains remains largely mysterious. A dry or immersed granular layer of grains, subjected to an external shear stress at low applied normal stress, exhibits a solid-like frictional behavior [4–7]; the frictional force is proportional to the applied normal stress, and does not significantly depend on the shear rate. The result holds true for geophysical materials at high pressure [8]. In addition, the free surface of a granular material, when tilted, is recognized to start flowing above a threshold angle, the angle of avalanche, which is usually of about 30 degrees [3,9]. There is an intrinsic relation between the frictional properties of the granular material and the angle of avalanche as the static properties of granular systems can be discussed in terms of frictional properties between different granular layers [3]. Obtaining a continuous description of these systems is difficult as the result of a given experiment can depend on the preparation of the sample; for instance, the flowing properties of the material depend on the density of the initial granular packing, and on the possible asymmetries of its internal structure [10]. Moreover, the avalanche angle

generally depends on the *resting time* (the time the system remained at rest previous to the measurement) [11]. Such *ageing* of granular matter is also reported in friction experiments [12]. Thus, identifying the potential physical origins of the observed ageing would help to find a continuous description of the flowing properties of granular matter from the initial avalanche to continuous flow.

Various studies have shown strengthening with time of granular layers at geophysical pressures [8]; at large applied stress, creep at the points of contact between grains can occur and account for the increase of the resistance of the material to the shear. Creep is expected not to play any significant role when the overall stress applied to the material is reduced. Nevertheless, even at low applied stress, one observes a significant increase of the static friction coefficient with time when sliding a plate across the free surface of a dry granular layer [12]. The avalanche angle of dry granular matter also generally depends on time [11, 13, 14]. The question of the physical origin of such ageing at low applied stress has been partially answered for systems of grains placed in a humid atmosphere. Capillary condensation of water vapor between the packed particles accounts for ageing of dry granular matter. The formation of liquid bridges at the contact points introduces adhesion forces between the grains leading to a significant increase of the avalanche angle and of the static frictional force [15, 16]. Moreover, chemical reactions can occur within the liquid bridges and adhesion forces between grains can be larger than those predicted when taking into account only the capillary forces [14].

In order to test if there were remaining ageing effects in absence of capillary condensation, friction experiments have been performed in immersed granular materials at low applied stress [12]. Glass grains immersed in water still exhibit an unexplained ageing: the strength of the sample

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increases logarithmically with the resting time, and a puzzling creep of the granular material at rest is observed. The condensation of liquid bridges cannot account for ageing of immersed systems, and the physical origin of the phenomenon still remains to be discussed in details.

Ageing can be induced by a slow compaction of the material; an increase of the material compacity leads to an increase of the frictional force, and hence of the avalanche angle. In addition, in immersed granular matter, the presence of thin liquid films around the contact points can delay the flow or the avalanche leading to an apparent increase of the thresholds. Indeed, the measurements are performed at small velocity in friction experiments, and at small rotation speed in rotating drums in order to sense quasi-static properties. If the typical hydrodynamical time necessary to pull two grains apart becomes large, an apparent increase of the thresholds can be observed. Compaction can also lead to changes in a bulk adhesion force (Van der Waals interactions, electrostatics). If ageing originates from compaction and packing rearrangements, the dynamics of the phenomenon is governed by the hydrodynamics. Finally, ageing can originate, when observed, from chemical reactions that gradually bond the grains together.

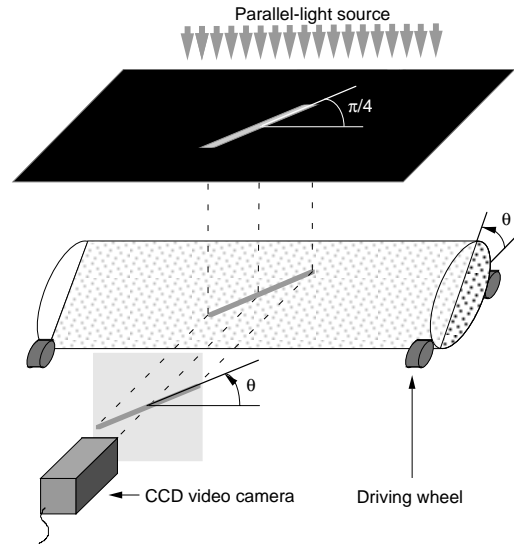
The present article reports measurements of avalanche angles performed in immersed granular material proving that ageing of immersed granular matter, when present, only originates from chemical reactions that occur at the surface of the grains.

