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# State-of-the-art: rheological characterisation of wastewater treatment sludge

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**Abstract**

Since wastewater treatment has been a subject of interest, non-Newtonian flows, such as sludge flows, have been widely studied in the literature, most of the time from a rheological point of view. It is well known that the hydrodynamic behaviour of sludge flows is of prime importance to optimise process parameters of wastewater plants on one hand, as well as those of excess sludge retreating processes on the other hand. The present study is devoted to a synthesis of articles dealing with the rheological characterisation of biological wastewater treatment plant sludges (activated or concentrated ones) and some other concentrated suspensions such as microorganism ones. Attention is notably given to the rheological methods used and to rheological equations and behaviours observed in the different articles. Correlation proposed by some authors linking rheological properties to physico-chemical parameters of suspensions, or to some operating parameters of a process involving the use of sewage sludges are also given in this literature review.


*Keywords:* Biological wastewater treatment; Rheology; Sludge; Literature review

## 1. Introduction

During the second part of the twentieth century, the question of water pollution has taken worrying proportions whereas, as the same time, water consumption increased together with the demographic explosion. In industrialised countries the reduction and the control of water consumption is linked to the optimisation of processes for industrial and domestic wastewater treatment. As concerns the improvement of the different wastewater treatment processes already existing, there is still a lot to do, in particular to achieve a better understanding of the flow properties of wastewater and sewage sludges. In such a wide context, this article proposes a review of the literature devoted to rheological characterisation of non-Newtonian sludge flows. Non-Newtonian sludge flows are first encountered in wastewater treatment processes (e.g. activated sludge wastewater treatment processes) in which they are linked with hydrodynamic phenomena such as stirring or settling and to mass oxygen transfer. Such non-Newtonian flows are also important in processes of treatment of the excess of sewage sludges produced by wastewater treatment plants.

Most of the articles been reviewed in this work are those studying the link between rheological properties of sludge flows and the parameters of the process in which sludges are operated, in particular activated sludge wastewater treatment processes or excess sewage sludge treatment processes (e.g. anaerobic digestion process). Rheology is an interesting tool for the characterisation of the hydrodynamic of sludge suspensions applied to the optimisation of the different processes in which sludges are operated. However, there is an important lack of unity in literature data. It becomes thus not easy to make a synthetic review of these works. This fact is firstly due to the nature of the systems under consideration (complex biological systems with non-Newtonian and often time-dependent behaviours) inducing important space-time variations of sludge samples, as well as a dependence of experimental results from preliminary manipulations the sludge has been submitted to. The type of viscometer used (capillary, rotational, or systemic one) in a given study

*Abbreviations:* CAP, capillary rheometer (Tables 1–6); CCR, coaxial cylinders rheometer (Tables 1–6); CCdcR, double couette coaxial cylinders rheometer (Tables 1–6); CCwgR, wide gap coaxial cylinders rheometer (Tables 1–6); ST, shear-thinning (Tables 1–6); PPR, plane–plane rheometer (Tables 1–6); HRR, rheoreactor with a helical ribbon impeller (Tables 1–6); RT, rheoreactor with a turbine impeller (Tables 1–6); THX, thixotropic (Tables 1–6); VE, viscoelastic (Tables 1–6); VP, viscoplastic (Tables 1–6); ORL, organic loading rate ([17] in Table 2); VS, volatil solid content ([17] in Table 2); FS, fixed solid concentration ([17] in Table 2); SG, specific gas production ([17] in Table 2); $U_{sg}$, superficial gas rate ([17] in Table 2); $T_s$, hydraulic residence time ([17] in Table 2)

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**Nomenclature**

| | |
|---|---|
| $A_i, B_i, C_i, \ldots,$ $1 < i < 24$ | coefficients of correlations of Tables 1–6 |
| $d$ | impeller diameter (m) |
| $k_{\rm L}a$ | global coefficient in mass transfer ($s^{-1}$) |
| $K$ | consistency index (Pa s m) |
| $K_{\rm MO}$ | Metzner–Otto constant |
| $K_{\rm p}$ | laminar constant of the power curve |
| $m$ | flow index |
| $N$ | agitation rate (rad $s^{-1}$) |
| $N_{\rm p}$ | dimensionless power number |
| Nrs | critical speed for off-bottom suspension (rad $s^{-1}$) |
| $P$ | agitation power (W) |
| $Re$ | dimensionless Reynolds number |
| SS | suspended solid concentration (g $l^{-1}$) |
| $t$ | time (s) |
| $T$ | temperature (K) |
| $V$ | fluid volume ($m^3$) |
| $W$ | water content (%) |

*Greek letters*

| | |
|---|---|
| $\dot{\gamma}$ | shear rate ($s^{-1}$) |
| $\Phi_{\rm p}$ | particle volume fraction |
| $\eta$ | viscosity (Pa s) |
| $\eta_{\rm a}$ | apparent viscosity (Pa s) |
| $\eta_{\rm a,sp}$ | specific apparent viscosity |
| $\eta_{\rm B}$ | rigidity or Bingham's parameter (Pa s) |
| $\eta_{\rm C}$ | Casson's parameter (Pa s) |
| $\eta_{\rm e}$ | effective viscosity (Pa s) |
| $\eta_{\rm F}$ | fluid phase viscosity (Pa s) |
| $\eta_\infty$ | viscosity of the Newtonian plateau at high shear rates (Pa s) |
| $\lambda$ | characteristic time of the viscoelasticity of the material (s) |
| $\rho$ | density (kg $m^{-3}$) |
| $\tau$ | shear stress (Pa) |
| $\tau_{\rm Y}$ | yield stress (Pa) |
| $\tau_{\rm Y}^{\rm b}$ | parameter of the Bingham's model (Pa) |
| $\tau_{\rm Y}^{\rm c}$ | parameter of the Casson's model (Pa) |
| $\tau_{\rm Y}^{\rm hb}$ | parameter of the Herschel–Buckley's model (Pa) |

is also responsible for important variations between the results summarised in the different articles. Sludges are particularly difficult materials to characterise in a quantitative manner that is both based on fundamental data and useful for process management. Some parameters have been mathematically defined to apply to laboratory scaled well-defined systems (for example monodisperse suspensions of spherical particles). However, they have not proved their efficiencies for full-scale systems involving complex materials such as actual sludges involved in various actual processes required for wastewater or sludge treatment. It is then possible to list for a given sludge, on one hand physico-chemical properties that can be measured on a theoretical point of view (e.g. concentration or size distribution, particle shape and density, surface chemistry, particle electrokinetic properties or conductivity, colloidal stability, etc.), on the other hand, the technical properties and attributes of those sludges (e.g. foaming or sticking properties, settling or flotation ones, pumping properties, drying characteristics or dewaterability). Unfortunately, most of the former properties are poorly related to the latter ones. Let us consider the example of the capillary suction time (CST) test. While CST measurements have been correlated with sludge dewaterability by different authors [1–4], in some cases no correlation have been found [5]. The difficulty is linked to the fact that the CST test does not quantify any particular, fundamentally-based physical parameter of sludges. As concerns the electrokinetic properties of sludges, they have recently been correlated to conditioning and dewatering processes [5]. However, the methodology used to determine surface properties in concentrated suspensions have to be better developed and the relationship between electrical properties of colloidal particles and process performances when sludge treatment applications are concerned, needs to be further validated.

Rheological characterisation of sludges represents one of the best examples of a fundamentally-based properties that has also been correlated to actual processes. Rheological measurements have thus been widely used to characterise suspensions. In particular, numerous mathematical models have been developed to describe the relation between shear stress and shear rate in the case of industrial suspensions or pastes such as melt chocolate [6,7]. Conversely, there are a relative few number of studies dealing with the application of rheological characterisation to activated or concentrated sludges. However, it appears essential to pay a particular attention to these rheological studies. This is due to the evident importance of the apparent viscosity of sludges on their behaviour in different processes of both wastewater treatment (in particular on the hydrodynamic and transfers phenomena in aeration and settling tanks) and sewage sludges treatment (disposal, dewatering, physico-chemical conditioning, anaerobic digestion, etc.)

## 2. Rheology of suspensions applied to sludges

### 2.1. Fields of application

#### 2.1.1. Activated sludge wastewater treatment processes

Biological wastewater treatment processes using activated sludges are widely spread due to their low operation cost.

