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Rheometric study for elaborating transparent model of cohesive sediments for local investigations of erosion behaviour

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Abstract:
In this paper, the behaviour of cohesive sediments is studied using rheometric experiments. Results have shown that the treatment of natural sediments using gamma irradiation leads to an important reduction of their original mechanical strengths. The dynamics of evolution of sediment structure during ageing gives rise to a characteristic time written as a power law of the rest period. The observation of the solid/liquid transition (similar process to the erosion of sedimentary bed) allows us to obtain an exponential law of the inverse of applied shear stress. It is very unlikely that these dynamics are affected by gamma irradiation treatment. For better understanding of the phenomena regarding the erosion process, it is suggested to study these dynamics locally by using PTV and LIF techniques. These optical techniques require the use of transparent model materials having very similar rheological properties to those of natural sediments. Two yield stress fluids (gels) that have been specifically prepared using carbopol and laponite are proposed within this framework.

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1. Introduction and work scope

In ports and estuaries, sediments accumulate in some regions and are eroded elsewhere. The long term management of these areas demands an understanding of the erosion and transport behaviours of sediments which are usually cohesive. Describing these erosion properties requires either in situ or laboratory experiments for evaluating the critical shear stress. Different techniques and experiments conducted so far have revealed that this task is complicated. TOLHURST *et al*. (2000) state that the erosion process is not universal since there exist two types of erosion (surface and mass erosion) which could happen consecutively or independently. An essential characteristic related to these erosion modes is the onset of a break-up process. The same authors also underline the importance of the amplitude and the duration of increments of the mechanical solicitations applied to a sedimentary bed. Furthermore, the identification of the starting point of erosion process following these solicitations is very delicate and may lead to consider different threshold levels according to the quantity of matter in suspension involved (e.g. detectability of the amount of sediment accumulated in suspension).

From the physical point of view, the erosion phenomenon corresponds to a transition between the solid and the liquid state which requires an accurate characterization of the dynamics of mechanical behaviour over a large scale of deformation. The rheometry can fulfil these demands in the framework of mechanical solicitations in simple shear obtained by confining studied material between two tools. The relative ease in performing tests allows us to investigate the impact of different factors such as concentration, nature and size of mineralogical components, and organic and polymeric components, on the mechanical behaviour.

Experimental rheological studies have revealed the complex and time dependent character of sediments which depends on numerous factors (concentration of colloidal and coarse particles, content of organic material, biological activity, etc.). The thixotropic property and the existence of yield stress have been also reported, and a better understanding of these properties has been possible over the years thanks to local approaches with different systems exhibiting the same properties. Such local studies indicate the coexistence of fluid and solid regions within the sample that develop spatially and temporally according to material conditions (RAYNAUD *et al.*, 2002; OVARLEZ *et al.*, 2009). Henceforth, observations related to the solid/liquid and the liquid/solid transition linked to the concept of yield stress and flow behaviour, are directly dependent upon the conditions of mechanical solicitations (distribution, time and increasing rate) (COUSSOT *et al.*, 2002; UHLHERR *et al.*, 2005).

This view of the erosion phenomenon, like the transition between two physical states, poses many legal questions. With regards to the difference in the nature of mechanical solicitation between erosion and rheometric tests, are there any possible links enabling us to predict the erosion behaviour from just “simple” rheometric tests? A specific study can be conducted in this way, but it demands, on the one hand, to eliminate time dependent character of sediment due to biological component, and on the other hand, to simultaneously obtain macroscopic information characterizing the dynamics and local information of material structure which will arrange in a heterogeneous manner. Which techniques could be employed then?