## 2 Experimental setup and protocol

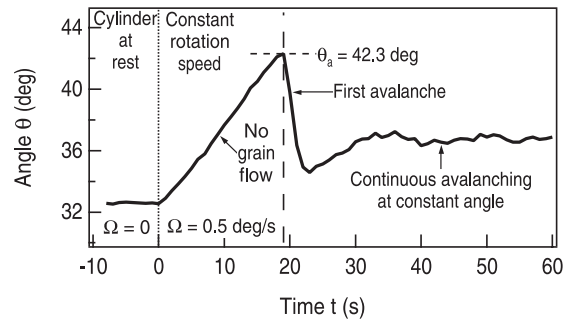
The experimental setup (Fig. 1) consists of a horizontal glass tube (inner diameter 19 mm, length 190 mm) which can be rotated around its axis at constant angular velocity  $\Omega$  (0.3 to 8.0 deg/s) with the help of a DC motor. The cylinder is half filled with granular material, and the remaining free space (the void space between the grains and the other half of the inner volume) is filled with a liquid (special attention is paid not to leave any gas bubble trapped in the system).

In immersed systems, because of strong hydrodynamical interactions, the free surface of the granular material deforms significantly close to the side walls. In order to avoid these boundary effects, we measure the angle  $\theta$ , that the upper surface of the granular packing makes with the horizontal plane, far from the two ends of the cylinder (*i.e.* at least 80 mm away from the walls while the radius of the cylinder is less than 10 mm): one CCD video camera, whose optical axis is horizontal and perpendicular to the axis of rotation, images light coming along the vertical axis from a horizontal slit and reflected by the free surface of the heap. When the slit makes a 45-degree angle with the axis of the cylinder, the image of the slit makes the same angle  $\theta$  with the horizontal axis. One computer controls the angular velocity  $\Omega$ , digitizes the images, and makes hence possible to measure  $\theta$  within 0.2 degrees every second.

The three different samples of granular material used in the experiments consist of glass beads (sodosilicate



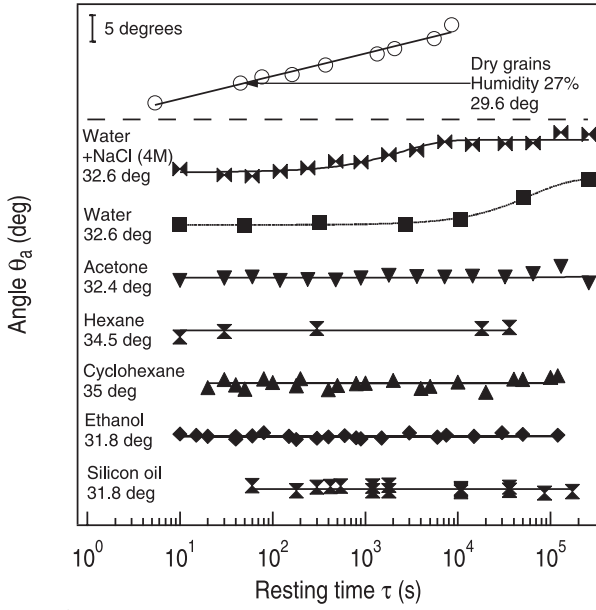
**Fig. 1.** Perspective view of the experimental setup. The granular material half fills a horizontal cylinder (circular cross section) which can be rotated around its axis. One CCD video camera, whose optical axis is horizontal and perpendicular to the axis of rotation, images light coming along the vertical axis from a horizontal slit and reflected by the free surface of the heap.