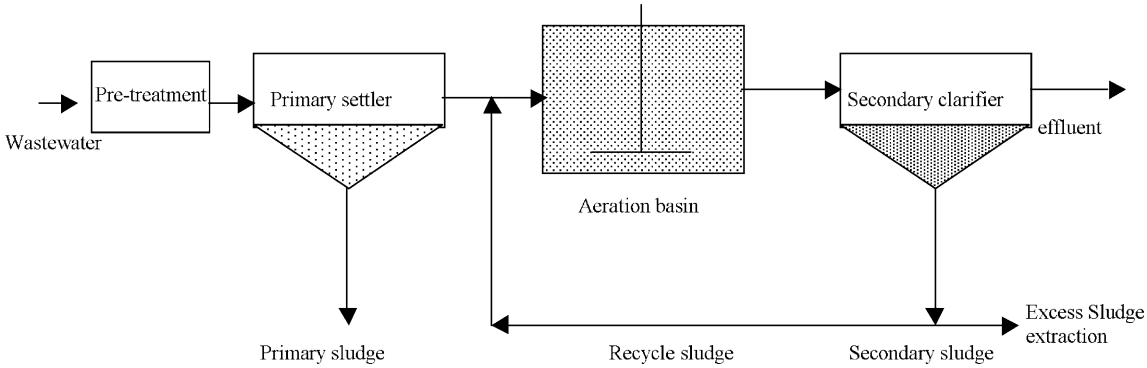


Fig. 1. Scheme of an activated sludge wastewater plant.

They are used for both domestic and industrial wastewater. They aim at putting into contact in an aerated tank the flow to be treated with microorganisms (biomass in suspension) used to digest the pollution. Bacterial flocs previously formed are further separated from pure water in a secondary settler (in the case of the conventional process shown in Fig. 1) or in a membrane filtration module (in the case where a membrane bioreactor is used).

Most of the sludge is recycled in the aeration tank in order to maintain a constant microorganism concentration. The aeration and the subsequent separation of activated sludge (diluted suspension of average suspended matter concentration between 3 and $7\,\mathrm{g\,l^{-1}}$) are the key steps to improve the working conditions of the wastewater treatment.

The aeration tank must ensure two principal functions:

- Aeration of the medium allowing oxygen transfer to the microorganisms.
- Complete suspension of the medium in order to maximise the specific surface between bacterial flocs and the flow to be treated.

As a consequence, optimising a biological wastewater treatment plant implies to model hydrodynamic phenomena in aeration and settling tanks. It is thus of prime importance to study the rheological nature of activated sludges, since this latter will strongly influence the flow properties under stirring or settling conditions of sludge suspensions. The apparent viscosity of activated sludges will also be an important parameter in predicting the oxygen transfer phenomena on flocs surface and thus the level of wastewater purification. In this context some recent articles have been involved in the rheological characterisation of non-Newtonian activated sludges flows [8–11]. Among these studies, some of them also propose to correlate observed rheological behaviours to a fundamental physico-chemical phenomenon strongly influencing the working conditions of the activated sludge wastewater plant, for example:

- Oxygen transfer in the aeration tank.
- Settling of flocs in secondary settlers.

#### 2.1.2. *Excess sewage sludge treatment processes*

Primary and secondary sludges produced in wastewater treatment plants are composed of a complex mixture of organic and mineral, dead and alive matter, that is further treated using specific processes of treatment of these excess sewage sludges. Such processes aim at providing a material useable in classical fields of conversion of biological wastes such as agricultural reuse [12], dumping, incineration or thermochemical conversion [13]. The main goal of the treatments applied to rough sewage sludges is the reduction of both volumes and injuries. At the exit of wastewater treatment processes, sewage sludges are composed of 99% of water and occupy considerable volumes. Sewage sludges are also highly fermentescible matter releasing strong odours. As a consequence, in order to reduce sludges volumes and injuries, they have to be submitted to various treatments (concentration, aerobic stabilisation or anaerobic digestion, conditioning, dewatering, storage, etc.). During these operations rheological properties of sludge suspensions will strongly influence working conditions and scaling-up calculations of tanks, settlers, pumping stations or installations for sludge transport and storage. For all these reasons, some articles have been devoted to the rheological study of more or less concentrated sludges, in order to optimise such processes of treatment of sewage sludges [5,6,12,14–22].

### 2.2. *Rheology of suspensions: theory*

Rheology is the science describing the deformation of a body under the influence of stresses. In the case of Newtonian fluids, the shear stress $\tau$ is linearly related to the shear rate or strain rate $\dot{\gamma}$, according to the Newton equation:

$$\tau = \eta\dot{\gamma} \quad (1)$$

It is convenient to represent the behaviour of flowing materials by means of flow curves, that is graphs of shear stress against strain rate, also called rheograms (Fig. 2). The flow curve of a Newtonian fluid (Fig. 2a) is a straight line through the origin of slope $\eta$.

A Newtonian viscosity will then be constant, for a given fluid or suspension, at constant pressure, temperature and

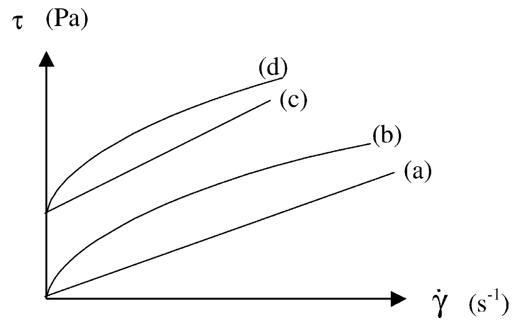


Fig. 2. Schematic flow curves for model time-independent materials. (a) Newtonian; (b) Shear thinning; (c) ideal Bingham plastic; (d) actual plastic.

concentration in solid matter. In the case of diluted enough suspensions that remain Newtonian, the relationship between viscosity and particle concentration is given by the Einstein equation:

$$\eta = \eta_{\mathrm{F}}(1 + 2.5\Phi_{\mathrm{p}}) \quad (2)$$

This latter equation is a key to the relationship between the viscosity of a suspension and its dewaterability. It shows that an increase in the flocs volume fraction and thus an increase in viscosity can obviously be obtain by increasing the concentration in solid particles, but also by incorporating water into the flocs structure. In the case of wastewater sludges, both biomass of flocs and polymers used in sludges conditioning can incorporate substantial amount of water, thus influencing the viscosity of the suspension.

However, wastewater and sewage sludges suspensions are invariably non-Newtonian fluids, the shear rate being non-linearly related to the shear stress. The apparent viscosity of such materials is then a function of the shear rate. The most commonly used equations to represent the non-Newtonian behaviour of sludges suspensions are the Ostwald or power law Eq. (3), the Sisko Eq. (4), the Bingham Eq. (5), the Herschel–Buckley Eq. (6) or the Casson Eq. (7).

$$\tau = K\dot{\gamma}^{m} \quad (3)$$

$$\tau = \eta_{\infty}\dot{\gamma} + K\dot{\gamma}^{m} \quad (4)$$

$$\tau = \tau_{\mathrm{Y}}^{\mathrm{b}} + \eta_{\mathrm{B}}\dot{\gamma} \quad (5)$$

$$\tau = \tau_{\mathrm{Y}}^{\mathrm{hb}} + K\dot{\gamma}^{m} \quad (6)$$

$$\sqrt{\tau} = \sqrt{\tau_{\mathrm{Y}}^{\mathrm{c}}} + \sqrt{\eta_{\mathrm{C}}\dot{\gamma}} \quad (7)$$

Eqs. (3) and (4) used with a consistency index $m < 1$ model shear-thinning (Fig. 2b), i.e. a decrease in the material apparent viscosity when the strain rate is increased. In the case of plastic models (Eqs. (5)–(7), Fig. 2c and d) there is a solid component and a certain well-defined stress, called the yield stress $\tau_{\mathrm{Y}}$ must be reached before flow starts. Its presence is due to the resistance solid particles oppose to deformation, until the applied stress exceed the yield strength of the solid phase and that the sludge show flow. It is commonly admitted that the yield stress of suspensions is linked to the existence of a interconnected three dimensional network of flocs [23]. The value of the yield stress corresponds to the stress needed to be applied to overcome the cohesion Van-Der-Waals forces and induce the flow of the suspension [24]. Magnin and Piau [25] underline that the presence of a yield stress interferes with mass and heat transfers. Byron-Bird et al. [26] have also widely reviewed the flow and heat transfer problems encountered in the presence of yield stress fluids. More generally, it can be noted that, for a given suspension, the value of the yield stress increases with increasing the solid volume fraction. Conversely, at a constant solid concentration, the yield stress decreases when sludge flocs are disrupted under shear. This property is used in a specific method of control during sludge conditioning [15].