The techniques like Nuclear Magnetic Resonance (NMR), Particle Image Velocimetry (PIV), Particle Tracking Velocimetry (PTV), Laser or Acoustic Doppler Velocimetry (LDV or ADV) can respond to these objectives. They are different from each other by their intrusive characters (with or without seeding tracers), spatial and temporal resolutions, as well as requirements about fluid properties (optical transparency, homogeneity). The technique using Laser-Induced Fluorescence (LIF) may also provide qualitative information about the diffusion and deformation phenomena within material. The choice of optical based techniques implies consequently the use of transparent model materials. This constraint offers the possibility of preparing materials with tuned, well controlled, and stable properties. It only remains therefore to define the nature of the properties to be controlled.

In this paper, we focus on two crucial rheological parameters, yield stress and thixotropy, of natural cohesive sediments by performing creep tests for characterizing dynamics feature that may not be identified accurately from only classical flow tests. In addition, this sediment will be treated by gamma irradiation in order to underline the importance of biological component. The obtained results constitute a reference for fabricating model materials such as those names suggested previously by POUV *et al.* (2010). Thus, we will detail the preparation protocol of two transparent model materials having similar behaviour to that of natural cohesive sediments. These fluids (a carbopol gel and a mixed laponite-carbopol gel) are characterized by very close yield stress values but very different thixotropic feature which is a key parameter rarely taken into account in erosion study. It should be noted that carbopol (polymer) and laponite (synthetic hectorite clay) are amongst popular additives which are largely used and studied in the field of rheology. The description of these main products can be found in the paper of PIAU (2007) and the thesis of MONGONDRY (2003).

2. Methodology

2.1 Samples and characteristics of natural sediment

The sample of marine sediment was collected in February, 2008, from Cherbourg harbour (Lat. 49°40'05.00"N, Long. 01°36'11.00"W) near the sea route of the East Pass. The sample was distributed equally in four hermetic kegs of 6 l which were transported on the same day to the site of Ionisos Company in Sablé sur Sarthe (72). The gamma irradiation treatment (dose of 35 KGy at the periphery) was applied to two kegs only. The total duration between the shipment and the return of the kegs to the laboratory was 3 days. This initial treatment allows us to obtain, for the same storage conditions, one sample characterized by normal biological activity (increase/decrease of bacteria, production of extracellular substances…) and another characterised by the fact that both bacterial populations and polymeric substances are damaged or even destroyed. Afterwards, in this paper, they will be called natural and irradiated samples, respectively.

The last step of the preparation consists in a sieving procedure executed in three steps using the normalized sieves with an ultimate cut-off size of 125 µm. The pH values of both kinds of sediment are close to 7.5. The mass concentration of solid particles was determined from three samples for each type of treatment ("natural" and "irradiated"), by considering the weights measured before and after oven-drying (24 h at 70°C). We get the values of 36.1±1.7% and 36.0±0.1% for the natural and irradiated sediments, respectively. On average, both systems have the same concentration. However, variability is higher for the natural system. The sediment dry density of each sample was measured by helium pycnometry (Accupyc1330, Micromeritics). We find that all the samples have a mean density of 2.58 g/cm^3 with very little variability (0.3%). The analysis allows us to judge the homogeneity of material for whichever sample studied. No further analysis has been made since the main objective of rheometric tests is not to develop a correlation between mechanical behaviour and material components.

2.2 Preparation of model sediments

The preparation of carbopol gel is started by mixing the presumed weight of carbopol powder (0.3 wt. %) (Ultrez 21, Noveon) in ultrapure water (14.45 wt. %) (Chromasolv 34877, Sigma Aldrich) using a magnetic stirrer (at 500 rpm for 20 min). The appropriate quantity of glycerol (85 wt. %) (24397.236, VWR International) is then added into the mixture and stirred at 1100 rpm for 20 min. Then, a rest time of 20 min allows carbopol particles to hydrate and swell freely in the solution. The last step of the preparation consists in neutralizing the system by adding a small amount of triethanolamine (0.25 wt. %) (Fluka 90280, Sigma Aldrich). During the neutralization, the mixture is stirred manually by using a metal spatula until the gel becomes visibly homogeneous (about 20 to 30 min).