**Fig. 2.** Angle  $\theta$  as a function of time  $t$ . The angle  $\theta$  initially increases linearly with time  $t$  ( $0 < t < 19$  s). This regime corresponds to the solid-body rotation of the granular packing with the angular velocity  $\Omega$  previous to the avalanche. At  $t \simeq 19$  s, the grains start flowing, and  $\theta$  decreases suddenly. After a short transient regime ( $19 < t < 30$  s),  $\theta$  reaches a constant value which corresponds to the continuous avalanching of the granular layer ( $D = 100 \mu\text{m}$ , water + HCl, pH = 1,  $\tau = 120$  s).

glass, Matrasur corp.) having three different typical diameters ( $D = 57 \pm 7$ ,  $112 \pm 12$ , and  $225 \pm 25 \mu\text{m}$ ). As the diameter of the cylinder is at least 10 times the typical diameter of the grains, we expect it to have little or no effect on the avalanche angle [17].

In order to measure the avalanche angle  $\theta_a(\tau)$  for a given resting time  $\tau$ , we first renew the sample by rotating the cylinder at angular velocity  $\Omega = 8$  deg/s for 240 s (5 rotations), and then set  $\Omega$  to 0. We then obtain systematically a smooth free surface of the granular packing that makes a well-defined angle  $\theta_i$  with the horizontal plane [18]. We wait for a time  $\tau$ , set  $\Omega$  to a small value  $\simeq 0.5$  deg/s, and record the angle  $\theta$  as a function of time  $t$  (Fig. 2). We define  $\theta_a(\tau)$  as the maximum value reached



**Fig. 3.** Angle of avalanche  $\theta_a$  as a function of resting time  $\tau$  for glass beads immersed in various fluids. The curves are shifted for convenience but plotted with the same scale. The avalanche angle at zero resting time is indicated for each curve. The size of the markers corresponds to the error bars ( $D = 100 \mu\text{m}$ ). For comparison, the top end curve corresponds to results obtained in the case of a dry granular material placed in a humid atmosphere (humidity 27%,  $D = 200\text{--}250 \mu\text{m}$ , from Bocquet *et al.* [11])

by  $\theta(t)$  previous to the first avalanche. The dispersion of the measurements is of about 2 degrees. We use the same sample to perform several measurements for various resting times  $\tau$ .

### 3 Experimental results and qualitative analysis

We measured the avalanche angle  $\theta_a(\tau)$  for glass beads immersed in various interstitial fluids for resting times  $\tau$  ranging from 10 seconds to 3 days. We worked with polar and non-polar fluids, having different viscosities. A significant increase (typically by 5 degrees) of  $\theta_a(\tau)$  with  $\tau$  is observed when the glass beads are immersed in water whereas no change is observed in the other immersing fluids (Fig. 3). The experimental results show clearly that ageing of the avalanche angle  $\theta_a$  in immersed granular materials depends on the physical and/or chemical properties of the immersing fluid. In the following paragraphs, we discuss in detail the relevance of the different phenomena that could lead to the observed ageing of the avalanche angle in water.

#### 3.1 Compaction of the granular material

The increase of the avalanche angle could result from a change in the compacity of the material with time  $\tau$ . The

angle of avalanche would be larger for larger compacity. The presence of the interstitial fluid has been shown to influence the compaction process; A larger viscosity of the interstitial fluid slows the compaction process, and increases, because of lubrication, the final compacity of immersed granular materials submitted to tapping [19]. Nevertheless, a change in the compacity of the granular packing during the waiting time  $\tau$  is not relevant for explaining our experimental results. Indeed, no ageing of the granular material is measured in fluids less (acetone,  $\eta = 0.3 \times 10^{-6} \text{ m}^2\text{s}^{-1}$ ) or more (ethanol,  $\eta = 1.2 \times 10^{-6} \text{ m}^2\text{s}^{-1}$ ) viscous than water ( $\eta = 0.9 \times 10^{-6} \text{ m}^2\text{s}^{-1}$ ). If compaction limited by the viscous fluid plays a role in the ageing process, the corresponding variations of the avalanche angle are not measurable in our experiments.

