

Chemical Engineering Journal 142 (2008) 40-47

Chemical Engineering Journal

www.elsevier.com/locate/cej

In situ triphasic rheological characterisation of activated sludge, in an aerated bioreactor

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Received 17 July 2007; received in revised form 21 September 2007; accepted 5 November 2007

Abstract

This study is devoted to the triphasic *in situ* rheological characterisation of an activated sludge, with total suspended solid (TSS) ranging from 10 to 35 g/L, and operated in a bioreactor under different stirring and aeration rates. The originality of this work is that flow properties are directly measured inside the bioreactor. Under low mechanical shear rates (below 0.1 s^{-1}) the internal structure of the suspension (configuration of structural units) is driven by the air plume. Due to the shearing of air bubbles, apparent viscosities are strongly lowered by the injection of air but almost independent of the quantity of air (in the range 2–6 L/min). Under high mechanical shear rates (above 100 s^{-1}), the configuration of structural units (i.e. flocs in the case of activated sludge) is only dependent on the mechanical shearing and totally independent on the presence or absence of air. The viscosity of the broth is constant whatever the air flow rate is (0–6 L/min). We also observe a decrease in shear-thinning properties of aerated suspensions compared to non-aerated one, with a plateau above 2 L/min. The effect of TSS at constant air flow rate shows that an increase in TSS induces an increase not only in apparent viscosities but also in shear-thinning properties.

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Keywords: In situ rheology; Apparent viscosity; Activated sludge; Aeration rate; Bioreactor

1. Introduction

During the last decades, the question of water pollution has taken worrying proportions whereas, at the same time, water consumption increased mainly due to the demographic explosion. Industrialised countries now aim at reducing the pollution of water to preserve their water supply. This latter point particularly implies to optimise the treatment processes of both industrial and domestic wastewater.

Among the different wastewater treatment processes, activated sludge biological treatments are widely spread, due to their high efficiency and low operation cost. During such treatments, polluted influents are mixed in an aeration tank containing aerobic microorganisms. A great part of the organic pollution is then degraded by the biomass. In such an aeration tank, the suspension mainly composed of microorganisms suspended in wastewater (at solid contents generally ranging from 2 to 5 g/L in a conventional process and almost up to 15 g/L for a basin

coupled with a membrane solid-liquid separation) is called activated sludge. The next step consists in the separation of the solid phase from purified water, either in a settling tank (conventional processes) or in a membrane (membrane bioreactor (MBR) processes). The main advantage of MBR is to allow higher solid working concentrations than in the conventional process where a limitation occurs due to the poor ability of concentrated sludge to settle. In both types of processes, a great part of the power consumption of the plant is used: (i) to ensure a sufficient aeration to the microorganisms (in MBR the aeration must also limit the membrane fouling) and (ii) in the case of MBR, to separate the solid phase from the liquid phase. The optimisation of both aeration and separation steps, is thus of prime importance. For this purpose, it is necessary to take into account the hydrodynamic of aeration and settling tanks, which mainly depends on the rheologically complex, non-Newtonian nature, of activated sludge suspensions.

Rheological characterisation has often been used as a precious investigation tool to describe the flow properties of activated and sewage sludge suspensions [1–7].

Some studies have furthermore tried to link the flow properties of sludge suspensions to their processing [8–16]. However,

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^{1385-8947/\$ –} see front matter 0 2007 Elsevier B.V. All rights reserved. doi:10.1016/j.cej.2007.11.007

Nomenciature	
d	impeller diameter (m)
Κ	consistency index $(Pa s^n)$
Kp	laminar constant of the power curve
$\dot{K_{MO}}$	Metzner–Otto constant
п	flow index
Ν	agitation rate (s^{-1})
$N_{\rm p}$	dimensionless power number
P	agitation power (W)
$Q_{ m air}$	air flow rate ($L \min^{-1}$)
Re	dimensionless Reynolds number
TSS	total suspended solid (g L^{-1} or kg m ⁻³)
Greek letters	
Ϋ́	mechanical shear rate (s^{-1})
Ϋмо	Metzner–Otto shear rate (s^{-1})
$\dot{\gamma}_{bubbles}$	shear rate due to the air plume (s^{-1})
η_{a}	apparent viscosity of the broth (Pas)
ρ	density (kg m^{-3})
τ	shear stress (Pa)

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in these studies, the rheological properties of sludge suspensions have almost always been determined *ex situ*, sampling the sludge from the process and introducing it inside a classical rheometer. In the case of non-Newtonian materials such as sludge suspensions, exhibiting complex rheological properties such as viscoelasticity, and thixotropy, we may expect more relevant measurements by minimising sludge manipulation and thus performing an *in situ* rheological characterisation. In particular, as sludge flow properties depend on a lot of parameters such as sludge previous history (pre-shearing rates and times) it seems obvious that *in situ* rheological measurements will give access to more accurate properties than measurements performed after sampling, storing and introducing the sludge in a classical rheometer.