## 3. Rheometric methods

The determination of non-Newtonian rheological properties of a given fluid can not be related to a single value of its viscosity. It is linked to the determination of the whole rheogram, that is to say, the plot of shear stress versus shear rate. In the case where a rotational or a systemic rheometer is used (excepted when systemic rheometers are equipped with close-clearance impellers) a difficulty is encountered in non-Newtonian media when it comes to the calculation of the value of shear rate and shear stress in the gap. This is due to the fact that the shear rate and shear stress are spatially averaged over the fluid volume contained in the gap. This can lead to important errors, for example in the case of a plastic fluid under low flow conditions that may include a significant proportion for which the yield stress has not been exceeded and no flow occurs. Conversely, the fluid fraction in motion induces a flow that will be averaged over the whole fluid volume, leading to calculate a value of the viscosity reflective of neither the fluid in motion, nor the fluid at rest. In most of the fields of application of rheology, this problem is avoided through the use of rheometric devices of small dimensions, in order to minimise the spatial variations in shear rate and shear stress. In this respect, most of the suspensions and in particular wastewater sludges are problematic because of the size of flocculated structures that contributes to most of their flow properties and that would be destroyed in such conventional configurations. Another problem can interfere with the rheological characterisation of suspensions and is due to the possible settling of the solid phase during measurements. To solve this particular point, it will be shown latter that the use of a specific rheometric device can lead to a reduction of sedimentation [9,27–29]. As concerns the first point, it appears to date inevitable that rheological measurements on suspensions must remain as bulk properties. It comes that suspensions that are truly plastic in nature, can be characterised as power law fluids, since it is not possible to apply a shear stress that uniformly exceed the yield stress value by a small amount. To avoid this problem, the usual practice consists to extrapolate the linear portion of the rheogram to the ordinate to obtain the assumed true value of the yield stress. To complete the difficulties to obtain reliable rheological measurements on suspensions, we must consider the thixotropic nature of most of the sludges, in other words their time-dependant behaviour. Such a behaviour is mostly linked to the flocculent tendency of those suspensions. As a consequence, the previous history of a sample will be determining for its rheological response. If the suspension has not been sheared, then particles are strongly flocculated and the viscosity is high. Conversely, if the sample has been submitted to a pre-shear sequence, particles are more or less disrupted, leading to a decrease in the observed viscosity. According to some authors [20,31], the power required to maintain flow in a suspension can be divided into three components:

1. The power required to disrupt the large-scale particle network and to maintain a stress overcoming the yield stress.
2. The power required to overcome viscous resistance.
3. The power required to continuously break bonds between groups of particles within flocs.

The first point corresponds to the plastic response of the suspension, the second point is the Newtonian response whereas the last point is linked to the thixotropy of the medium. This latter component is not always observable. Indeed, flocculation phenomena being dynamic in nature, if a stationary equilibrium is rapidly obtain, then no hysteresis will be observed and the third point will be seen as a shear-thinning behaviour. More generally, it can be said that no commercially available rheometer allows to completely avoid all kinds of artefacts, as far as suspensions are concern. For this reason, there is no universally used device to study the rheological behaviour of sludges. Among the different studies under consideration, it will be shown that various devices and geomotries have been used (capillary or rotational rheometers, rheoreactors).

### 3.1. Rheometers and geometries used for sludges

#### 3.1.1. Capillary rheometers

Among the different apparatus and geometries that can be used to determine rheological properties of sludges, the capillary rheometer is the simplest one. It is also rather sensible to low viscosity (diluted sludges) and of a low cost. This device induces the sample to flow laminary at a controlled rate due to a pressure drop applied between the extremities of a capillary tube of known length and diameter. Data obtained are then couples of differential pressure and fluid flow rate. Slatter [22] uses a tube rheometer. His choice is justified by the fact that he proposes to study the relation between rheological properties of sludges and operating conditions of a sludge pumping process. He has reviewed the advantages and disadvantages of using a capillary rheometer:

*Advantages*:

- The capillary rheometer is mechanically simple (as it has been already underlined, this implies a low cost).
- There is a geometric similarity with pipelines that can be useful for scaling-up of processes involving flows of sludges in tubes.
- High shear rates can be attained.
- Diameter dependent effects can be measured.

*Disadvantages*:

- The sample is subjected to varying shear rate and shear stress over the tube cross section. However, these two parameters are generally calculated at the wall.
- The sample can not be subjected to a sustain flow, allowing to measure time-dependant effects such as thixotropy (excepted when data obtained by varying the tube length are compared [32]).
- Relatively large sample volumes are required.

We can also add the fact that, as in the case of concentric cylinders rotational devices, a problem can occur due to the size of the flocculated structures beside the diameter of the tube or the gap between cylinders. Slatter [22] describes in his article, a modified capillary device called the balanced beam tube viscometer. It is composed of two pressure vessels which are located at either end of a beam and connected by transparent tubes of different diameters (5, 13, 18, 28 and 46 mm). A known pressure of compressed air forces the sludge to flow through a selected tube at a controlled rate. The evolution with time of the mass contained in the load cell is registered, indicating the flow transferred through the tube. The principal advantage of this device is that the sludge flow is not measured with a classical flowmeter but calculated from the variations in mass measured by simple weighing. As a consequence, the accuracy is much higher than that of a flowmeter and very low flow can be measured. Among the articles we have reviewed, some have been working on the rheological characterisation of suspensions using commercially available capillary rheometers. It is notably the case of the work of Behn [32], in which digested sludges are involved, or other authors [6,33] that have worked with concentrated sludges. Moeller and Torres [18] have worked on the rheological characterisation of primary and secondary sludges treated by aerobic and anaerobic digestion, using a Brookfield rheometer. Poitou et al. [34] can also be cited, since they have studied the rheological and mechanical behaviour of pasty wastewater sludges using different devices such as a capillary rheometer but also a plane–plane rotational rheometer working in sinusoidal oscillations, a compression cell and a specific squeezing device. In another work, three capillary rheometers of varying diameters (5.8, 11.6 and 15 mm) have been employed to compare the performances of different rheometers, to register rheological properties of filamentous microorganisms suspensions [35].

This work has demonstrated that the capillary device can be used to perform rheological measurements on filamentous micro-organisms, taking care to avoid artefacts due to wall slipping, appearing in the case of the smallest diameter.

*3.1.2. Rotational rheometers equipped with concentric cylinders*

Rotational rheometry associated with a concentric cylinder geometry has been from afar the most employed device in the studies reviewed in this work. The reason for this is certainly linked to the great variety of commercially available devices of this kind, numerous laboratories are equipped with. Such a rheometer is composed of two concentric elements (inner and outer cylinders). One of these two elements is rotated at a controlled rotation rate in order to shear the sample contained in the gap. Shear stress is determined measuring the resistant torque on one of the two elements. Slatter [22] has also reviewed advantages and disadvantages of this kind of rheometer.

*Advantages*:

- Time dependant effects can easily be measured.
- These rheometers are widely spread commercially available devices.
- Such apparatus are compacts and can be used for routine controls.
- Small sample volumes are required.
- Rheograms can be obtained directly connecting a PC to the rheometer.

*Disadvantages*:

- The annular gap must be larger than the largest particles of the sample. On the other hand, this gap has to be as small as possible in order to minimise correction factors and avoid turbulences.
- There is no accurate indication of the transition between laminar and turbulent regimes.
- Centrifuge forces but also sedimentation can cause a particle size distribution and a concentration gradient to appear in the gap, as well as a decrease of the measured torque with time that can interfere with the thixotropy of the sample.

Studies including rheological measurements on suspensions performed with this type of rheometer are the most commonly encountered. Indeed, Behn [32] has both used a capillary rheometer and a rotational one to determine non-Newtonian flow properties of wastewater sludges as a function of the concentration in solid matter and temperature. Abu-Orf and Dentel [5] have worked on the influence of stirring parameters in conditioning tanks on rheological properties of sludges undergoing physico-chemical treatment. Lolito et al. [20] have also used a rotational rheometer with concentric cylinders to demonstrate the influence of various treatment (dewatering, stabilisation) on the rheological properties of sewage sludges. Chavarria [36] has used an identical device in a study of the rheological behaviour of biological flocs. In the case of Grant and Robinson [35] they have compared different rheometers to elucidate rheological characteristics of filamentous microorganisms suspensions. They conclude that capillary rheometers with large enough diameters (to avoid wall slippage), together with rotational ones can be employed for rheological measurements on filamentous suspensions. Rotational rheometry is also used to characterise rheological properties of sewage sludges as a function of the water content and of the temperature [37]. In another work [38], the influence of the temperature on the value of the parameters of plastic or pseudo-plastic rheological models has been studied for primary and secondary sludges, using a rotational device. Dick and Ewing [39] have worked on the rheological characterisation of activated sludges using a modified rotational apparatus equipped with concentric cylinders plus a large gap and heterogeneous surfaces. Some authors [16,17,19] use a similar tool to follow the evolution of rheological properties of sludges under anaerobic digestion. To avoid the problem due to the size of flocculated structures beside that of the annular gap, it is possible to work with sieved sludges [16,17]. This method eliminates in the case of these precise works particles exceeding 0.84 mm in diameter and has been validated provided that the rheological properties of the sieved sludge is representative of the behaviour of the whole sample. Indeed, in the case of the sludges considered in this article, fine particles were representing more than 80% of the total mass. In other works, the problem of the size of suspended particles is avoided using a rheometer equipped with a large annular gap [12,39]. However, this choice implies to use specific calculations to obtain the accurate value of shear rate and shear stress as a function of the position in the gap. This is done by extracting from experimental data the variations of the torque per unit length versus the rotation rate of the moving cylinder giving the function $\dot{\gamma}(\tau, r)$ inside the gap. To conclude with rotational rheometry, it must be noted that some authors have chosen to associate such an apparatus with a geometry differing from the common concentric cylinders. Sutapa [8]and Pagny [9] have worked with slightly concentrated activated sludges (suspended solid concentration $\leq 42\,\mathrm{g\,l^{-1}}$). This led them to make the choice of a double gap geometry to limit settling during rheological measurements on one hand, and on the other hand to enhance sensibility in measuring the shear stress. In the case of calcium sulphate suspensions [40], sludges devoted to land-spreading [12] or pasty suspensions [34], a plane geometry is generally used. The use of rough planes also allows to limit wall slippage [40].

