Determination of a rheophysical scheme of a sludge from diluted to divided consistency with a rotational rheometer

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ABSTRACT

Identifying sludge rheological behaviour over a wide concentration range is essential for optimizing the sludge management. This work looks at defining the rheological properties of a municipal sewage sludge on a range of total solids (TS) content of 2 to 44.2 wt. %. Results highlight that, contrary to the literature, solid-like properties/concentration relationship cannot be represented by a single power law model on the whole range of TS content but by a model combining two former laws. For TS < 6 wt. %, the behaviour is governed by viscous forces and follows a power law. In the region of TS > 14 wt. %, the behaviour is governed by frictional (granular)-like forces which are evidenced by the onset of dilatancy, and follows the Eilers law. In the range 6 < TS < 14 wt. %, the behaviour is in a transitional phase and governed by a competition between the viscous and the frictional contribution. Finally, a rheophysical scheme is proposed, in which the rheological behaviour is related to the consistency of sludge.

Keywords: Rheology, Sludge, Plastic, Yield stress, Friction, Viscous forces, Dilatancy, Rheophysical scheme

1. Introduction

Sludge production is increasing every year and is becoming a real challenge for the waste water treatment plants (WWTP) worldwide. Thermal drying constitutes an important step to reduce the volume and consequently improve the management of wastewater sludge. However, during drying process, the sludge passes through a highly cohesive phase, which is referred to the sticky (plastic) phase of sludge [1–11]. This can be especially challenging for agitated dryers where the sludge tends to stick to the dryer walls and the agitator. This alters its hydrodynamics and causes operational issues, thereby significantly reducing the dryer

capacity and increasing the difficulties to control the residence time and the homogeneity of the drying operation [4,9,12–14].

Several alternative methods are reported in the literature. Ferrasse et al. [14], Kudra [9] and Charlou [2] performed drying experiments in a stirred tank and considered the mechanical torque of the stirring paddles as a rheological signature. Ruiz and Wisniewski [15] adopted the Atterberg Limits, widely applied in civil engineering, to correlate the consistency of the sludge (liquid, plastic and divided granular-like) to its drying and shrinkage aptitudes. Peeters et al. [1] and Li et al. [16] adapted the shearing test used in powder engineering to identify the stickiness properties by mapping the adhesive and cohesive stresses of sludge at increasing TS content. Although these technics can be valuable to map the region where sludge exerts its sticky behaviour during the drying operation, they are not suitable for the purpose of determining the rheological properties.

From a general point of view, waste water professionals and academics agree on the existence of a plastic phase between 15 and 60 wt. % of total solids (TS) [1,2,9,14–20]. This sticky phase comes from rheological and textural modifications of sludge. Identifying sludge rheological characteristics during its sticky phase is therefore a must-do in industrial sludge dryers so as to optimize their hydrodynamic design and efficiency.

However, the literature focusing on concentrated sludge is quite poor [21–26]. This is mainly because carrying out relevant measurements is highly challenging as fractures occur, leading to underestimated or non-intrinsically rheological properties [2,24–26]. Indeed, as no local particles rearrangement is possible under shear, the pasty sludge fractures as in the case of cement, clay or plasticine pastes [27,28]. For instance, appearance of fractures in sludge sheared between parallel plates starts at around 14 wt. % TS according to Mouzaoui et al. [26]. Battistoni [22] and Hammadi et al. [29] succeeded in investigating the rheological properties of sludge having TS content up to 35.1 wt. % without fracturing problems, after the sludge had undergone a pre-treatment by sieving and intense pre-shear. Applying an intense pre-shear prior to the measurement is fairly common [30]. However, these pre-treatments not only irreversibly altered the composition but also the microstructure of the sludge and the moisture distribution [31]. It is very likely that the properties thus determined are not representative of the characteristics of the original sludge. An alternative to avoid fracturing in conventional rotational rheometers is to investigate the rheological properties on a low range of deformation only [24,25]. For larger deformation, the experimental results were poorly reproducible because of the very likely occurrence of fracture.