#### 3.2 Van der Waals interaction

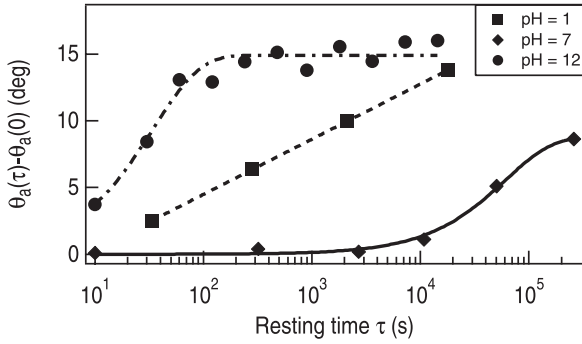
Van der Waals interaction between the grains at the contact points could be a source of cohesion in the granular packing. A change in this interaction with the resting time  $\tau$  then could induce ageing of the avalanche angle. Ageing is observed in water (Hamaker constant  $A \simeq 1.2 \times 10^{-20} \text{ J}$ ) while no ageing is measured in the other fluids. We estimated the Hamaker constants for the interaction of glass beads in the different fluids used in the experiments (ethanol and acetone:  $A \simeq 8.2 \times 10^{-21} \text{ J}$ , hexane:  $A \simeq 5.7 \times 10^{-21} \text{ J}$ , cyclohexane:  $A \simeq 2.5 \times 10^{-21} \text{ J}$ ) [20]. The interaction should be only decreased by 30% in ethanol and acetone, for instance. Nevertheless, no ageing is observed for glass beads immersed in these fluids (Fig. 3). As a consequence, Van der Waals interaction cannot be responsible for the observed increase of the avalanche angle when the glass beads are immersed in water.

#### 3.3 Electrostatics

Electrostatic interaction between grains could also be a source of cohesion in the granular system. Nevertheless, we observe an important increase of the avalanche angle even if electrostatic interactions are screened by ions (Fig. 3); In a 4M-NaCl solution, the Debye length ( $\sim 2 \text{ \AA}$ ) is much smaller than the typical surface roughness of the grains ( $\sim 10 \text{ nm}$ ), and the electric conductivity of the solution ensures that the grains are not charged. It is then clear that electrostatic interactions are not responsible for the observed ageing of the material.

#### 3.4 Chemical reactions

The cohesion in the system could result from chemical reactions occurring at the glass-water interface. In order to check this assumption, we performed experiments with glass beads immersed in water at various pH (Fig. 4); Ageing is found to be faster in acid (pH = 1) or basic (pH = 12) than in pure water (pH = 7). Indeed, the avalanche



**Fig. 4.** Angle of avalanche  $\theta_a$  as a function of resting time  $\tau$  for water at various pH. Squares: pH = 1, diamonds: pH = 7, circles: pH = 12 ( $D = 100 \mu\text{m}$ ).

angle is increased by 5 degrees in less than 20 seconds at pH = 12, after 2 minutes at pH = 1, and in about 10 hours at pH = 7. Moreover, the angle of avalanche  $\theta_a$  does not depend qualitatively in the same way on the resting time  $\tau$  when the pH is changed: for pH  $\simeq 7$ ,  $\theta_a$  depends almost exponentially on time whereas  $\theta_a$  increases logarithmically with time for pH  $\simeq 1$ . Hydrodynamics clearly does not control the observed ageing of immersed granular materials as a change in the pH cannot account for changes in the hydrodynamical properties of the system. Thus, as shown in the following section, the dynamics of the ageing observed for glass beads in water is only governed by the chemical kinetics.

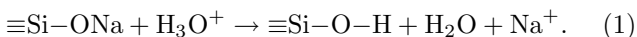
## 4 The case of glass beads immersed in water

It is well known that chemical reactions can occur at the glass-water interface. They can lead to adhesion forces between the grains, and hence to an increase of the avalanche angle with time. In Section 4.1, we summarize the chemical reactions involved. In Sections 4.2 and 4.3, we present respectively a theoretical determination of the cohesion force induced in the system and a kinetic model. In the last Section 4.4, we discuss the experimental findings in the framework of our model.