*3.1.3. Systemic rheology*

There is a last category of rheometers that can be encountered in the case of rheological studies involving sludges. These apparatus are composed of small stirred vessels called rheoreactors that can be equipped with different stirring devices (vane stirring device or helical ribbon in most of the cases). The vane method has been described in the literature

[6,15] as a vane rotating at a rate $N$ in a volume of fluid $V$. The power drawn by the stirrer is then $P$ and the mean shear rate created in the fluid is defined by the relation proposed by Camp and Stein [41]:

$$\dot{\gamma} = \sqrt{\frac{P}{\eta_a V}} \tag{8}$$

where $\eta_a$ is the apparent viscosity.

In such a system, the main problem is linked to thixotropic phenomena, which depend above all on the local intensity of the turbulence created rather than on a mean power consumption. Furthermore, the application of Eq. (8) requires to know the value of the apparent viscosity. However this latter is, in the case of non-Newtonian systems under consideration, a function of the shear rate. It comes that the determination of rheological properties is submitted to an uncertainty principle as the one of Heisenberg. From a practical point of view, some authors [6,15] have observed, using such a vane system to follow the evolution of the power required to stir a sludge under conditioning process, that the addition of the polymer induces an important increase in apparent viscosity. However, this increase in viscosity rapidly disappears, due to the adsorption of the polymer on particles surfaces. A second increase in viscosity then follows, due to the flocculation of the solid phase. During a last step, the apparent viscosity gradually decreases as the flocs become large enough to be broken by shear streams. This phenomenon has also been described by Langer et al. [53]. As a consequence, the apparent viscosity of such a system is strongly dependent of the time and, in the case where a constant stirring power is applied, the average shear rate will also fluctuate with time. For this reason, in the case of suspensions as it has been underlined by Dentel [15,52], the value given to $\eta_a$ in Eq. (8) must never be taken as the value of the viscosity of the fluid phase. In the case of the agitation of sludges, which structure is most of the time evolving with time, the choice of an helical ribbon impeller has been made in different works [9,27–29,35], in order to limit settling phenomena. Indeed, such a phenomenon is known to influence the determination of flow properties of suspensions, in particular when low concentrated suspensions are concerned. As a consequence, in the case of diluted suspensions, notably activated sludges (suspended solid concentrations of about 3–7 $\mathrm{g\,l^{-1}}$), most of the authors work on their rheological characterisation using concentrated suspensions. In such a context, the use of a close-clearance impeller with a high pumping axial flow allows to limit the artefacts due to settling. A rheoreactor equipped with a helical ribbon has then been used to perform rheological flow measurements on filamentous fungi suspensions [27,28]. These measurements have been performed using the Metzner–Otto's principle [42] that define an effective viscosity, based on the generalisation for non-Newtonian media of the relation existing in an agitated vessel between the dimensionless power number ($N_p$) and the Reynolds number ($Re$).

$$N_p = \frac{P}{\rho N^3 d^5} = \frac{K_p}{Re} = \frac{K_p \eta_e}{\rho N d^2}, \quad \text{then} \quad \eta_e = \frac{P}{K_p N^2 d^3} \tag{9}$$

At a rotation rate value of $N$ corresponds an effective shear rate $\dot{\gamma}_e$ related to the rotation speed of the impeller by the Metzner–Otto constant characterising the stirrer geometry ($K_{MO} = 30$–$40$ for helical ribbon used by Ducla et al. [27])

$$\dot{\gamma}_e = K_{MO} N \tag{10}$$

Grant and Robinson [35] have also studied the rheology of filamentous microorganisms, using a rheoreactor successively equipped with a helical ribbon and a turbine radial impeller. They have compared results obtained with this kind of rheometer with results provided by more conventional devices (capillary and rotational rheometers). They draw the conclusions that, for the type of microorganisms under consideration, the rheoreactor is not the most adapted geometry. However, they have underlined that in the case of non filamentous particles (e.g. cellulose particles) such a device is on the contrary more accurate that classical rheometers. More recent works have permitted to use this type of geometry to perform oscillatory dynamic measurements [29]. Pagny [9] uses a rheoreactor to characterise the flow properties of slightly concentrated activated sludges (suspended solid concentrations varying between 5 and 110 $\mathrm{g\,l^{-1}}$) in flow and dynamic modes. This author also compares results obtained with this geometry with those obtained with a double Couette rotational device. This leads to the conclusion that the helical ribbon impeller limits particles settling during rheological measurements excepted in the case of the smallest concentration, for which settling phenomena remain important in both configurations.

### 3.2. Rheological measurements

In the case of flocculated suspensions there are two kinds of measurements useful to characterise the structure under consideration. Using these two kinds of measurements, complementary informations about the internal structure of the suspensions can be obtained. On one hand, the flow or shear measurements allow to characterise the structure of the suspension under laminar flow, i.e. its viscous and plastic properties. On the other hand, dynamic measurements characterise viscoelastic properties of the material, submitting this latter to sinusoidal deformations. Dynamic rheology also allows to determine the limit between viscoelastic and plastic behaviours, i.e. a value of the yield stress.

#### 3.2.1. Flow measurements

These measurements are the simplest to carry on and thus have been widely used in previous studies devoted to the rheological characterisation of suspensions. The Couette experiment is reproduced with coaxial cylinders or with a cone and plate or plate and plate geometry, in order to determine the laminar stationary flow curve (rheogram) of the product

(Fig. 2). In the most commonly encountered case of coaxial cylinders, one of the two cylinders is rotated, creating in the sludge contained in the annular gap:

(i) A constant shear rate (shear rate imposed apparatus). The shear stress is then deduced from the value of the torque measured on the axis of the cylinder at rest.
(ii) A constant shear stress (shear stress imposed apparatus). The shear rate is then determine, measuring the flow generated in the gap.

It should be underlined that constant shear stress experiments are particularly interesting, as far as viscoplastic properties are concerned. They are indeed useful to characterise more accurately the yield stress value of plastic materials, performing flow experiments with a sufficiently low rate of increase in shear stress, to determine from which value the suspension begins to flow. The use of a capillary rheometer also allows to draw the rheogram of a sludge, simply converting the experimental data (differential pressure applied between the tube extremities, flow rate) in calculated data (wall shear stress, wall shear rate). Once the rheogram has been drawn, the rheological model that gives the best fit of the experimental data has then to be found among classical plastic or pseudo-plastic models. The parameters of the retained model are then calculated by regression. In the case of wastewater sludges suspensions, most of the rheological studies have been devoted to the determination of the single flow properties of the sludge [5,6,10,16–20,22,32,36–39]. Due to similarities existing between rheological behaviours of wastewater and sewage sludges and other concentrated suspensions, some studies dealing with the rheological characterisation of concentrated suspensions of microorganisms are also cited in this work [27,28,35]. Finally, in addition to the determination of the flow properties of sludges, some authors have chosen to perform dynamic oscillatory measurements, in order to characterise in the most complete manner the structure under consideration [8,9,12].

### 3.2.2. Dynamic measurements with sinusoidal oscillations

In some studies, dynamic measurements are use to complete informations drawn from flow measurements. Viscoelastic properties of the material (Section 4.4) are then involved and can be evaluated due to rheological dynamic measurements. Such a measure implies to apply to the suspension a known sinusoidal deformation $\gamma^* = \gamma_0\,e^{i\omega t}$, of low amplitude $\gamma_0$, above the critical value $\gamma_c$, that limits the linear viscoelastic range of the material. The sinusoidal stress in the sample $\tau^* = \tau_0\,e^{i\omega t+\delta}$ is then measured together with the phase angle difference $\delta$. It can be noted that the span of the linear viscoelastic domain of a concentrated suspension can be considerably reduced (sometimes <0.1% in deformation), compared to the one of melt polymers that can reach 30% in deformation [40]. The response of the suspension will first be translated in terms the phase angle difference $\delta$ between complex deformation and stress (with $\delta \to 0°$ for the perfect elastic solid of Hook and $\delta \to 90°$ for the ideal liquid of Newton); and then in terms of the complex modulus ($G^* = \tau^*/\gamma^*$), from which a viscous component ($G''$) and an elastic component ($G'$) will be calculated.