The values of pH and conductivity of the gel, measured after 24 hours, are around 7.13 and 4.90 µs/cm, respectively. In order to limit ageing effect and obtain reproducible results, the fabricated gel is homogenized using an Ultra-turrax tool (at 11000 rpm for 2 min) before being distributed in different tubes for centrifugal process (at 5000 rpm for 20 min) that leads to completely removed air bubbles. Finally, we achieve a transparent gel with refractive index of 1.446 at 17°C (measured using a Euromex refractometer) and density of 1.223 g/cm^3.

Laponite-carbopol gel is obtained by a mix of 45.9 wt. % of laponite suspension at 1 wt. % (2 days old), 39.1 wt. % of carbopol gel at 0.3 wt. % (2 days old) and 15 % of glycerol. The preparation of carbopol gel follows the same procedure as described above but without the final stirring using the Ultra-turrax. The preparation protocol of laponite dispersion at 1 wt. % involves two steps to be conducted during two straight days. During the first day, two mixtures with the same weight are produced: a direct dissolution of NaCl (S/3160/53, Fisher Scientific) at 2 10^{-3} mol/l in ultrapure water, and a dispersion of laponite powder (RD, Rockwood Additives) with concentration of 2 wt. % in ultrapure water. The latter dispersion is subjected to 11000 rpm for 15 min by using the Ultra-turrax. During the second day, both systems are entirely mixed together at 11000 rpm for 15 min by means of the same homogenizer. This is a laponite suspension with a concentration of 1 wt. % and ionic strength of 10^{-3} mol/l. The mix between glycerol, laponite and carbopol suspensions is subjected to vigorous 11000 rpm for 2 min using Ultra-turrax. To finalize the preparation, a centrifugal process is operated at a speed of 5000 rpm during 3 min in order to get rid of air bubbles. Thus, we could obtain a transparent gel with refractive index of 1.392 and density of 1.153 g/cm^3.

2.3 Rheometric techniques

Rheometric tests are carried out using two controlled-stress rheometers: an AR1000 (TA Instruments) employed with a plate-plate geometry (40 mm in diameter) for natural sediments, and a Rheoscope1 (Thermo Fisher Scientific) employed with a plate-plate geometry (28 mm in diameter) for model sediments. The surfaces of both geometries are covered with a mesh of pyramidal tooth (base of 1 mm x 1 mm, height of 0.867 mm) directly machined within the material. These cleat geometries have similar characteristics to the one developed by NICKERSON & KORNFIELD (2005) which helps to overcome wall slip problems. Complementary studies have shown that this kind of geometry can also improve the homogeneity of shear fields over a much larger range of shear stress values (POUV *et al.*, 2009).

A gap of 2 mm, which is sufficient to deal with the size of particles present in the specimens of natural sediments, is set for the cleat geometry PP40. However, a gap of 1 mm is fixed for the cleat geometry PP28. In view of the cleat dimensions, the effective shear layer is bigger than the gap. The extra layer to be taken into account for correcting rheometric parameters values could be determined by following the calibration method proposed by NICKERSON & KORNFIELD (2005). In our case, we have to consider respectively the values of 165 µm and 108 µm for each plate of geometries PP40 and PP28.

3. Results of rheometric experiments

3.1 Flow behaviour

The flow tests investigated here consist, firstly, in putting the natural sediment in a relative state depending on the established protocol (preshear at 100 s^{-1} during 300 s followed by a rest of 300 s) owing to the thixotropic character, and secondly, by applying a set of solicitations with increasing and then decreasing amplitudes (total duration of 40 min). We control either shear stress (CSS) or shear rate (CSR) via a fixed stress loop. The flow curves of both natural and irradiated sediments obtained for these two solicitation modes (figure 1) prove clearly that the irradiated system is mechanically less resistant since the necessary levels of shear stress for reaching the flow state over the same interval of shear rate are systematically smaller. This assessment of mechanical biostabilization of sediment is in agreement with the description of biological effects presented by WINTERWERP & VAN KESTEREN (2004).