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Recently, Mouzaoui et al. [26,32] developed a specific procedure to systematically take into account fractures under shear experiments. The experimental procedure is based on the exact determination of the surface effectively sheared by applying a classical stress sweep with measurements at a reference strain value. The implementation of this procedure allowed the correct determination of viscoelastic characteristics from 14 to 45 wt. % TS. Experiments bear strong similarity with granular pastes. As the TS content increased, the existence of a normal force, certainly due to dilatancy, was highlighted. Dilatancy is a common characteristic of granular pastes and concentrated colloidal suspensions. Disordered dense packing of hard grains cannot undergo a shear without simultaneously expanding in the direction perpendicular to the shear plane [33,34]; hence the interest of simultaneously measuring the normal force and the tangential shear stress. The dilatancy appears beyond a certain critical concentration which corresponds to the formation of an infinite network of solid particles throughout the material, called jamming value or percolation threshold of frictional contacts. For example, this critical volume fraction is 42 % for a mixture of cement paste and polymers [35] and 38 % (corresponding to 65 wt. % TS) for a silicate paste flocculated calcium [34,36]. Near the percolation threshold, Mansoutre et al. [36] have shown that an additional contribution to the yield stress appears simultaneously with the significant increase of the dilatancy. This additional contribution has been attributed to the frictional energy dissipation by direct contacts between particles [34,36].

However, these results were only focused on the control of edge effects (fractures) occurring on pasty sludge under rheological measurements: they didn't investigate the impact of solid concentration on sludge rheological properties.

This work focuses on the study of the effect of solid concentration on rheological properties of sludge in order to understand how they evolve during drying processes, especially during the plastic phase. A sludge without any pre-treatment (sieving or pre-shear) is characterized on a wide range of solid contents by using a classical rotational rheometer, widely available. The whole of this study will make it possible to propose a rheophysical phase diagram in which the rheological behaviour is related to the consistency of sludge.

2. Material and methods

2.1. Material

Sludge is sampled at the wastewater treatment plant (WWTP) of Albi city (France). The dewatered digested sludge is obtained after centrifugation, its initial total solid (TS) content is 20 wt. % and the volatile solid (VS) content is of 63 % (of dry weight). Samples at different

TS contents are prepared in the laboratory by centrifugation (at 30 000 rpm or 108 800 g for duration ranging from 30 minutes to 24 h, at controlled temperature of 20°C) or by dilution (with addition of deionized water and smooth mixing using a spatula).

All samples are stored at 4°C to ensure no biological variability. Homogenization of diluted samples is checked after preparation. It is found that sludge can be considered stable after a period of 10 h as rheological characteristics did not evolve after this period of time (Figure 1). Thus, diluted samples are stored at 4°C for at least 10 h, to ensure reproducible measurements.



Figure 1. Stability of measurements of diluted sludge at 10 wt. % TS stored at 4°C. The rheological properties are constant 10 h after the preparation by dilution.

2.2. Composition and textural characterisation of sludge

The final TS content is determined by drying the wet sample in an oven at 105 °C for 24 h [37]. The volatile organic content is determined after igniting the dry solids at 550°C for 2 h [38].

To assess the consistency of the sludge (ie. its plastic or divided granular-like state), the plastic limit, TS_P, is quantified using standardized soil mechanics trials (Atterberg limits).

 TS_P corresponds to the TS content at which transitions between plastic and divided granularlike states occurs. Experimentally, it corresponds to the maximum concentration at which the sludge can be rolled into threads of 3 mm in diameter without breaking into pieces [39,40].

2.3. Rheological measurements

Rheological measurements are performed with a controlled stress rheometer RS600 instrument (HAAKE), piloted by RheoWin software, at room temperature. The torque range varies from 0.1 to 200 000 μ Nm.

To avoid wall slip, the experiments are carried out with serrated parallel plate geometry (radius 10 and 17.5 mm) at a constant gap of 2 mm. The upper plate is supplied with a sensor allowing to register the normal force F_n (detection range: 0.02-30 N).

Before each measurement, the sludge sample is subjected to a constant dynamic strain (γ =0.3 %) in the linear viscoelastic range (LVE) during 300 seconds to ensure reproducible results [26].