If we suppose that a material can be described, in its linear viscoelastic domain, by a viscous (irreversible viscous flow) and an instantaneous elastic (reversible elastic recovery) components that are represented by the Maxwell model, then:

$$\eta\dot{\gamma} = \tau + \lambda\frac{d\tau}{dt} \tag{11}$$

$\lambda$ being a time characterising the elasticity of the material. Then:

$$G^* = \frac{\tau^*}{\gamma^*} = \frac{\eta i\omega}{1+i\lambda\omega} = G' + iG'' \tag{12}$$

Thus, the value of the storage or elastic modulus in phase with the deformation will be:

$$G' = \frac{\eta\lambda\omega^2}{1+\lambda^2\omega^2} \tag{13}$$

And the loss or viscous modulus in phase with the deformation rate is as follows:

$$G'' = \frac{\eta\omega}{1+\lambda^2\omega^2} \tag{14}$$

If we make an analogy with the Newton equation, we can also define a complex viscosity:

$$\eta^* = \frac{\tau^*}{\dot{\gamma}^*} = \eta' - i\eta'' \quad \text{with} \quad \eta' = \frac{G''}{\omega} \quad \text{and} \quad \eta'' = \frac{G'}{\omega} \tag{15}$$

Friction loss properties, in phase with the deformation rate $\dot{\gamma}$ being represented by $\eta'$, whereas the elastic properties are represented by $\eta''$.

To illustrate rheological dynamic method, variations of $G'$ and $G''$ as a function of $\omega$, for a model viscoelastic fluid are shown in Fig. 3. Authors that have chosen to study the rheological characterisation of activated and sewage sludges combining flow rheometry with dynamic rheometry are Sutapa [8], Pagny [9], and Baudez [12]. With respect to flow measurements previously described, dynamic measurements

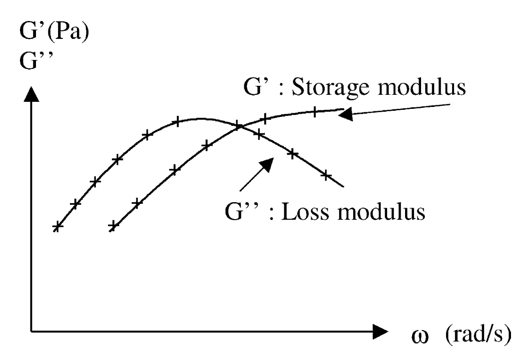


Fig. 3. Schematic variation of the storage and loss modulus as a function of angular velocity for a model viscoelastic fluid.

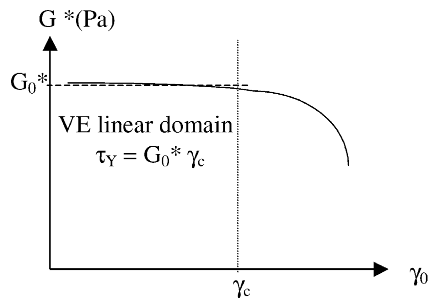


Fig. 4. Schematic representation of the linear viscoelastic domain and calculation of $\tau_Y$.

also allow to provide a value of the yield stress of a given plastic material. This is done by performing dynamic measurements at an average frequency (generally about 1 Hz) to determine the critical amplitute of deformation $\gamma_c$ above which the linear domain takes end. Above this deformation, the complex modulus $G^*$ is no more a constant and begin to decrease. The product between the complex modulus on the plateau and the critical deformation gives directly the value of the yield stress (Fig. 4). The interest is then to compare values of the yield stress obtained by extrapolation of the flow curve at zero shear rate with dynamic values.

Sutapa [8] observes that yield stress values measured by both methods are of the same order of magnitude, the accuracy of values obtained from dynamic measurements been higher.

## 4. Rheological behaviour of concentrated suspensions

### 4.1. Shear-thinning behaviour

A fluid is said to show shear-thinning when its apparent viscosity decreases as the shear rate increases. A lot of works dealing with the rheological characterisation of concentrated suspensions have chosen to represent the rheological state equation of these materials, using a shear-thinning model (Eqs. (3) and (4)). A synthetic review of these studies is given in Table 1. It can be noticed that on the range of shear rates under consideration, the power law Ostwald model is the most commonly employed, probably due to its simplicity. Some authors [18,27,28,32] have thus chosen to model the behaviour of concentrated suspensions using this power law model, whereas other authors [8,9] have worked on a wider shear rate range and thus observe a constant viscosity plateau in the region of high shear rates. For this reason, they use the Sisko model to represent flow properties of activated sludges. Chavarria [36] has proposed his own rheological model for characterising biological flocs suspensions (Table 1). Other authors [20,35,38] have tried to represent the rheological behaviour of concentrated suspensions using both a viscoplastic model (Table 2) and the shear-thinning Ostwald model (Table 1). The question to solve is to know whether concentrated suspensions are yield stress fluids or not. It appears that most of the authors consider that, in the presence of sufficient solid particles concentration, there is effectively a rigidity of the suspension structure that must be overcome to induce flow. In such a context, what we need to know is whether the value of the yield stress is significant or not, that is to say, whether a viscoplastic model will be necessary to model the suspension flow or not. The existence of a yield stress in the case of microorganisms suspensions has clearly been demonstrated by Ducla et al. [27]. However, the order of magnitude of this yield stress being low (and beside the limit of precision of the rheological device), and also because the process considered was operating at high shear rates, the authors proposed to model the obtained flow curves, using the power-law model. As concerns the influence of physico-chemical and operating factors on the parameters of the chosen rheological state equation, only the influence of the solid concentration has generally been described [8,9,18,20,27,28,32,35,36]. Behn [32]and Manioladis and Bishop [38] have also studied the influence of temperature on the parameters of the Ostwald equation.

### 4.2. Viscoplastic behaviour

Viscoplastic materials are defined as materials which show flow when a yield stress value is exceeded. Studies that have used viscoplastic models (Eqs. (5)–(7)) to represent the rheological behaviour of concentrated suspensions are reviewed in Table 2. As it has been previously underlined, the existence of a yield stress has been previously discussed in different works [15,25,30,39]. The question was to know if measured yield stresses were not artefacts due to the lack of precision of rheological devices at low shear rates [43]. At that date, apparatus being more and more accurate, in the case of aggregated concentrated suspensions such as wastewater sludges, it is commonly admitted that there are yield stress fluids. However, as we have seen in the last paragraph, when working at sufficiently high shear rate or shear stress, some authors can chose to model wastewater sludges using a simple shear-thinning equation. Nevertheless, the existence of the yield stress being, in the case of concentrated suspensions, linked to the formation of a fractal interconnected network, it appears interesting to study how its value evolves with various physico-chemical or operating parameters. In articles cited in Table 2, the influence of the solid concentration [10,12,19,20,22,35,37–39] or that of the temperature [37,38] on rheological parameters has been reported.

### 4.3. Thixotropy

Thixotropy is defined as a reversible time-dependent decrease of viscosity when a constant shear rate or shear stress is applied to a fluid. Beside the non-Newtonian characteristics just cited, wastewater and sewage sludges are also thixotropic systems. This latter property especially enhances the difficulties to obtain reliable rheological measurements