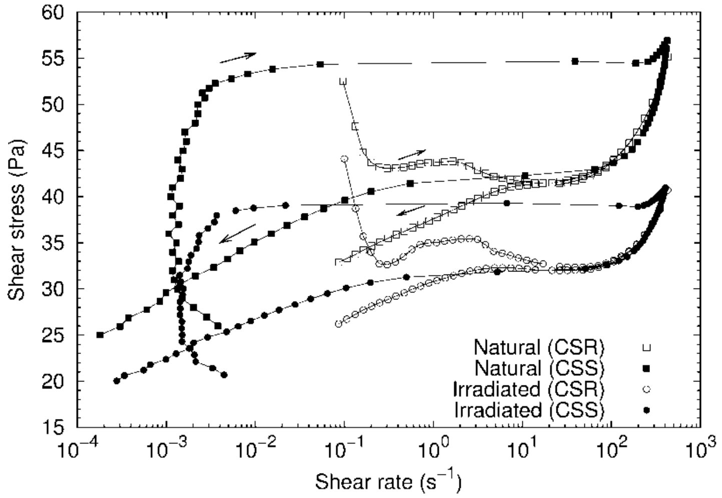

Figure 1. Controlled-shear-stress (CSS) and controlled-shear-rate (CSR) flow curves of natural (square) and irradiated sediments (circle). The arrows indicate the charge and discharge of the system.

The curves obtained with the CSS method present an early stage with little variation of shear rate. This regime underlines the solid character of sediment which deforms elastically under the effect of increasing shear stress. Above the critical stress value (about 52 Pa for natural sediment and 38 Pa for irradiated sediment), a brutal transition appears and is materialized by a quasi-plateau of shear stress which in fact corresponds to an instable flow. It has been suggested that flow stability is only reached beyond a critical shear rate value associated with a homogeneous state of fluid within the gap. It is important to note that the solid/liquid transition is characterized by a proper dynamic which implies that the critical stress corresponds to an observable change imposed by the characteristic time of experiment. Therefore, its value is bigger than the yield stress one which initiates the transition. At the end of the plateau, the flow becomes homogeneous and the shear rate is compatible with the level of imposed shear stress. From that point, both the shear rate and the shear stress increase according to a progressive break-up process of sediment structure. This state of broken structure endures, although the shear stress begins to decrease from the maximum value as we clearly see in the fact that the bottom curve stays below the upper one. As soon as the system reaches the value of critical shear rate (10 s^{-1} for natural sediment and 30 s^{-1} for irradiated sediment), a bifurcation towards the solid state takes place and the shear rates decrease rapidly.

Even though the curves obtained from the CSR experiments are different from the previous ones, they are fully coherent. For the charge stage, we can see a non monotonous evolution as long as the flow is unstable (coexistence of solid and sheared zones), the shear rate is lower than the critical one. For a developed stable flow characterized by a homogenous shear of material, the discharge curve superimposes on the one obtained from the CSS test for the same material. In this region, there are little differences between the upper and lower curves. This suggests that the structural break-up kinetics is rather slow and that only then will the whole gap be sheared in a homogenous way. Lastly, for the discharge curves, the bifurcation phenomenon appears again once the shear rate value becomes lower than the critical one. As controlling conditions differ, it gives rise to different distributions of stress within the sample, and thus to different curves.