Furthermore, in the case of aerated processes, *in situ* measurements will allow to access the flow properties of the suspension under aeration, which is not possible in a conventional rheometer.

In the literature, only Abu-Orf and Dentel [8], in the case of conditioning processes, have tried to determine the optimum polymer concentration based on shear stress–shear rate characteristic curves directly measured inside the conditioning vessel (measurements on the solid–liquid system), as a function of the polymer dose. However since we have measured viscosity curves of aerated activated sludge as a function of air flow rates, this is not possible to compare their results with the one presented in this paper.

The rheological properties of sludge suspensions encountered at different stages of water purification process are also known to be correlated to the economical yield of these operations. In particular, in the case where the biological purification of water is performed in a MBR, the total suspended solid (TSS) concentration of the sludge can be increased compared to a classical aeration basin (>10 g/L), since the separation step is no more limited by the sludge ability to settle. Such an increase in sludge solid concentration induces an increase in apparent viscosity of the suspension that can strongly affect the oxygen transfer in the liquid phase, leading to insufficient aeration rates of the micro-organisms and thus to low purification yields [17]. As an example, the cost of aeration in a wastewater treatment plant (WWTP) has been evaluated at about 60% of the overall running cost [18].

However, because of the complex rheological behaviour of biological suspensions, the rheological characterisation of wastewater sludge, in relation to its processing in aeration bioreactors of different geometries [19,20] or MBR [13–16] has only been recently studied in the literature. Furthermore, to our knowledge, it has never been done using *in situ* measurements. As a consequence, the rheological properties of activated sludge have also been poorly taken into account in aeration tanks modelling.

In such a context, this work is devoted to the in situ rheological characterisation of an activated sludge sampled in a municipal WWTP, concentrated in order to achieve TSS concentrations from 10 to 35 g/L and aerated with different air flow rates in a mechanically stirred bioreactor. The 10-35 g/L range in TSS has been chosen as a potential range of solid content inside an MBR. Indeed, in most of the studies, the mean solid concentration in an MBR is, in the current state of the art, in the range of 3–15 g/L. But some recent studies have worked in a higher solid concentration range reaching 20 g/L [21] or even 30 g/L [22]. Furthermore, it is relevant to investigate the rheological flow properties of aerated activated sludge in a wide range of solid concentrations, since in the case of submersed MBR, the solid concentration can be much more higher close to the membrane, due to the accumulation of biomass [23]. The knowledge of how an aerated suspension will flow in this range of solid concentration can thus be of prime importance to understand phenomena occurring close to the membrane, such as fouling or clogging phenomena.

By *in situ* rheological characterisation, we mean that the apparent viscosity of the sludge has been directly determined inside the bioreactor on the triphasic medium (sludge suspension under aeration). This is the originality of the present work, since in previous works dealing with the rheological characterisation of activated sludge in relation to their processing in wastewater treatment plant (WWTP), sludge has always been sampled from the process to further perform its rheological characterisation in a "classical" rheometer. What has been characterised in these previous works is thus the flow properties of the sampled suspension, while we have here measured the flow properties of the suspension under aeration, inside the process. We thus expect to achieve a better representation of the apparent viscosity of an activated sludge under aeration and mechanical stirring inside an aeration basin or a MBR.

Sludge rheological properties, in terms of viscosity curves have been determined as a function of (i) the solid content of the sludge in terms of TSS concentrations, the investigated range of TSS is 10–35 g/L; (ii) the stirring rate of the bioreactor, the range of variation is from 0.0476 to 11.95 or 0.0476 to 257 rpm depending on the TSS content; (iii) the air flow rate of aeration, the air flow rate varied from 0 to 2 or 0 to 6 L/min, also depending on the TSS content.