A sweep of shear stress τ (corresponding to γ =0.1 to 200 %) is performed at 1 Hz frequency. To avoid fracturing, the stress sweep is performed according to the experimental procedure of surface correction developed by Mouzaoui et al. [26,32]. This procedure consists of applying a stress sweep in successive steps of constant dynamic stress of increasing intensity. Prior each constant stress step, a step at a reference state defined by a constant dynamic rotational angle ϕ_{ref} is applied. The latter corresponds to a constant dynamic reference strain γ_{ref} =0.3 % chosen in the linear viscoelastic plateau (LVE), where, by definition, stress and strain are proportional. By applying this reference state, edge effects (fractures) lead to a decrease of the corresponding stress and complex modulus (stress and strain ratio). As the complex modulus must be identical at the reference state before and after fractures, the surface really sheared (S) can be calculated, allowing the correction of the stress τ and the corresponding strain γ .

All tests have been made in triplicate to evaluate the reproducibility of results.

3. Results and discussion

3.1. Sludge composition and textural characterization

Samples with TS content ranging between 2 and 44.2 wt. % have been obtained. The plastic limit characterising the transition between plastic state and divided granular-like state is around $TS_P=43$ wt. %.

As long as TS is lower than 14 wt. %, the surface profile of the sample looks like a cone when measuring tools of the rheometer are moved away, reflecting that the surface was fully sheared (no fracture). Then, for TS higher than 14 wt. %, the surface profile displays concentric circles and a plane surface in the center, reflecting the existence of fractures (Figure 2, right). Thus, the transitions between liquid and plastic states can be considered around $TS_L=14$ wt. %.



Figure 2. Surface profile of sludge samples between parallel plates at the end of experiments.

According to these measurements, the plastic state ranges between 14 and 43 wt. % TS. This result is consistent with the scientific literature. Indeed, ranges of 21-38 wt. % TS and 16-42 wt. % TS can be derived for sludge having a VS content of 53 % and 75 %, respectively [15,19].

3.2. Rheological parameters determination

Figure 3 presents the evolution of moduli (G' and G''), shear stress (τ) and normal stress (σ =F_n/S) as function of strain (γ) for a sludge with TS=20.6 wt. %. Data show that both moduli are constant and strain-independent below γ_{LVE} =0.5 %. G' starts to become strain-dependent above γ_{LVE} , pointing the end of the LVE region. When γ exceeds 3 %, the Payne-like effect [41] appears: G' decreases monotonically and G'' goes through a maximum before decreasing well. This highlights a significant disruption of sludge internal structure [42]. The Payne effect is a feature of soft glassy materials that is observed in variety of materials such as emulsions, gels, colloidal suspensions [43] and sludge [42,44]. Then, at higher strain (10-15 %), moduli decrease together until a crossover point at a strain around γ_y =160 % where the corresponding yield stress is equal to τ_y ~8.1 kPa. This point marks the transition from a viscoelastic solid behavior to a viscoelastic liquid one [42,45,46]. Beyond the crossover point, G'' becomes higher than G' as the strain increases, reflecting the viscous nature of the medium, as noticed by Mansoutre et al.[36].

Normal stress measurement clearly highlights 3 distinct zones: a decrease below a critical strain γ_c , then a jump of the normal stress (σ) before an abrupt fall beyond γ_f corresponding to the decrease of G'' (Figure 3). The jump is typical of dilatancy in granular pastes [34,36]. This behaviour has also been reported for sludge by Mouzaoui et al., [26].



Figure 3. Evolution of the moduli G' and G'', the shear stress τ and the normal stress σ as function of strain γ for a sludge at TS=20.6 wt. %.

3.3. Impact of concentration on rheological parameters

Figure 4 illustrates the dependence of the yield stress (τ_y) on TS. The critical concentration from which a yield stress can be detected is 2 wt. %. This value is in agreement with the existent literature [21,30,47–49]. The maximum shear stress that the RS600 rheometer can reach with a plate-plate geometry of 10 mm radius is $\tau_{max}=2.M_{max}/\pi.R^3 =127$ kPa. For TS>34.3 wt. %, the yield stress values exceed τ_{max} and measurements are no longer possible. Thus, the impact of TS on the yield stress will be limited to TS<34.3 wt. %.