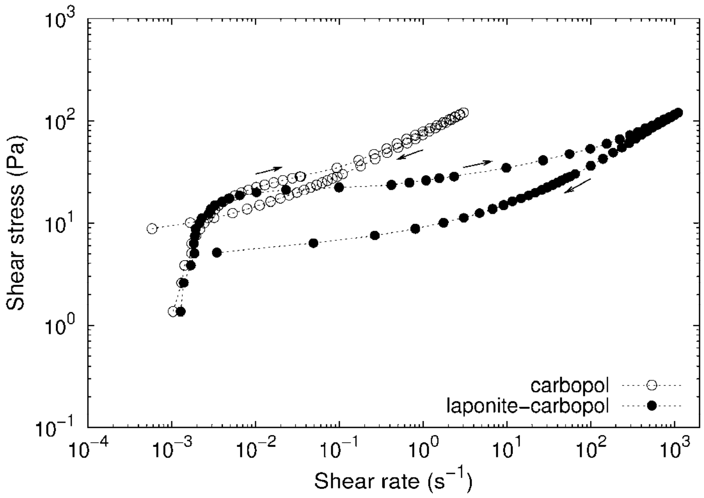

Figure 2. Controlled-shear-stress (CSS) flow curves of model sediments. The arrows indicate the charge and discharge of the system.

Regarding model materials, only CSS flow tests (preshear at ±1 s^{-1} during 90 s, rest time of 120 s, total duration of shear stress application of 60 min) are carried out. With these experimental conditions, we obtain similar behaviours compared to those of natural sediments reported previously. The flow curves depicted in figure 2 reveal that the two studied materials present the same elastic solid response. We can estimate that the yield stress values of these materials are very close to 16 Pa. The onset of the flow goes along very progressively for carbopol gel and more brutally for laponite-carbopol gel like in the case of natural sediments (steepness of the stress quasi-plateau). The critical shear rate value of carbopol gel (0.01 s^{-1}) is a lot smaller than the one for laponite-carbopol gel (2 s^{-1}). The difference between the upward and downward curves suggests that laponite-carbopol gel possesses a more pronounced thixotropic character. Therefore, it is the system which has the closest characteristics to the natural sediment used in this study.

3.2 Ageing dynamics

For evaluating the strength level of sediment achieved after a rest period *tw* following the conditioning step (300 s of pre-shear at 100 s^{-1} which is greater than the critical shear rate value), we perform some creep tests with the same shear stress value of 40.8 Pa for natural sediment and 28.8 Pa for irradiated sediment. These lower values compared to the critical stresses are selected with a view to approach at best the yield stress values which, however, will not be determined precisely. Four values of rest time *tw* are picked out (300, 900, 1800 and 3600 s). This time is classically viewed as an ageing stage leading to structural reorganization. Consequently, time origin is fixed after the pre-shear step. PHAM VAN BANG *et al.* (2007). This reorganization leads to an increase of yield stress level with rest time. It means that results of erosion tests become directly dependent on mechanical history of sediment (solicitation and rest). It is important not to confuse this ageing phenomenon corresponding to the recovery of material structure, with the evolution of properties throughout storage period because of biological and organic components. In fact, both phenomena exist, but with different time scales.

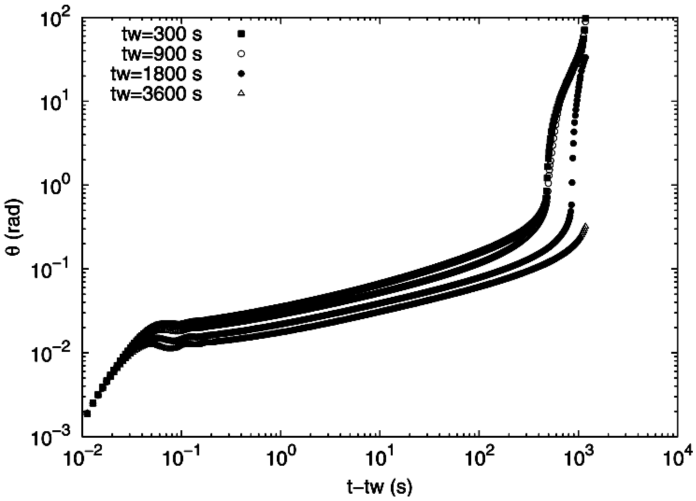

Figure 3. Creep curves (40.8 Pa) of natural sediment obtained for different rest time.