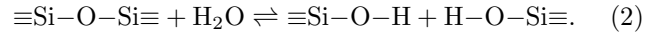
### 4.1 Chemistry of glass in water

The chemical reactions that occur at the glass surface in contact with water have been widely studied because of their practical importance. The surface chemistry of the glass in contact with water is quite complicated. The authors are not specialists in the field, and give below only the basic features that can be of some interest for the theoretical description of the experimental results given above. The chemical reactions that occur at the glass-water interface can be summarized as follows [21, 22]:

(i) Exchange of ions  $\text{Na}^+$  (or  $\text{K}^+$ ) with  $\text{H}_3\text{O}^+$  from solution in a region close to the free surface:



(ii) Formation ( $\rightarrow$ ) of silanols  $\text{Si}-\text{OH}$  resulting from breaking of  $\text{Si}-\text{O}-\text{Si}$  bonds at the water-glass interface, and repolymerization ( $\leftarrow$ ) of a  $\text{Si}-\text{O}_2$ -rich layer on the surface:



Dealkalization of the surface (Eq. (1)) dominates for solutions of  $\text{pH} < 9$ , whereas network dissolution by hydroxyl ions (Eq. (2) ( $\rightarrow$ )) dominates for solutions of  $\text{pH} > 10$ . Both processes are limited by diffusion of  $\text{H}_2\text{O}$  and  $\text{H}_3\text{O}^+$  within the repolymerized (corroded) layer. The corrosion rate decreases drastically when the thickness  $w$  of the layer increases. As a consequence,  $w$  reaches an asymptotic value  $w_\infty$  within a few hours (corrosion rate is only drastically decreased, but still occurs. Evolution of the system would be noticeable only over months).

Repolymerization occurs within the network that forms the corroded layer. The bonding of the silanol groups (Eq. (2) ( $\leftarrow$ )) is an activated process and the distances between the silanol groups are randomly distributed within the corroded layer. In the following, we will assume that the number of ( $\text{Si}-\text{O}-\text{Si}$ )-bonds per unit volume, and hence the strength  $\Phi$  of the corroded layer, increase logarithmically with time [23].

It is important to notice that the corrosion leads to a significant swelling of the corroded layer. The relative change in volume of the corroded region is of about 5%.

### 4.2 The resulting cohesion force

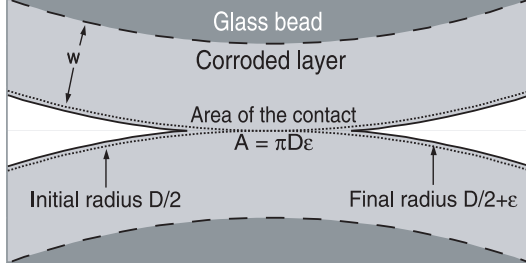
We can depict the ageing of glass packings in water as follows: the glass beads are initially rigid and in solid contact. When the beads are immersed in water, chemical reactions occur leading to the formation of corroded layers (thickness  $w$ ). The resulting swelling  $\epsilon$  (proportional to  $w$ ) of the material leads to the formation of bridges between the grains (Fig. 5) [24]: there results an adhesion force  $f_{ad}$  between two grains, which is proportional to the number of ( $\text{Si}-\text{O}-\text{Si}$ )-bonds in the contact plane, and hence to the surface area  $A = \pi D \epsilon$  times the strength  $\Phi$  of the repolymerized material. As the number of contacts between grains per unit volume is proportional to  $1/D^3$ , the adhesion force per unit volume  $F_{ad}$  scales like  $\Phi \epsilon / D^2$ . The bulk force  $F_{ad}$  increases the avalanche angle  $\theta_a$  according to:

$$\tan \theta_a = \left(1 + \frac{F_{ad}}{\rho g \cos \theta_a}\right) \tan \theta_a(0) \quad (3)$$

where  $\rho$  is the density of the grains,  $g$  the gravity constant, and  $\theta_a(0)$  the avalanche angle for  $F_{ad} = 0$  [16]. Linearization of equation (3) leads to  $\theta_a - \theta_a(0) = (\sin \theta_a(0) / \rho g) F_{ad}$ . The asymptotic increase of the avalanche angle  $\theta_a(\infty) - \theta_a(0)$ , obtained when the asymptotic thickness  $w_\infty$  of the corroded layer is reached, is then expected to scale like  $1/D^2$  where  $D$  is the typical diameter of the grains.