The Ostwald power law model has then been used to represent the viscosity curves. The two parameters of this model, i.e. the consistency index (K in Pa sⁿ) and flow index (n) have been calculated using a simple linear regression in log–log scale. Variations of these two parameters as a function of TSS and air flow rates are then discussed.

2. Experimental

2.1. Activated sludge

The activated sludge used in this work comes from an urban WWTP of 165,000 eq. inh. (City of Aix en Provence, France). The sludge sampled in the recirculation loop between aeration basins and secondary settlers is at about 3 g/L in TSS.

In order to obtain TSS concentrations ranging from 10 to 35 g/L, sludge has been concentrated by soft (gravimetric) filtration using simple coffee filters (average size of pores around $100 \,\mu\text{m}$).

2.2. Bioreactor and in situ rheological device

The bioreactor is a 2.65 L Plexiglas vessel of diameter 0.15 m. It is equipped with a double helical ribbon impeller (HRI) rotating rather close to the wall (d/D = 0.63). It can be underlined that, due to the high pumping capacity of the HRI [24], in the following experiments the pre-sheared sludge suspensions were flowing, even at the smallest rotation speed, and even in the far corners of the vessel (no dead zones). The bioreactor is also aerated by injection of compressed air through a soft porous membrane ensuring fine bubbles diffusion through the whole bottom surface, without clogging (as presented in Fig. 1). The temperature is kept at 20 ± 0.2 °C due to circulation of water in a coil immersed in the vessel. The temperature control coil has not been represented in Fig. 1 for clarity purposes.



Fig. 1. Schematic representation of the experimental set-up.

The apparatus used to perform *in situ* rheological characterisation is a torque-meter Rheomat 30 (CONTRAVES) measuring the agitation torque due to the rotation of the HRI in the aerated sludge. The rotation speed has varied between 0.0476 and 11.95 rpm for the 10 g/L sludge or between 0.0476 and 257 rpm for more concentrated suspensions, in order to roughly stay in the laminar region even under aeration. The whole set-up is shown in Fig. 1.

Before each viscosity curve measurement, zeroing the torque has been performed with the HRI rotating in air at the maximum rotation speed (257 rpm), under different air flow rates from 0 to 6 L/min sparged through the porous membrane. It can be underlined that, whatever the gas flow rate from 0 to 6 L/min, the zero torque value has not been affected by the presence of air sparged at the bottom of the vessel. This is an important point, so that it can be considered that, when rotating the HRI in the sludge suspension, torque variations under different air flow rates are basically due to broth viscosity changes and not to torque measurement disturbances due to the injection of air below the stirrer. Then, at each rotation speed, the torque due to the suspension viscosity has been measured as follows: the sludge has first been pre-sheared during 30s at 257 rpm (the torque recorded at this rotation speed was still constant during the whole experiment for a given sludge), then the rotation speed has been fixed at the desired value and the torque has been recorded during 30 s. The value of the torque remained stable during this time lag, ensuring that, even at the lowest shear rate, the whole suspension (no dead zone) was already in its flow region and not in the region where the solid structure can rebuilt even under finite but sufficiently small shear rates [25].

Viscosity curves measurements have then been performed using the Metzner–Otto's principle [26] that defines an apparent viscosity η_a (Pas), based on the generalisation for non-Newtonian media of the relation existing (in the laminar region) in an agitated vessel between the dimensionless power number (N_p) and the Reynolds number (Re) (Eq. (1)).

$$N_{\rm p} = \frac{P}{\rho N^3 d^5} = \frac{K_{\rm p}}{Re} = \frac{K_{\rm p} \eta_{\rm a}}{\rho N d^2} \quad \text{then} \quad \eta_{\rm a} = \frac{P}{K_{\rm p} N^2 d^3} \tag{1}$$

where *P* (W) is the mechanical agitation power related to the agitation torque *C* (Nm) and to the mechanical rotation rate *N* (s^{-1}) by the following equation:

$$P = 2\pi N C \tag{2}$$

where K_p is dimensionless and corresponds to the laminar power curve constant.