Figure 3 shows the temporal evolution of angular deviation (*e.g.* deformation) of the geometry in the case of natural sediment. A single linear response associated to elastic solid behaviour is recorded very early. It is followed by an oscillatory regime corresponding to the coupling between the viscoelastic response of the material and the inertia of the rheometer. After that, the evolution becomes continuous and we can observe a hierarchy of strain levels with the rest time. For longer periods, a complex transition towards a flow regime materialized by the bending of the curves is revealed. The longer the rest time lasts, the later the solid/liquid transition will appear.

For characterizing ageing dynamics, creep experiments are used to find out the periods of time taken to reach an arbitrary compliance level *J0* (*e.g.* deformation/stress ratio) . The log-log representation of the time values for *J0*=1.08 10^{-2} Pa^{-1} in function of the rest time reveals the same behaviour, described by a power law with an exponent μ equal to 1.43, for the natural and the irradiated sediment (figure 4.a). The exponent μ does not depend on *J0*. The physical origin of the ageing process remains to be determined. In any case, the presence of polymeric substances does not seem to be involved even if the mean molecular weight should be lower for the irradiated system.

The power law allows us to normalize the time and to obtain a master creep curve for smaller deviation angles (figure 4.b). For greater deviation angles, the dynamics of solid/liquid transition depend on structural network built up.

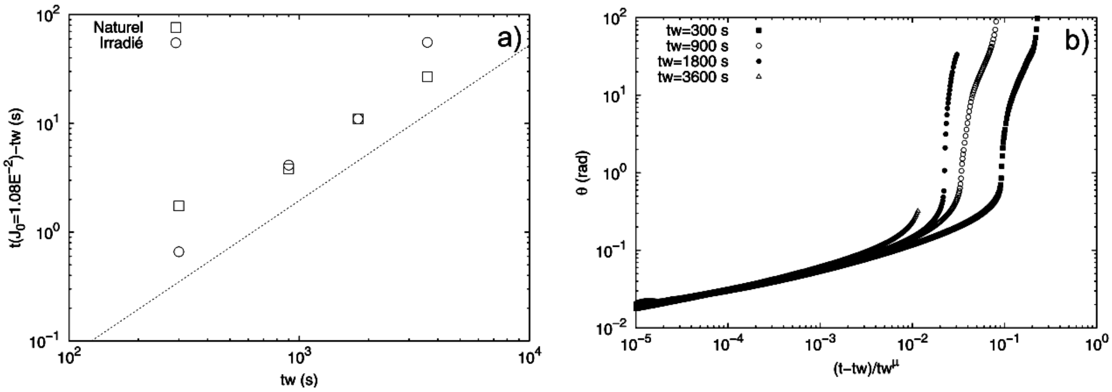

Figure 4. a. Evolution of time to reach the same compliance level for natural and irradiated sediments (the line materializes a slope of 1.43). b. Use of aging law for normalizing creep curves.

Figure 5 shows clearly that the two model sediments exhibit ageing phenomena. The exponents of both power laws (0.40 and 0.55 for carbopol and laponite-carbopol gels, respectively) are coherent with the results found in literature (0.91 for concentrated carbopol gel at 1.8 wt. % (TABUTEAU, 2005), between 0.5 and 0.7 for concentrated colloidal suspensions (DEREC *et al.*, 2000)). The ageing exponent of carbopol gel is smaller than the one for laponite-carbopol gel because its thixotropic character (rate of structural build-up) is much less pronounced. This result differs from the case of natural/irradiated sediments, for which the mechanical resistances are not the same but the thixotropic characters seem to be identical (same characteristic time). These features accentuate the importance of characterizing mechanical behaviour of material from the viewpoint of both thixotropy and yield stresses.