The literature on sludge rheology suggests that the yield stress evolves following a power law model of (TS-TS₀), where TS₀ corresponds to the lowest concentration below which no yield stress is noticeable, 2 wt. % in our case [21,30,47–49]. However, it is not possible in our case to describe the experimental values by a single power law model over the full range of investigated concentrations (Figure 4). The evolution of the yield stress values highlights two regions: values increase slightly up to a critical concentration TS_c close to 6 wt. %, then, above this concentration, a change in curvature occurs suggesting the appearance of an additional contribution. The same macroscopic behaviour is reported in the literature of clay suspensions [50] and polymer/nanocomposites systems [51–55]. The critical concentration,

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where the change in curvature occurs, is defined as the rheological percolation threshold. Thus, parameters were described according to a first power law model up to the critical concentration, then above this concentration following a second power law model. Pignon et al. [50] have well described the change in rheological behaviour near the rheological percolation threshold, by light scattering measurements. They have shown that, below the rheological percolation threshold, the structure may be seen as a beam-type alignment of aggregates, leading to the formation of a mechanically weak fibrous structure. Beyond this critical concentration, the structure may be seen as a continuous and dense network of aggregates, which is mechanically more resistant. Hence, the first increase can be related to the viscous forces in the sludge (hydrodynamic effects of flocs and physico-chemical interactions), while the second increase can be attributed to the formation of a continuous network by physical contacts between components.



Figure 4. Evolution of the experimental τ_y (\circ) as function of TS. The straight line (—) is the usual model used to fit the sludge rheological parameters.

At TS higher than $TS_L=14$ wt. %, fractures are highlighted within the sludge during shearing tests (Figure 2) indicating that frictional interactions between solid compounds take place, similarly to what happens with granular pastes, such as concrete, fresh cement, flocculated calcium silicate paste and mixtures of sand-clay minerals [34–36,56,57]. These frictional effects induce dilatancy (Figure 5). The dilatancy appears only when the concentration is above 14 wt. % TS which may be considered as a jamming threshold, TS_j , like in granular pastes [34,36]. Thus, above $TS_j=TS_L=14$ wt. %, the sludge becomes a dilatant material. In a dilatant behaviour, the yield stress is no longer associated with viscous forces but rather with frictional contacts (hard interactions) between particles. This suggests that, above TS_j , sludge

joins systems dominated by contact forces, where the main mechanism of energy dissipation is frictional.



Figure 5. Evolution of the mean value of the normal stress jump with TS.

Based on the previous observations, three specific regimes involving different interaction mechanisms can be defined:

- Viscous regime for TS < TS_c=6 wt. %: sludge behaviour is governed by viscous forces (hydrodynamic effects of flocs and physicochemical interactions). TS_c is the rheological percolation threshold or the minimum solids concentration required to have physical contacts.
- Frictional (or granular)-like regime for $TS > TS_j=14$ wt. %: sludge behaviour is governed by frictional contacts which are evidenced by the onset of dilatancy.
- Transitional phase for $TS_c < TS < TS_j$ where no dilatancy is detected. This observation may be attributed to the fact that particles which are in jamming situation (physical contacts) are mainly deformable objects. The system is in a transitional phase between viscous and dilatant regime.

Below the rheological percolation threshold $TS_c=6\%$, parameters follow a power law model (Equation 1). Above TS_c , parameters follow a second power law model (Equation 2). When the concentration exceeds the jamming value $TS_j=14\%$, parameters can be described by Eilers law (Equation 3) that is currently used for granular suspensions of spherical particles [58–60]. These models are presented in Figure 6.

$$\tau_{y,v} = a (TS - TS_0)^n$$
 Equation 1

 $\tau_{y,transition} = p (TS - TS_c)^q$

$$\tau_{y,fr} = \tau_{y,0} \left(1 + \frac{1.25TS}{1 - \frac{TS}{TSm}} \right)^2$$
Equation 3

Equation 2

Where a, p, n, q, $\tau_{y,0}$, TS₀, TS_c and TS_m are the parameters of the models (Table 1). TS₀ is the critical concentration below which no yield stress is detected. $\tau_{y,0}$ corresponds to the suspending fluid yield stress. It is associated to the yield stress at TS₀. TS_m is the maximum packing solids concentration derived from Eilers equation. It corresponds to the maximum of solid particles that can be placed in a volume of fluid.

Table 1. Parameters of models describing the evolution of the yield stress (τ_y) with the TS content.