At a rotation rate value of *N* corresponds an effective shear rate $\dot{\gamma}_{MO}$ (s⁻¹) related to the rotation speed of the impeller by the Metzner–Otto dimensionless constant characterising the stirrer geometry (Eq. (3)).

$$\dot{\gamma}_{\rm MO} = K_{\rm MO}N\tag{3}$$

A calibration of the set-up has been carried out with two model fluids at 20 °C to determine the K_p and the K_{MO} constants. The fluids used are a Newtonian solution of pure glycerol and an aqueous solution of guar at 1 wt.%. In the range of shear rates



Fig. 2. Viscosity curves obtained with the bioreactor equipped with the HRI (R30 HRI) and with classical rheometer and geometry (AR550 CC).

studied here, the guar solution flow is still in its shear-thinning region. An average value of the K_p and K_{MO} constants of the setup have thus been determined at respectively 393 and 50. The Metzner–Otto method has been widely used in chemical engineering researches to estimate the average shear rate in agitated vessels [27–32]. However, this method makes approximates, notably by supposing that the K_{MO} and thus the average shear rate in the vessel are constant at a given rotation speed, even in a non-Newtonian media.

To ensure that the $K_{\rm MO}$ value quoted above is precise enough, we have compared for a 35 g/L sludge (highest TSS studied here) under no aeration, viscosity curves given by the set-up presented in this study and a classical rheometer (AR550, TA instrument), equipped with a classical small gap Couette geometry ($R_i = 14$ mm, $R_e = 15$ mm). The results obtained are presented in Fig. 2.

We can see in this figure that the viscosity curves obtained with both classical rheometer and geometry and our set-up are sufficiently close to each other (even at small shear rates), to validate the assumption that the K_{MO} can be kept at a constant value of 50, in the range of rheological properties and shear rates investigated here.

3. Results and discussion

3.1. Effect of the air flow rate at constant solid content (TSS) on the viscosity curve

In situ viscosity curves are presented in Fig. 3, in terms of apparent viscosity as a function of shear rate (in log–log scales) for aeration rates varying between 0 and 2 L/min for the smallest TSS (10 g/L, a) and 0 and 6 L/min for other solid concentrations (15–35 g/L, b–f).

In this figure it can first be seen that the effect of aeration on the apparent viscosity of the broth is important at low shear rates. In this low shear region, the apparent viscosity of aerated suspensions is divided by one order of magnitude (factor 6-20 for the apparent viscosity at lowest shear rate) when compared to the non-aerated suspension (0 L/min curves). However, this decrease in viscosity of the aerated suspension seems to tend towards a plateau as a function of the value of the air flow rate. Indeed, if we look more carefully for instance at the 10 g/L viscosity curve (Fig. 3a), we can observe that the decrease in viscosity due to the injection of air at low shear rate is important for the first flow rate (1 L/min) but relatively less important for the next rates (1.5 and 2 L/min). This is also the case for other TSS concentrations, the effect of air injection on apparent viscosity seems to tend to a limit, as for high gas flow rates (2–6 L/min), the curves are almost superimposed (Fig. 3b–f).

In the high shear region, we can first observe for TSS between 15 and 35 g/L that the last point (or two last points) indicate the beginning of the transitional region, since the viscosity stops decreasing and goes up again. However, the most interesting result comes from the fact that the effect of air injection on apparent viscosity decrease is less important than in the low shear region (Fig. 3a) and even that there is no viscosity decrease in the presence of air above a certain shear rate, whatever the gas flow rate value (Fig. 3b–f). Indeed, it is clearly seen in those figures corresponding to TSS value from 15 to 35 g/L, that at sufficiently high shear rates (above about 100 s^{-1}), the apparent viscosity value is independent of the presence of air. In Fig. 3a, the highest shear rate value is only of 10 s^{-1} but it can already be seen that the aerated viscosity values tend towards the non-aerated ones.

At least on a qualitative point of view, a comparison of these results can be proposed referring to the work published in the field of power consumption characterisation in non-Newtonian aerated media (this comparison can indeed only be qualitative since in these studies the stirrer of reference is a Rushton turbine impeller (TD6), while we have worked here with a closeclearance, basically laminar, impeller). In aerated stirred vessels, it is well known that the injection of air induces a power reduction (compared to the un-aerated power) in a certain Reynolds number range [33]. When the liquid phase is furthermore rheologically complex, an inhibition of the effect of the gas flow rate value has also been observed (in a certain Reynolds number range, which value depends on the overall rheological properties of the medium) [34,35]. In our case, we also observe, in the low shear region, that the air injection induces a power reduction and thus a reduction in the apparent viscosity of the aerated broth. However, whatever the gas flow rate value (above 2 L/min), the apparent viscosity values are quite constant in the presence of air. In our bioreactor, where a non-Newtonian complex biological suspension is operated under different HRI rotation speeds and different gas flow rates, we also observe a partial inhibition (above 2 L/min) of the effect of the gas flow rate value at low rotation rates (low shear region).