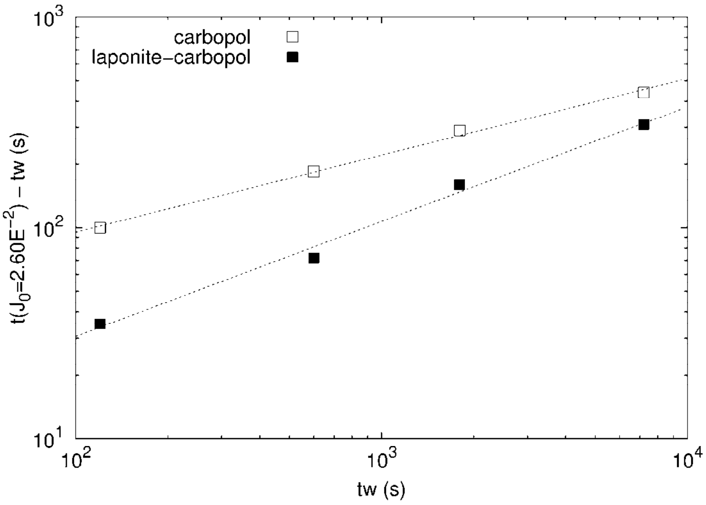

Figure 5. Values of time for J0 = 2.60 10^{-2} Pa^{-1} as a function of the rest time for model sediments. The two lines materialize respectively a slope of 0.40 and 0.55 for carbopol and laponite-carbopol gels.

3.3 Dynamics of solid/liquid transition

Starting from the same initial structural state obtained by conditioning the specimen of sediment by means of a pre-shear phase (5 min at 100 s^{-1}) followed by a rest of 5 min, we again investigate the behaviour of material through creep tests for different values of shear stress around the yield point. We analyze these experiments by focusing on the temporal evolution of shear rate and ignoring the first oscillatory response. For natural sediment, the evolution shown in figure 6.a presents an initial decreasing step with respect to a power law in function of time. When the applied shear stress and the time scale are large enough, the decrease is followed by a temporal minimum corresponding to a qualitative change of material behaviour. The temporal abscissa of the minimum increases once the shear stress decreases. The phase following the minimum indicates the flow regime of material.

This minimum is the only measurable objective quantity reported earlier by CATON & BARAVIAN (2007) for their study with other fluids and called transition time. That being said, the local arrangement of material within the gap until reaching transition time remains to be examined. The semi-logarithmic representation of the transition time versus the inverse of applied shear stress (figure 6.b) reveals similar exponential evolution $a.exp(b/\tau)$ for natural and irradiated sediments. One remarkable thing is that the same b value seems to characterize both materials. The values of a are different due to the fact that both materials do not have the same strength level. It is logical to obtain a shorter transition time for a weaker strength.