Parameters	а	р	$\tau_{y,0}$	n	q	TS ₀	ΤS _c	TS _m
	(kPa)	(kPa)	(kPa)	(-)	(-)	(wt.%)	(wt.%)	(wt.%)
τ_y	$0.25 \ 10^{-2}$	$1.02 \ 10^{-2}$	0.24 10 ⁻²	0.65	2.49	1.9	6	55

In this study, a value of $TS_m=55$ wt. % is found to best fit the experimental data. $TS_m=55$ wt. % corresponds to a volume concentration of $\phi_m=42$ %, which is low by comparison with the maximum packing volume of rigid circular particles which equals to 64 % [61]. This is not surprising because sludge particles are far from being spherical hard spheres.



Figure 6. Description of experimental value by three models: $\tau_{y,v}(...)$, $\tau_{y,transition}(--)$ and $\tau_{v,fr}(--)$.

However, in physical point of view, the use of several models to describe the experimental values of the same material against the concentration is not accepted. The experimental values can be described by a single model combining a viscous and a frictional contributions following Equation 4 (Figure 7). The viscous contribution being dominant in the range TS < $TS_c=6$ wt. %, while the frictional contribution in the range TS > $TS_j=14$ wt. %. Neither is dominant in the transitional phase: $TS_c < TS < TS_j$.

$$\tau_{y} = c[a(TS - TS_{0})^{n}] + (1 - c) \left[\tau_{y,0} \left(1 + \frac{1.25TS}{1 - \frac{TS}{TS_{m}}} \right)^{2} \right]$$
Equation 4

Where a, n and c are model parameters (table 1). The value of c allows to take into account the transition between viscous and frictional regimes, whose nature evolves with the nature of the contact. If the evolution of $(\tau_y - \tau_{y,fr})/(\tau_{y,v} - \tau_{y,fr})$ is represented as function of TS (Figure 7), it can be observed that c is equal to 1 in the viscous regime and tends to 0 in the frictional regime. After a linear fitting, c in the transitional phase, is found equal to: -0.125 TS + 1.75.



Figure 7. Evolution of the parameter c with TS (above). The model proposed to fit the experimental values of τ_v (below).

As mentioned previously, the investigation of the yield stress values is limited to TS=34.3 wt. %. In order to investigate the rheological parameters on the whole range of TS (from 2 to 44.2 wt. %), the shear stress at the end of LVE plateau, τ_{LVE} , has been considered (Figure 8). It has to be noticed that the yield stress τ_y is propositional and roughly equal to 8 times τ_{LVE} on the entire range of concentration. From a physical point of view, this result means that the rheological behaviour of sludge is governed by the same interactions, whatever the strain. Increasing the deformation doesn't change the nature of these interactions, as confirmed by the values of parameters n and c (Table 2).



Figure 8. The model proposed to fit the experimental values of τ_{LVE} .

Table 2. Parameters of the model proposed to fit the yield stress (τ_y) and the shear stress at the end of LVE (τ_{LVE}) as function of the TS content.

Parameters		τ_y	τ_{LVE}	τ_y/τ_{LVE}
a (kPa)		$0.25 \ 10^{-2}$	$0.03 \ 10^{-2}$	~8
n (-)		0.	-	
$\tau_{y,0} \text{ or } \tau_{LVE,0} \text{ (kPa)}$		$0.24 \ 10^{-2}$	$0.03 \ 10^{-2}$	~8
	TS < TS _c	-		
c (-)	$TS_c < TS < TS_j$	-0.125 T	-	
	$TS > TS_j$	0		

A rheophysical scheme, in which the rheological behaviour is related to the texture of sludge, can be proposed (Figure 9). Sludge may be described as a diluted suspension at low concentrations ($TS < TS_c$). When TS exceeds the plastic limit TS_P , the sludge can no longer

be considered as a continuous material and will be referred as a divided granular-like. The classical rotational rheometer is no longer adapted, what explains the large fluctuations in measurements of dilatancy in the vicinity of TS_P . When $TS_L < TS < TS_P$, the sludge can be described as a plastic or granular paste as it exhibits strong similarities with granular materials. Finally, for $TS_c < TS < TS_L$, the material is in an intermediate state between diluted suspension and granular paste: sludge can be described as a concentrated suspension or a soft paste.