### 4.3 Chemical kinetics

The simplest way to describe the kinetics of corrosion, that accounts for the asymptotic thickness  $w_\infty$  of the



**Fig. 5.** The contact region between two grains. The swelling of the repolymerized layer leads to the formation of a bridge between the grains. The adhesion force is proportional to the surface area  $A = \pi D \epsilon$ .

corroded layer, consists in writing the rate of corrosion  $r = r_0(1 - w/w_\infty)$  (this local equation, written at the glass-water interface, does not depend on the diameter of the grains  $D$ , but the initial rate of corrosion  $r_0$  depends on the pH (Eq. (1)). The rate  $r$  corresponds to the volume of corroded layer that forms per unit volume and unit time. It can be written, taking into account the geometry of the granular packing,

$$r = r_0(1 - w/w_\infty) = n \frac{dV_c}{d\tau} = \frac{6C}{D} \frac{dw}{d\tau}. \quad (4)$$

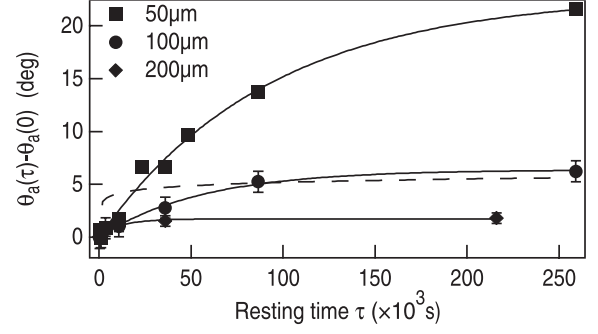
Indeed, the number  $n$  of grains per unit volume writes  $n = 6C/\pi D^3$ , where  $C$  is the compacity of the granular packing, and the volume  $V_c$  of corroded layer around a single grain  $V_c = \pi D^2 w$ . The thickness  $w$  of the corroded layer, and hence  $\epsilon$ , increase exponentially with the resting time  $\tau$ . The characteristic time of ageing writes  $\tau_a = 6Cw_\infty/Dr_0$ , and hence scales like  $1/D$ .

#### 4.4 Discussion

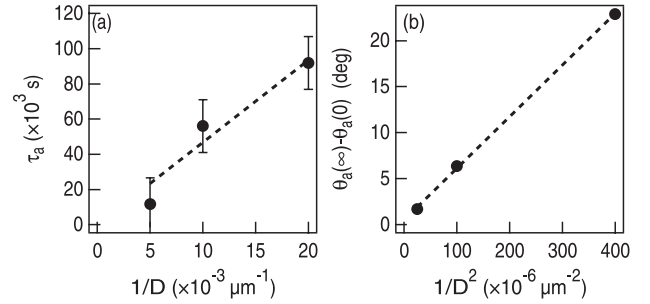
The adhesion force  $F_{ad}$  theoretically scales like  $\Phi\epsilon/D^2$ . Thus, the increase of the avalanche angle can be due either to the strengthening of the corroded layer ( $\Phi$  depends logarithmically on time) or to the increase of the area of contact  $A$  ( $\epsilon$  increases exponentially with time). The strengthening of the corroded layer is expected not to depend significantly on the pH, while the corrosion rate, by contrast, should be strongly dependent on the concentration of the protons. The dynamics of ageing is then expected to depend even qualitatively on the pH.

##### 4.4.1 Effect of the bead size in pure water (pH $\simeq 7$ )

For pH = 7,  $\theta_a(\tau)$  increases almost exponentially with the resting time  $\tau$  (Fig. 6). The characteristic time  $\tau_a$  is found to scale like  $1/D$  (Fig. 7a), and the asymptotic variation  $\theta_a(\infty) - \theta_a(0)$  scales like  $1/D^2$  (Fig. 7b). The quantitative variation of the avalanche angle is compatible with the typical adhesion force already estimated in former experiments [25]. In the framework of our theoretical description, these experimental scalings are in agreement with an ageing process limited by the formation of the corroded



**Fig. 6.** Angle of avalanche  $\theta_a$  as a function of resting time  $\tau$  for various beads diameters (water, pH = 7). Dashed line: water + HCl, pH = 1,  $D = 100 \mu\text{m}$  (old sample).