Table 1
Shear-thinning modelling of dense suspensions

| Ref. | Studied suspensions | $\dot{\gamma}$ range (rheometer) | Proposed model | Parameters modelling |
|---|---|---|---|---|
| [32] | Digested sludges<br>SS=10 to 147 $g.L^{-1}$<br>T=16 – 20 - 25 – 32°C | ST+ THX<br>4 -7000$s^{-1}$<br>(CAP)<br>and 2-50 $s^{-1}$<br>(CCR) | Ostwald | Effect of SS :<br>$K = A_1 SS^{B_1}$<br>$m = C_1 - D_1 \log(SS)$<br>Effect of T: small beside the effect of SS |
| [36] | Sewage sludges<br>SS=7.7 to 36 $g.L^{-1}$ | ST<br>(CCR) | $\log\dot{\gamma} = \alpha_2 + \beta_2 \log\eta$ | Effect of SS : $\alpha_2 = A_2 + B_2 \log SS$<br>$\beta_2 = C_2 SS - D_2$ |
| [38] | I and II sludges<br>SS=20 to 66 $g.L^{-1}$<br>T= 10 to 25°C | ST<br>Or VP<br>(CCR) | Ostwald or Bingham (table 2) | Small effect of SS<br>Effect of T:<br>m almost independent of T ; $K = A_3 e^{-B_3 T}$ |
| [27] | microorganisms suspensions<br>$\Phi_p$ = 0.102 to 0.2 | ST<br>40-3700$s^{-1}$<br>(HRR) | Ostwald | Effect of the solid volume fraction :<br>$\log K = A_4\Phi_p - B_4$ and $\log m = -C_4\Phi_p - D_4$ |
| [28] | microorganisms suspensions<br>$\Phi_p$ = 0.1 à 0.3 and Ca alginate suspensions<br>$\Phi_p$ = 0.24 à 0.42 | ST<br>0.3 to 20$s^{-1}$<br>(HRR) | Ostwald | Effect of the solid volume fraction :<br>K ↑ when $\Phi_p$ ↑. |
| [35] | Suspensions of 3 different microorganisms<br>SS=3 to 15.5 $g.L^{-1}$ | ST or VP<br>45-4000 $s^{-1}$<br>(CAP)<br>1-460 $s^{-1}$<br>(CCR)<br>0-150 $s^{-1}$<br>(HRR)<br>0-40 $s^{-1}$(TR) | Ostwald or Bingham or Casson (table 2) | Effect of SS :<br>$K = A_5 SS^{B_5}$ , m(SS) ↓<br>parameters varying for each kind of microorganisms |
| [8] | Domestic and industrial activated sludges or laboratory sludges<br>SS=3.3 to 42 $g.L^{-1}$ | ST<br>$10^{-2}$to $10^{3}$ $s^{-1}$<br>(CCdcR) | Sisko | Effect of SS :<br>$\eta_\infty = A_6 10^{B_6 SS}$<br>$K = C_6 10^{D_6 SS}$<br>$m = E_6 + F_6 \log(SS)$ |
| [9] | Activated sludges<br>SS=5 to 110 $g.L^{-1}$ | ST<br>$10^{-2}$to 200$s^{-1}$<br>(HRR)<br>(CCdcR) | Sisko | Effect of SS :<br>$\log(\eta_\infty) = A_7 \log(SS) + B_7$<br>$K = C_7 SS + D_7$<br>$\log(m) = E_7 + F_7 \log(SS)$ |
| [20] | Activated and digested sludges,<br>SS=12 to 57 $g.L^{-1}$ | ST or VP<br>0-1300$s^{-1}$<br>(CCR) | Ostwald or Bingham (table 2) | Effect of SS : K and m = $(A_8 SS + B_8) SS + C_8$<br>or K and m = $D_8 SS^{E_8}$ |
| [18] | I and II sludges, anaerobic and aerobic digested sludges<br>SS = 7 to 13 $g.L^{-1}$ | ST<br>2-30$s^{-1}$<br>(CAP) | Ostwald | Influence of W, DCO, DBO, N , pH, SS, TVS :<br>K ↓ when W ↑, no influence of the other parameters<br>For m : no correlation has been found |

with such fluids. In particular, since it is not possible to introduce the sample in the rheometer without shearing it more or less, the rough rheological behaviour of such a system can never be observed. Most of the rheological studies devoted to wastewater or sewage sludges only cite the thixotropic properties of studied systems and remind that they can induce some artefacts in rheological measurements [39]. To reduce these possible artefacts, it is recommended to perform rheological measurements on concentrated suspensions always proceeding in the same way. The sample is generally submitted to a pre-shear phase, in order that different samples all acquire the same degree of internal structure before rheological characterisation can be performed [10,12]. Behn [32] uses capillary tube viscometers of the same diameter

Table 2
Viscoplastic modelling of dense suspensions

| Ref. | Studied suspensions | $\dot{\gamma}$ range (rheometer) | Proposed model | Parameters modelling |
|---|---|---|---|---|
| [39] | Activated sludges<br>SS=1.5 à 9 $g.L^{-1}$ | VP and THX (CCR) | Bingham | Effect of SS : $\tau_Y^b = A_9 e^{B_9 SS}$ |
| [38] | I and II sludges<br>SS=20 to 66 $g.L^{-1}$<br>T= 10 to 25°C | ST Or VP (CCR) | Bingham or Ostwald (table 1) | Small effect of SS<br>Effect of T :<br>Small effect on $\eta_B$; $\tau_Y^b = A_{10} e^{-B_{10} T}$ |
| [35] | Suspensions of 3 different microorganisms<br>SS=3 to 15.5 $g.L^{-1}$ | ST or VP<br>45-4000 $s^{-1}$ (CAP)<br>1-460 $s^{-1}$ (CCR)<br>0-150 $s^{-1}$ (HRR)<br>0-40 $s^{-1}$(TR) | Bingham or Casson or Ostwald (table 1) | Effect of SS :<br>$\tau_Y^b = A_{11} SS^{B_{11}}$ $\eta_B = C_{11} SS^{D_{11}}$<br>$\tau_Y^c = E_{11} SS^{F_{11}}$ $\eta_C = G_{11} SS^{H_{11}}$<br>parameters varying for each kind of microorganisms |
| [22] | Digested sludges<br>SS=32 to 66 $g.L^{-1}$<br>($SS_{max} = 425 g.L^{-1}$) | VP<br>0-1000$s^{-1}$<br>(CAP) | Herschel -Buckley | Effect of SS : $\tau_Y^{hb} = A_{12}(SS^3/SS_{max} - SS)$<br>$K = \eta_{eau}(1 - SS/SS_{max})^{-B_{12}}$<br>$m = 1 - C_{12} SS^2 - D_{12} SS$ |
| [20] | Activated and digested sludges,<br>SS=12 to 57 $g.L^{-1}$ | ST or VP<br>0-1300$s^{-1}$<br>(CCR) | Ostwald or Bingham (table 1) | Effect of SS :<br>$\tau_Y^b ; \eta_{B\,;} = (A_{13} SS + B_{13}) SS + C_{13}$<br>or $\tau_Y^b , \eta_{B\,;} = D_{13} SS^{E_{13}}$ |
| [37] | Mineral sludges<br>SS=50 to 150 $g.L^{-1}$<br>Critical water content : $W_{CR}$ = 98%<br>(for Newtonian sludges) | VP<br>0-1000$s^{-1}$<br>(CCR) | Bingham | Effect of W : $\tau_Y^b = A_{14} e^{[B_{14}(W_{CR} - W)]}$<br>$\eta_B = \eta_F C_{14} e^{[D_{14}(W_{CR} - W)]}$<br>Effect of T :<br>$(\tau_{Yo}^b / \tau_Y^b - 1) = E_{14}(T - T_0/100)$<br>$(\eta_{BO}/\eta_B - 1) = F_{14}(T - T_0/100)$ |
| [19] | Sludges undergoing anaerobic digestion<br>SS(t)=30 to 40 $g.L^{-1}$<br>SS(t)=20 to 30 $g.L^{-1}$ | VP<br>0-1000$s^{-1}$<br>(CCR) | Herschel -Buckley or Bingham | $\tau_Y^{hb}(t)$ and $K(t)\downarrow$ then $\tau_{Yc}^{hb}(SS)$ and $K(SS)\uparrow$<br>and $m(t)\uparrow$ then $m(SS)\downarrow$<br>$\tau_Y^b(t)$ and $\eta_B(t)\downarrow$ then $\tau_Y^b(X)$ and $\eta_B(X)\uparrow$ |
| [16] | I and activated sludges, digested sludges<br>SS=8 to 350 $g.L^{-1}$<br>T=5 to 55 °C | VP<br>0-100$s^{-1}$<br>+ THX<br>(CCR) | Bingham | Effect of TVS :<br>$\tau_Y^b = A_{15}[VS]^{B_{15}}$ or $\tau_Y^b = C_{15} e^{[D_{15}(VS)]}$<br>$\eta_B = E_{15}[VS]^{F_{15}}$ or $\eta_B = G_{15} e^{[H_{15}(VS)]}$<br>Effect of T : $\tau_Y^b = I_{15} e^{\Delta E/RT}$ $\eta_B = J_{15} e^{\Delta E'/RT}$ |
| [17] | Sludges + organic fraction of wastewater undergoing anaerobic digestion acido and methanogenic phases<br>SS=20 to 100 $g.L^{-1}$ | VP<br>0-100$s^{-1}$<br>(CCR) | Bingham | Acidogenic phase:<br>$\tau_Y^b = A_{16} e^{-B_{16}(\ln VS)} e^{C_{16}(\ln VS)^2} e^{-D_{16}/\ln T}$<br>$\eta_B = E_{16} e^{F_{16}(VS)} e^{G_{16}(OLR)} e^{H_{16} Ts} e^{-I_{16}/T}$<br>Methanogenic phase :<br>$\tau_Y^b = J_{16} e^{K_{16}(VS)^2} e^{-L_{16}(FS)} e^{M_{16} SG}$<br>$\eta_B = N_{16} e^{O_{16}(VS)} e^{-P_{16}(FS)} e^{Q_{16} OLR} e^{-R_{16} SG}$ |
| [10] | Activated sludges<br>SS=0 to 20 $g.L^{-1}$ | VP + THX<br>0-900$s^{-1}$<br>(CCdcR) | Bingham | Effect of SS :<br>$\tau_Y^b$ et $\eta_B$ $\uparrow$ exponentially with SS |
| [12] | Pasty sludges for land-filling<br>$\Phi_p$ = 0.11 to 0.126 | VE $\tau < \tau_Y$<br>VP $\tau > \tau_Y$<br>(CCwgR)<br>(PPR) | Burgers<br>Herschel -Buckley | Effect of the stockage time ts :<br>All mechanical parameters $\downarrow$ with ts $\Rightarrow$ dimensionless equation $\forall$ ts :<br>$\frac{\tau}{\tau_Y} = 1 + \lambda(\frac{\dot{\gamma}}{\tau_Y})^m$ with $\lambda(\Phi_p) = A_{17} e^{-B_{17}\Phi_p}$ |