TS _c =	=6 wt. % $TS_j=TS_L=$	=14 wt. % $TS_P=43$	wt. % TS (wt. %)
Viscous forces	transitional phase	Frictional-like behaviour	
Diluted suspension	Concentrated suspension or soft paste	Plastic or granular paste	Divided granular-like

Figure 9. Rheophysical scheme corresponding to the rheological behaviour of sludge at different concentrations.

4. Conclusion

The rheological behaviour of a sludge has been experimentally studied on a total solids concentration ranging from 2 to 44.2 wt. % in order to better understand its behaviour during the drying process, especially during the plastic phase. This has been possible by using a classical rotational rheometer with the implementation of an original procedure of surface correction developed previously [26].

Different regimes have been identified and a rheophysical scheme, in which the rheological behaviour is related to the texture of sludge, is proposed. Sludge exhibits a viscous behaviour below $TS_c=6$ wt. %, a frictional (or granular)-like behaviour above $TS_j=14$ wt. %, and is in a transitional phase between TS_c and TS_j .

This work shows that, contrary to the literature, the sludge solid-like properties (yield stress, consistency index and moduli) cannot be represented by a single power law model over the whole range of concentration investigated. The experimental values can be described by the

sum of a viscous (power law) and a frictional (Eilers law) contributions. The viscous contribution being dominant in the range $TS < TS_c=6$ wt. %, while the frictional contribution in the range $TS > TS_j=14$ wt. %. A competition between the viscous and the granular contribution.

It has been shown that the yield stress τ_y is propositional and roughly equal to 8 times τ_{LVE} on the entire range of concentration. Thus, from the LVE plateau the yield stress can be deduced without investigating the whole range of deformation.

Moreover, this work shows that for TS higher than $TS_P=43$ wt. %, sludge is a divided granular-like material and the conventional rotational rheometer is no longer be used. For a better understanding of the flow evolution of sludge all along the dryer, and especially above the plastic limit, other tools are required.

2.2. Résumé

Le comportement rhéologique d'une boue a été étudié sur une large gamme de siccité allant de 2 à 44,2 %. Le but était de comprendre la nature des interactions qui gouvernent le comportement rhéologique de la boue afin de mieux comprendre son comportement dans le sécheur. Ce travail a été possible en utilisant un rhéomètre conventionnel avec la mise en œuvre de la procédure de correction de surface présentée au chapitre 3.

Les résultats obtenus soulignent que la relation entre les propriétés de type solide et la concentration ne peut être représentée par une loi de puissance, pourtant classiquement utilisée dans la littérature. Dans ce travail, nous avons montré que la modélisation des données expérimentales peut être réalisée sur la base d'un modèle composé d'une loi de puissance et d'une loi d'Eilers. La première composante domine pour des siccités inférieures à 6 %, où le comportement est gouverné par des forces visqueuses. La deuxième composante domine lorsque la siccité est supérieure à 14 %. Le comportement est gouverné par des forces de friction qui se manifestent par l'apparition de la dilatance. Aucune des deux composantes n'est dominante dans une gamme de concentration intermédiaire, entre 6 et 14 %. En effet, le comportement est dans une phase de transition.

Grâce à ce travail, un schéma rhéophysique a été proposé, dans lequel le comportement rhéologique est lié à la consistance des boues.

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3 Rhéologie des boues déshydratées à consistance granulaire et plastique

Dans ce travail, nous allons étudier les propriétés d'écoulement des boues déshydratées à consistance granulaire et mettre en évidence l'influence de la siccité sur leur fluidité, grâce à un rhéomètre granulaire. L'utilisation du rhéomètre granulaire est étendue sur les boues de consistance plastique pour caractériser la transition « matériau continu à divisé ». Les résultats obtenus sur les boues de consistance plastique sont ensuite comparés à ceux obtenus via l'utilisation d'un rhéomètre conventionnel.

3.1. Caractérisation rhéologique des boues déshydratées à consistance granulaire et pâteuse/plastique à l'aide d'un rhéomètre granulaire-Article 4

Ce papier est prêt à soumettre. Il fera l'objet d'une publication dans une revue de rhéologie des poudres (à choisir prochainement). Le document est fourni ci-après.