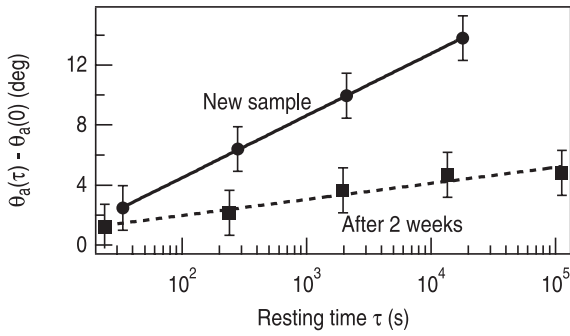
**Fig. 7.** (a) Characteristic time  $\tau_a$ , and (b) asymptotic variation of the angle of avalanche  $\theta_a(\infty) - \theta_a(0)$  as functions of the beads diameter  $D$  (water, pH = 7).

layer, provided that the strength  $\phi$  of the corroded layer is a slow varying quantity. In order to validate this last assumption, the evolution of  $\theta_a(\tau)$  for water pH = 1 is given in Figure 6. A sample that remained for more than two weeks in solution previous to measurement is used in this experiment (see next section). The asymptotic thickness of the corroded layer is then reached previous to the experiment, and strengthening is only due to changes in  $\phi$ . The increase of the avalanche angle due to strengthening of the corroded layer is hardly noticeable on this linear plot for  $\tau > 2 \times 10^4 \text{s}$ , and we can hence consider that  $\phi$  is a slow varying quantity.

##### 4.4.2 Ageing in acid water (HCl, pH $\simeq 1$ )

For pH = 1,  $\theta_a(\tau)$  is found to increase almost logarithmically with  $\tau$  (Fig. 4). For such a pH, according to equation (1), the rate of corrosion is expected to be much larger as the concentration of protons is  $10^6$  times larger than for pH = 7. Corrosion takes place very quickly, and the increase of  $F_{ad}$  is mainly limited by the variation of  $\Phi$ . As  $F_{ad} \propto A\Phi$ , the slope  $d\theta_a/d\log(\tau)$  is expected to be proportional to the asymptotic area of the contacts  $A$ .

In order to validate this conclusion, we performed one additional experiment with grains that remained for more than two weeks in water at pH = 1 previous to measurements (Fig. 8). In the case of the new sample, the area of contacts  $A$  reaches quickly its asymptotic value  $A_\infty$  corresponding to the asymptotic thickness  $w_\infty$  of the corroded layer. In the case of the old sample, the area of



**Fig. 8.** Angle of avalanche  $\theta_a$  as a function of resting time  $\tau$ . Circles: new sample, squares: the glass beads remained for more than two weeks in solution before measurement ( $D = 100 \mu\text{m}$ , water + HCl, pH = 1).

the contacts results only from crushing of the already corroded layer, and  $A$  is smaller than  $A_\infty$ . It appears clearly that the slope  $d\theta_a/d\log(\tau)$  is smaller in this latter case, in agreement with our theoretical prediction. Only chemical effects can account for such a behavior of the system, which depends on the time the beads spent in solution before measurements.