On a structural point of view, it is well known that number of complex fluids can be roughly described as concentrated dispersions of structural units, i.e. flocs or aggregates of flocs in the case of biological sludge. Indeed, structural units have been described by Quemada [36] as follows: in a number of complex concentrated dispersions, small primary elements form larger groups (clusters, aggregates, flocs, clusters of flocs, etc.). These groups are called structural units and have a size which depends on the applied shear through the hydrodynamic stress acting on the structural units. In the case of our experiments, we can then easily imagine that at low mechanical shear rates (low mechanical rotation speeds), the injection of air bubbles interacts with



Fig. 3. In situ viscosity curves at constant TSS: (a) 10 g/L; (b) 15 g/L; (c) 18 g/L; (d) 24 g/L; (e) 28 g/L; (f) 35 g/L.

the configuration of sludge flocs and strongly affects the steady state of structural units of the material. In these conditions, the internal structure of the suspension is mainly controlled by the non-mechanical shear due to air plume and so the apparent viscosity value. As an example, in Fig. 2b, the injection of air induces, at lowest mechanical shear rate, a reduction in viscosity from roughly 24 to 4 Pa s. To obtain an identical reduction under mechanical shearing, we can see that an average shear rate of 0.5 s^{-1} has to be applied (doted lines). We can then estimate the shear due to the rise of air bubbles in the suspension at a value of about 0.5 s^{-1} in this precise case ($\dot{\gamma}_{\text{bubbles}}$). Above a certain gas flow rate (2 L/min), a further increase in air flow rate does not affect suspension apparent viscosities, the internal structure

seems to tend to a steady state in the presence of air and under low mechanical shearing rates.

At high Reynolds number, i.e. in the turbulent region, it has also been described in works on gas dispersion in agitated tanks [33] that the power drawn by a stirrer (Rushton turbine) in a non-Newtonian aerated medium exhibits no power reduction at all due to air injection, the aerated power curve being superimposed to the un-aerated one in turbulent flows. In our case, we also make qualitatively identical observations. Above an average mechanical shear rate of 100 s^{-1} (end of the laminar region in the case of our bioreactor equipped with the HRI), the agitation torque, and thus the apparent viscosity of the aerated broth, is no more affected by the presence of air. The apparent viscosity values are constant without air injection (0 L/min) or in the presence of air, whatever the flow rate (2–6 L/min). In this "high" mechanical shear region, the hydrodynamic of the bioreactor is now only controlled by the mechanical stirring and no more dependent on the gas plume. On a structural point of view, this implies that the configuration of sludge structural units is only dependent on the mechanical shear rate and totally independent on the soft shear due to air bubbles. As a consequence, under sufficient mechanical stirring, the agitation torque and thus the apparent viscosity is constant whatever the air flow rate value (0–6 L/min).

3.2. Effect of the solid content (TSS) at constant air flow rate on the viscosity curve

In situ viscosity curves are presented in Fig. 4, in terms of apparent viscosity as a function of shear rate (in log–log scales) for TSS varying from 10 to 35 g/L and air flow rates 0 L/min (a), 2 L/min (b), 4 L/min (c) and 6 L/min (d).

In this figure a classical effect of the total suspended solid content can be observed. There is indeed an increase in apparent viscosity as the TSS concentration increases for each flow rate value. We can also note that the increase in viscosity with the increase in TSS is more important in diluted suspensions (for example between 10 and 15 g/L) than in more concentrated one (for instance between 28 and 35 g/L).

This is a well-known behaviour for sewage sludge that has already been observed by classical *ex situ* measurements in a great number of studies [1-3,7,9,10]. At high solid content, structural units of the suspension may be larger in size and

closer to each other, leading to higher apparent viscosities of the material.

3.3. Effect of the air flow rate and solid content on Ostwald rheological parameters

For each viscosity curve presented in this paper the Oswald shear-thinning model has been applied. The two resulting parameters, i.e. the consistency index K (Pa sⁿ) and flow index n have been deduced by simple linear regression in log–log. Values of the r^2 of the fit of experimental data by the Ostwald model vary from 0.92 (10 g/L viscosity curves under 2 L/min) and more than 0.998 for most of the viscosity curves under higher TSS.