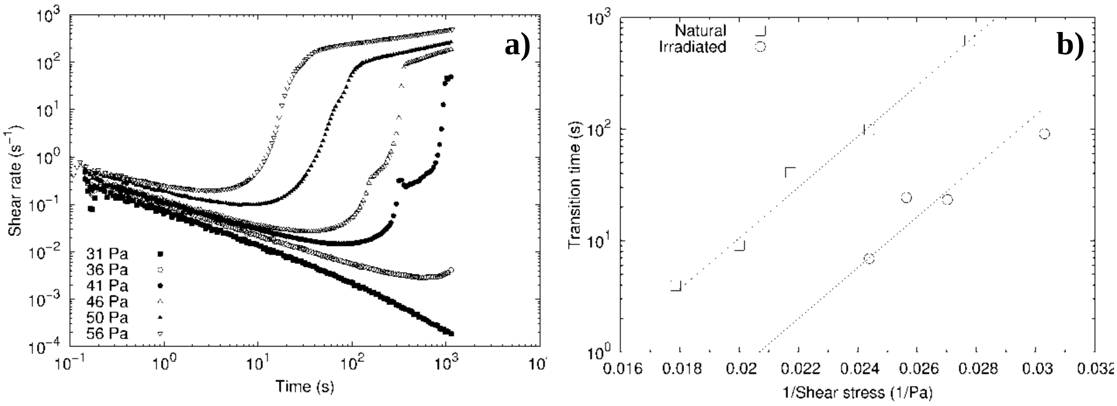

Figure 6. a. Evolution of shear rate obtained from creep tests (natural sediment). b. Evolution of temporal position of the minimum of shear rate for natural and irradiated sediments.

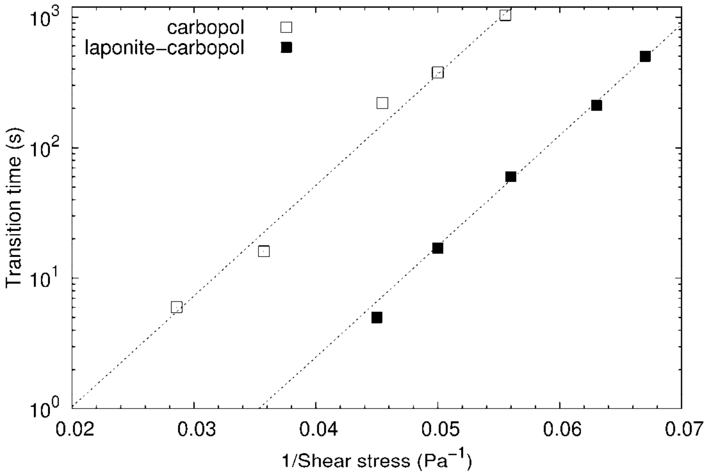

Figure 7. Evolution of temporal position of the minimum shear rate for model sediments.

Experiments with model sediments are carried out in a similar manner (*i.e.* pre-shear at ±1 s^{-1} for 90 s followed by a rest period of 120 s). Likewise, exponential evolutions of the transition time as a function of the inverse of shear stress are observed once again (figure 7). They are characterized by the same slope *b* but different coefficients *a* associated to the level of mechanical strength of each material. It is not surprising that the values of *a* and *b* for the model sediments differ from those of the real sediments since we understand that these two families do not have identical structural organization and interaction between their components.

These results have highlighted the importance of the observation time scale in the process of solid/liquid transition because of time dependent properties of sediment (thixotropy). This time scale is strongly connected to experimental conditions (stress level, initial state). The results have also pinpointed the characterization of the dynamics of deformation/break-up rather than the simple observation of material removal during erosion tests.

4. Conclusion

Flow tests are easily carried out and can give preliminary description of material properties if a large range of shear rate values is explored. The conducted experiments have shown that the studied sediments were fluids characterized by thixotropic thresholds and the gamma irradiation treatment leads to an important reduction of strength level of material since it damages stabilizing elements produced by microorganisms. Creep tests were also performed to investigate the dynamics of either structural evolution during ageing or solid/liquid transition. The former is characterized by a power law based on the rest time when the latter exhibits an exponential law of the transition time with the inverse of applied shear stress. The most remarkable fact is that these dynamics are not seemingly affected by the gamma irradiation treatment.

Taking these results into account, specific model fluids were developed and prepared by the use of ultrapure water, glycerol, carbopol (polymer) and laponite (synthetic clay of hectorite type). Basic characterization has proved that the two model fluids present close yield stress values and different thixotropic character. More importantly, it has been revealed that the behaviours of both fluids are very similar to those of natural sediments. The characterization of local dynamics for the same fluids is the aim of the upcoming study. This will be explored by tracking seeded particles in the case of rheometric and erosion flume tests. With regards to the current results, a particular interest will concern the response of material to different variations of applied shear stresses (e.g. gradual or instantaneous increase of stress values).

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