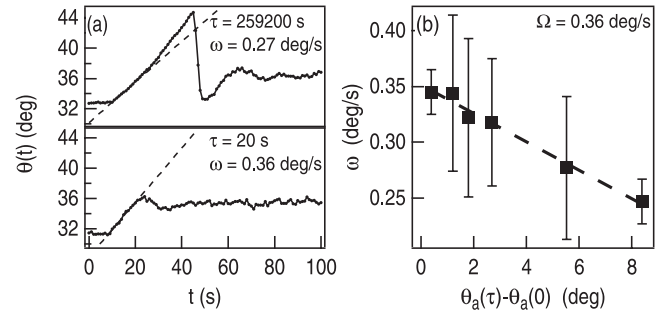
#### 4.4.3 Ageing in basic water (NaOH, pH $\simeq$ 12)

For pH = 12, the ageing is fast (Fig. 4). For such a high pH, the surface of the glass beads is attacked according to the reversible chemical reaction (Eq. (2)). The theoretical description given above cannot account for the experimental results pH > 10 as the kinetics model stands only for solutions at pH < 9. We do not discuss the kinetics in this case, but give the experimental results only in order to show once more that the ageing process strongly depends on the pH. They confirm again that ageing originates from chemical reactions.

## 5 Conclusion

Measurements of avalanche angles performed in a rotating drum prove that ageing of granular materials immersed in a fluid, not submitted to any additional external sollicitation (mechanical vibrations, temperature cycles, etc...), only originates, when present, from chemical reactions. Neither hydrodynamics nor electrostatic interactions play a significant role in the ageing process. The surface chemistry of glass is responsible for the ageing of the mechanical properties for glass beads immersed in water.

This work deserves to be extended by a detailed study of the relation between volumic adhesion force, frictional coefficients, and avalanche angles in granular materials. Indeed, we observe that the initial slope  $\omega$  of the curve  $\theta(t)$  depends on the resting time  $\tau$  (Fig. 9a). After ageing, the angular velocity of the free surface  $\omega = \dot{\theta}(t)$  is smaller than the driving angular velocity  $\Omega$ , proving that the system rearranges slightly without flow (creep) before the



**Fig. 9.** (a) angle  $\theta(t)$  as a function of time  $t$  for two different resting times  $\tau$  (Dashed lines: initial slopes), and (b) initial angular velocity  $\omega$  of the free surface as a function of the avalanche angle  $\theta_a$  (water + HCl, pH = 1,  $\Omega = 0.36 \text{ deg/s}$ ).

avalanche. The more difficult the avalanching is, the easier is the creep (Fig. 9b). Corrosion makes the grains stick to each other (avoiding flow), but softens the surface of the grains (allowing creep). One of us (JCG) previously observed creep during the waiting time leading to an increase of the static frictional coefficient in friction experiments [12]. The extension of the present work, already in progress, associating direct force measurement, friction experiment, and measurement of avalanche angles will be the subject of a further publication.

The authors are convinced that the experimental results presented in this article provide important clues for the understanding of the often observed ageing of immersed granular matter, and that they are also relevant in the general context of material ageing.

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18. At large angular velocity  $\Omega$  continuous avalanching of the granular material is observed as the viscosity of the fluid is low, and the diameter of the cylinder small (S. Courrech du Pont and P. Gondret, *private communication*). A sudden stopping of the rotation always leads to the same angle  $\theta_i$  between the free surface and the horizontal plane. The angle  $\theta_i$  depends slightly on the diameter of the glass beads and on the interstitial fluid (for instance,  $\theta_i = 21, 26,$  and  $28 \pm 1$  degrees for  $D = 57, 112,$  and  $225 \mu\text{m}$  in water). Nevertheless,  $\theta_i$  is well-defined for a given sample
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24. A microphotograph of a solid bridge between two grains can be found in Ref. [14]. Olivi-Tran *et al* observed the formation of solid bridges within the liquid bridges formed after capillary condensation. In this case, the size of the solid bridge is limited by the size of the liquid bridge. When the material is immersed in water, the size of the solid bridge is limited by the thickness of the corroded layer
25. From  $\theta_a(\infty) - \theta_a(0) \simeq 5 \text{ deg}$ , we estimate  $f_{ad} \sim 10^{-9} \text{ N}$  for  $D = 100 \mu\text{m}$ . One can estimate, from direct force measurements by Atkins and coll.,  $f_{ad} \sim 10^{-10} \text{ N}$  for colloidal silica particles ( $D = 50 \text{ nm}$ ,  $\text{pH} = 2$ ). D. Atkins, P. Kekicheff, O. Spalla, *J. Colloid Interface Sci.* **188**, 234 (1997)