Table 3
Viscoelastic modelling of dense suspensions

| Ref. | Studied suspensions | $\dot{\gamma}$ range (rheometer) | Proposed model | Parameters modelling |
|---|---|---|---|---|
| [12] | Pasty sludges for land-filling $\Phi_p = 0.11$ to 0.126 | VE $\tau < \tau_Y$ VP $\tau > \tau_Y$ (CCwgR) (PPR) | Burgers Herschel-Buckley | Effect of the stockage time ts : All mechanical parameters ↓ with ts ⇒ dimensionless equation ∀ ts : $\frac{\tau}{\tau_Y} = 1 + \lambda(\frac{\dot{\gamma}}{\tau_Y})^m$ with $\lambda(\Phi_p) = A_{17} e^{-B_{17}\Phi p}$ |
| [9] | Activated sludges SS=5 to 110 $g.L^{-1}$ | Linear VE for $\gamma < \gamma_c$ (HRR) (CCdcR) | - | $\tau_Y = \gamma_c G^*$ Effect of SS on yield stress values : $\ln \tau_Y = A_{18} \ln SS - B_{18}$ |
| [44] | Bacterial suspensions $\Phi_p = 0.58$ to 0.75 | VE $10^{-3}$ to $1 s^{-1}$ (CCR) | generalised Maxwell equation | Effect of the solid volume fraction : $G' \propto \Phi_p{}^{A_{19}}$ $\eta' \propto \Phi_p{}^{B_{19}}$ |

but different length to extract the thixotropic properties of digested sludges. Very few authors have tried to model the thixotropic character of activated or sewage sludges. Battistoni [16] has shown in his work that the surface of the hysteresis loop was exponentially related to the volatile suspended solid concentration, in the case of sewage sludges.

### 4.4. Viscoelasticity

When a shear stress is applied suddenly to a fluid, the initial value of the shear rate may not be maintained (time-dependent effect) for two distinct reasons. The first reason is that the structure of the material may have changed in some way. If the strain rate increases with time, due to the fact that some bonds between suspended particles have been broken, the suspension shows thixotropy as it has been discussed in the last paragraph. The second reason for a change of strain rate may be that part of the mechanical energy supplied to the suspension is stored as elastic energy (for example in interparticle bonds). Such systems are viscoelastic materials since they flow (viscous component) but when reducing the stress to zero, a partial elastic recovery is observed (elastic component). Sludges like other kind of suspensions have viscoelastic properties that can be underlined using dynamic rheological measurements. Among the articles using dynamic measurements to characterise dense suspensions, most of them have performed these measurements to complete the informations drawn from the flow curve. It is the case of Sutapa [8] or Pagny [9] that have worked with activated sludge suspensions. They use the dynamic mode to obtain more reliable values of the yield stress than the one obtained by extrapolating the rheogram at zero shear rate. Pagny [9] has observed that yield stresses of activated sludges are too low to be detected on flow curves that are thus modelled using the shear-thinning Sisko equation. Baudez [12] also uses dynamic measurements in combination with flow measurements. He proposes to model the behaviour of sludges devoted to land-spreading suspensions by a Burger type model. Other authors such as Toda et al. [44] have only used dynamic measurements for characterising the rheological properties of microbial concentrated suspension. They have studied variations of the storage and loss modulus as a function of the cell volume fraction and have concluded that the formation of the cell network was characterised by three steps corresponding to three levels of structure (Table 3).

## 5. Relation with physico-chemical properties of flocs or with the process

### 5.1. Relation with surface and charge chemistry or with structural properties of flocs

Forster [14,45], has demonstrated the relation existing between the surface composition of activated and digested sludge particles and their rheological properties. In a study of 1981, he has performed qualitative preliminary studies that lead to the conclusion that the non-Newtonian behaviour of such suspensions is probably linked to the surface charges of the particles of the flocs. In another study of 1983, he has submitted different kinds of sludges (activated and digested ones) to various enzymatic treatments leading to the selective lysis of determined surface groups (Table 4). He has then demonstrated that surface groups strongly influencing the rheological properties are proteins and polysaccharides, in the case of activated sludges and proteins and lipopolysaccharides, in the case of digested sludges. Subsidiary experiments have finally shown that the bound water content of the sludges was influencing their rheological

Table 4
Correlations between surface chemistry and structural properties of flocs and rheology of dense suspensions

| Ref. | Studied suspensions | rheology (rheometer) | Observed correlations |
|---|---|---|---|
| [24] | Activated and digested sludges<br>SS=10 to 50 $g.L^{-1}$ | THX<br>(CCR)<br>$\dot{\gamma} = 378 s^{-1}$ | Effect of SS : $\eta_a(\dot{\gamma} = c^{st}) = A_{20}e^{B_{20}.SS}$<br>with $\eta_a$ (activated sludges) > $\eta_a$ (digested sludges)<br>THX ↑ when SS ↑ , and for SS=$c^{st}$<br>THX (activated sludges) < THX (digested sludges)<br><br>Measurement of the electrophoretic mobility (σ):<br>σ ( activated sludges) > σ (digested sludges)<br>Effect of the pH for activated sludges (highly charged particles) :<br>when pH ↓ , σ ↓ , $\eta_a$ ↓ |
| [45] | Activated sludges<br>SS=18 to 26 $g.L^{-1}$<br>and digested ones<br>SS=29 to 35 $g.L^{-1}$ | THX<br>(CCR)<br>$\dot{\gamma} = 378 s^{-1}$ | For activated sludges : action of enzymes : lipase, cellulase or proteinase ⇔ $\eta_a$ ↓<br>For digested sludges : action of enzymes : lipase, or proteinase ⇔ $\eta_a$ ↓ |
| [47] | Latex and polystyrene suspensions<br><br>Volume fraction in primary particles :<br>$0.02 < \Phi_p < 0.2$ | VE<br>(CCdcR) | Effect of $\Phi_p$ :<br>$G'_\infty = A_{21}10^{B_{21}\Phi_P}$ $\gamma_c = C_{21}10^{D_{21}\Phi_P}$<br>$\frac{\eta_0}{\eta_F} = E_{21}10^{F_{21}\Phi_P}$ $\tau_Y = H_{21}10^{I_{21}\Phi_P}$<br>$\lambda = J_{21}10^{K_{21}\Phi_P}$ |
| [10] | Activated sludge<br>SS=0 to 20 $g.L^{-1}$ | VP + THX<br>0-900$s^{-1}$<br>(CCdcR) | Effect of the pH and thus of the zeta potential ζ :<br>$\tau_Y^b$ and $\eta_B$ ↑ exponentially with the pH |
| [11] | Activated sludge<br>SS = 3.5 to 4 $g.L^{-1}$ | VP<br>$\dot{\gamma} = 36.7 s^{-1}$<br>and<br>$\dot{\gamma} = 73.4 s^{-1}$<br>(CAP) | Effect of SS : when SS ↑<br>$\eta_{a,sp}(\dot{\gamma} = c^{ste}) = \frac{\eta_a}{\eta_{eau}} - 1$ ↑ , $\tau_Y^b$ ↑ , $\eta_B$ ↑<br>⇔ ↑ number of interparticle interactions that oppose to the suspension flow ⇔ ↑ erosion<br>⇔ ↑ [fine particles] ⇔ ↓ filterability ⇔ ↓ CST |