Fig. 5 then presents variations of the Ostwald parameters (a, flow index; b, consistency index) as a function of air flow rate for the different TSS.

In this figure, we can observe that, for all TSS values, the injection of air induces an increase of n and a decrease of K, i.e. a decrease in the shear-thinning properties of the broth. This reduction of the shear-thinning properties as a function of the gas flow rate also seems to tend to a plateau for air flow rates above 2 L/min, as it has previously been observed for the apparent viscosity of the broth in Fig. 2. For certain solid concentrations, the plateau is no always reached, but we may suppose that these curves would follow an identical evolution and that a plateau would be achieved at higher air flow rates. On a structural point of view, this is a direct consequence of what has already been explained above. Non-aerated suspensions (0 L/min) exhibit a certain level of shear thinning. Aerated suspensions (2-6 L/min)



Fig. 4. In situ viscosity curves at constant air flow rate: (a) 0 L/min; (b) 2 L/min; (c) 4 L/min; (d) 6 L/min.



Fig. 5. Ostwald model parameters evolution as a function of air flow rate: (a) flow index (n); (b) consistency index $(K, \operatorname{Pas}^n)$.

low shear viscosities are lowered by the presence of air bubbles (interaction between air bubbles and structural units configuration at low mechanical shear rates), while their high shear viscosities are the same as un-aerated suspensions. As a consequence, the slope of the log–log aerated viscosity curves (n - 1) is reduced compared to the un-aerated one and *n* increases in the presence of air. In the same way, the consistency of the broth decreases in the presence of air, and so the whole shear-thinning properties.

On the contrary, as concerns the effect of TSS content, it is observed that an increase in TSS induces an increase of the shear-thinning properties (increase in K and decrease in n). This is a classical result that can be explained by the fact that the more the solid content is high, the more the structural units of the material are important in size and then able to release high quantities of water under shear. A more concentrated sludge will then exhibit more pronounced shear-thinning properties.

4. Conclusion

In this study, *in situ* rheological characterisations of activated sludge with various solid content (TSS ranging from 10 to 35 g/L) have been investigated under different air flow rates (0 to 6 L/min). The apparatus used is a bioreactor equipped with a close clearance axial impeller (HRI), and coupled with a torque-meter.

Viscosity curves have been plotted for each TSS, as a function of air flow rate.

The Ostwald model has been used to represent the shearthinning behaviour of such aerated and un-aerated suspensions.

The most important result comes from the fact that at low shear rates (below 0.1 s^{-1}) the internal structure of the suspension (configuration of structural units) is driven by the air plume. As a consequence, low shear viscosities are strongly lowered by the injection of air but almost independent of the quantity of air (above 2 L/min). On the contrary, at high shear rates (above 100 s^{-1}), configurations of structural units are only dependent on the mechanical shearing and totally independent on the presence or absence of air. The viscosity of the broth is constant whatever the air flow rate is (0–6 L/min). A direct consequence of these observations is a decrease in shear-thinning properties of aerated suspensions compared to non-aerated one, with a plateau above 2 L/min.

Finally, the effect of TSS content at constant air flow rate shows, as in the case of classical *ex situ* measurements, that an increase in solid content induces an increase not only in apparent viscosities but also in shear-thinning properties.

These results are important and show that, in bioreactors such as MBR or aeration basins, in the low mechanical shear region (for example when no mechanical stirring occurs), the apparent viscosities of activated sludge (in the TSS range 10-35 g/L) are strongly affected by the presence of air, due to the interaction of air plume with the configuration of structural units of the suspension. On the contrary, under sufficient mechanical shear, the flocs configuration appears only driven by mechanical shearing and viscosities are totally independent on the presence of air. As a consequence in situ viscosities measurements under aerated conditions allow providing a more accurate description of the internal state of the activated sludge suspension under given air flow and shearing rates. Further taking into account this in situ viscosity values in the modelling of different phenomena such as oxygen transfer, should then allow achieving a better representation of this important process. In this goal, the experimental set-up presented in this work will next be used to perform coupled in situ measurements of apparent viscosities and oxygen transfer conductance (K_{1a}) as a function of different process parameters.

Acknowledgment

The authors are indebted to the WWTP of Aix en Provence-La pioline (France) for providing activated sludge suspensions.

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