behaviour and that this influence could be modified by the action of metal ions. Dollet [10] has studied the rheological characterisation of the state of flocculation of activated sludges. He has shown the effect of both solid content and pH on the value of the parameters of the Bingham equation. He has demonstrated that both $\tau_c^b$ and $\eta_B$ increased when the pH increased until 7, indicating a better cohesion of flocs (maximum $\eta_B$) and thus an optimal state of flocculation for pH between 6 and 7 beside acid pH. Paradoxically, the variations of the zeta potential as a function of pH indicate that $\zeta$ is strongly negative ($\cong -15$ mV) for pH between 6 and 7 and thus that we are far from the isoelectrical point. The interpretation given is then as follows. For pH between 6 and 7, the coexistence of both $COO^-$ and $NH_4^+$ functions in the polymeric matrix of flocs allows the spatial expansion of surface macromolecules. Then, due to the presence of divalent cations, these macromolecules are bound together, leading to the flocculation of particles. In a recent study, Mikkelsen [11] has demonstrated the existence of strong correlations between the mass fine particle concentration, the resistance to filtration (quantified by the CST) and the rheological properties of activated sludge suspensions of various concentrations. As far as the suspended solid concentration is increased, the author postulates that the development of an interparticle network (quantified by the increase in the rheological parameters value) is responsible for the restriction of flow and thus for an increase of the surface erosion of primary particles. The level of fine particles in the medium is subsequently increased, leading to a bad filterability (Table 4). During the last fifteen years, rheological properties of dense aggregated suspensions have also been interpreted in terms of the fractal concept [46]. The fractal models that have been proposed all assume that the aggregated structure transmits stresses applied to the suspension, through the chains of an elastic backbone. These chains can be either connected into a three dimensional space-filling network (as it is probably the case at low shear rates), or confined to the internal volume of flocs when the shear rate is enhanced and that the network has been disrupted. Adopting the network theories of polymer dynamics, that have much in common with colloidal networks, and incorporating the fractal concept, Potanin et al.

[47] have proposed a microrheological model to describe the rheological properties of a well-characterised polystyrene latex dispersion. Pagny [9] has then exploited their results to apply this microrheological model to slightly concentrated activated sludges. This model tries to establish the link between the flow properties of a given weakly aggregated suspension and the structural properties of its constitutive particles. The shear stress is calculated as the sum of a hydrodynamic and a structural part. The former is attributed to the hydrodynamic cores of fractal aggregates, which behave as a suspension of impermeable spheres. The latter accounts for the forces transmitted by chains of particles linking the aggregates into a transient network. The dispersion is thus described as a system of overlapping aggregates, which can be connected by chains gradually broken and created due to shear and thermal processes. A distinction is made between rigid and soft chains that can transform from one type to another. The former have multiply connected backbones, which deform as elastic rods, while the latter have at least one soft junction and deform without elastic resistance.

### 5.2. Relation with processes of sludges treatment

Some studies have tried to link rheological properties of concentrated wastewater suspensions with operating parameters of a stirring process [48]; a pre-treatment process [5] or a treatment process [21] of these sludges (see Table 5). Roche et al. [48] have proposed a correlation for the critical speed for off-bottom suspension as a function of the yield stress of activated sludges. Wichmann and Riehl [21] have worked on mineral wastewater sludges for agricultural purposes. Such suspensions have to be conformed to the German law (i.e. minimum yield stress of 25 kPa). The influence of the water content on the dewaterability of these sludges (linked to the CST value) and on the value of their yield stress has then been studied. Abu-Orf and Dentel [5] have followed the physico-chemical conditioning of wastewater sludges with polyelectrolytes ions, which is an important step that allows to enhance sludges dewaterability. They have studied the effect of the polymer dose, of the stirring rate in the flocculation tank and of the recycled filtrate flow on both the rheological properties of sludges measured with an on-line rheometer called the "zenofloc system" and on their filterability (CST).

### 5.3. Relation with oxygen mass transfer in bioreactors

As concerns the influence of the apparent viscosity of a liquid phase or of a suspension on oxygen mass transfer in an aerated medium, some studies (Table 6) have shown that the apparent viscosity value influences the global coefficient of mass transfer. Kawase and Moo-Young [49] have demonstrated in 1991, for yield stress fluids, that the global mass transfer coefficient decreased as $\eta_a^{-0.25}$. Other authors [8,50,51] have confirmed the same type of relation. However, the value of the negative exponent of $\eta_a$ varies greatly from one work to another, according to the medium under consideration. In all cases, it can be said that that the global mass transfer coefficient decreases when the apparent viscosity increases: $k_L a \propto \eta_a^{-z}$ with $0.25 < Z < 0.84$.

Such correlations are useful for the optimisation of the working conditions of bioreactors used in the field of wastewater purification as well as in the food industry. In

Table 5
Correlations between processes operating sludges and the rheology of dense suspensions

| Ref | Studied suspensions | rheology (rheometer) | Observed correlations |
|---|---|---|---|
| [48] | Activated sludges SS = 5.8 to 17 $g.L^{-1}$ | VP (CCdcR) | Variation of the critical speed for off-bottom suspension $Nrs = A_{22} \tau_Y^{0.33}$ |
| [21] | Concentrated mineral sludges SS=250 to 600 $g.L^{-1}$ | VP « vane shear test » | ↑ of the suspended solid concentration ⇔ ↑ of the yield stress measured by the « vane method » |
| [5] | Digested sludges under conditioning $\dot{\gamma} = 0$ to $70\,s^{-1}$ | VP | ↑ polymer dose (until optimal dose)<br>↑ polymer fixed onto the surface of particles (until maximum covering)<br>↑ high of the pick of the rheogram (= $\tau_Y$)<br>↓ filtration time<br>⇔ Optimisation of the dewaterability<br><br>↓ Agitation in flocculation tank<br>↓ Optimal dose of polymer |

Table 6
Correlations between apparent viscosity and oxygen transfer

| Ref. | Studied fluids | Rheology (Rheometer) | Effect of the apparent viscosity on the $k_L.a$ value |
|---|---|---|---|
| [49] | Carbopol solution and mycelia suspensions | VP | $k_L.a = A_{23} \frac{U_{sg}^{0.98}}{\eta_a^{0.25}}$ |
| [50] | Microbial culture | VP | $k_L.a = A_{24} U_{sg}^{0.33} \eta_a^{-0.66} \varepsilon^{0.66}$ |
| [8] | Domestic and industrial activated sludges or laboratory sludges SS=3.3 to 42 g.L$^{-1}$ | ST $10^{-2}$ to $10^{3}$ s$^{-1}$ (CCdcR) | $k_L.a = A_{25} \eta_a^{-B_{23}}$ with $A_{23}$ et $B_{23}$ varying with the type of sludge |
| [51] | Aqueous solutions of CMC and guar | ST $10^{-1}$ to $10^{2}$ s$^{-1}$ (CCR) | $k_L.a = A_{26} \eta_a^{-B_{26}}$ with $0.4 < B_{26} < 0.6$ |

activated sludge wastewater plants, the main problem is to determine the optimal suspended biomass concentration in order to treat a maximum of polluted water, without altering the $k_L a$ value and thus the purification ability of the medium. We can finally remark that the increase in the concentration in solid matter of the medium is also limited by filtration purposes.

## 6. Conclusions

In this study, a review of the literature devoted to the rheological characterisation of activated and sewage sludges has been performed. Such sludges are suspensions of particles with various shape and concentration, in particular, according to the origin of the material (activated sludge, digested sludge, etc.). This implies that various rheological behaviours can be encountered, as far as wastewater sludges are concerned. In this context, we have proposed to synthetise the main rheological methods used in rheometry of concentrated suspensions, discussing the advantages and disadvantages of each configuration. The main rheological equations used by the different authors to represent the shear-thinning, viscoplastic or viscoelastic properties of wastewater sludges are also reviewed in Tables 1–3. The thixotropy that frequently appears in such concentrated flocculated suspensions is also discussed. Finally, particularly attention is paid to articles that have tried to establish a link between rheological properties on one hand, and on the other hand the physico-chemistry of the flocs (Table 4); working conditions of processes in which sludges are operated (Table 5); or the oxygen mass transfer in bioreactors (Table 6). All these works have clearly demonstrated the difficulties in performing reliable rheological measurements on activated or sewage sludges. This fact is notably due to the great variability of sludges samples but also to the rheometric devices and methods themselves. However, for future works, rheology still appears to be an interesting tool to contribute to the characterisation of non-Newtonian sludge flows. Such a rheological characterisation is essential to achieve a better understanding of the structure and interparticle interactions inside bacterial flocs that is needed to optimise all kind of processes involving sludges. These processes are notably the separation processes (settling, dewatering, drying) the oxygen or substrate mass transfers in bioreactors of wastewater treatment plant and finally the transformation processes for sewage sludges reuse.

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