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# Rheological characterization of mixed liquor in a submerged membrane bioreactor: Interest for process management

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## Abstract

Rheological analyses of a submerged membrane bioreactor mixed liquor were performed in the aim of characterizing the mixed liquor present in the bioreactor and thus proposing a process management. These analyses pointed out that the mixed liquor was characterized by its viscoplastic property, which leads to a possible restructuring ability when a shear stress lower than the yield stress is applied. As the shear stress in the bioreactor is essentially generated by coarse bubbles, specific experiments were carried out in which coarse bubbles were injected in an intermittent way. The results of these experiments showed that this method could avoid damage to the mixed liquor. So working with intermittent coarse bubbles is useful to prevent fouling, keep good flocculation and minimize the energy cost.

*Keywords:* Submerged membrane; Bioreactor management; Rheology; Hydrodynamic shear stress; Aeration

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## 1. Introduction

There is increasing interest in the introduction of membranes into systems for residual water treatment and this has led to proposals for wide-reaching investigations, from biochemistry to bioprocess control. Compared to conventional processes, membrane filtration devices produce water of constant and higher quality; the problems linked to a lack of sludge sedimentation are avoided and the water retention time can be reduced, thus limiting the volume of the bioreactors. However, a major limitation of membrane filtration lies in membrane fouling. Thus, many investigations focus on this problem. Some research is more focused on how membrane fouling occurs and seeks to understand the phenomena involved in the formation of membrane fouling [1,2]. Other investigations concentrate on the way to prevent membrane fouling by an accurate filtration mode [3–5]. In the case of membrane bioreactors, it is recognized that aeration is a key parameter for the management and prevention of membrane fouling.

An efficient aeration mode generates shear stress, which can release deposit from the membrane or at least prevent the suspended solids from depositing on the membrane [6]. Nevertheless, Gui et al. [7] have recently shown that a too high aeration flow rate could have a bad effect, since it could be the cause of a breakdown of the flocculation.

In addition, aeration is reported to be the most expensive parameter in terms of energy consumption [8,9]. Eighty percent of the total energy cost in an SMBR can be due to aeration. Accurate management of this parameter thus appears to be essential. The aim of this work is to study the impact of aeration on the characteristics of the mixed liquor in a submerged membrane bioreactor. The work is specially focused on the response of the mixed liquor to various shear stresses. Rheological analysis was used for this investigation. In general, the rheological properties of activated sludge are reported to be pseudoplastic or pseudoplastic with initial yield stress [10,11]. Hasar et al. observed the same result for an activated sludge in a submerged membrane bioreactor (SMBR) [12]. But none of these studies have used these results to propose a method for process management.

The approach presented in this paper consisted first in finding a procedure to obtain repeatable results, especially looking at the thixotropy of the mixed liquor. Then a conventional rheo-

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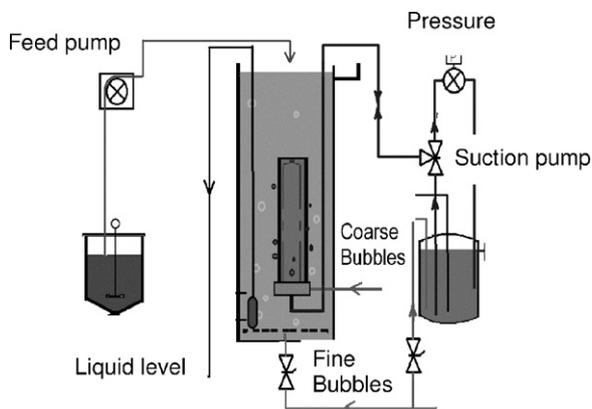


Fig. 1. Experimental device.

logical characterization was carried out for SMBR mixed liquor obtained with a synthetic effluent and with domestic wastewater. In the former experiment, a comparison was made with the rheological characterization of mixed liquor from an activated sludge process to be sure that the properties were specific to SMBR mixed liquor. The results obtained were analyzed from the process efficiency point of view in order to propose improved process management. The enhanced functioning conditions were tested first with a simulation of the process operation on the rheometer and then on the process itself.

## 2. Material and methods

### 2.1. Membrane reactor

The experimental study was performed on a 10 L submerged membrane bioreactor (Fig. 1). A bundle of U-shaped poly-sulfone hollow fiber membranes provided by Polymem S.A. (Toulouse, France) was submerged in the bioreactor. The membrane characteristics are reported in Table 1.

Transmembrane pressure, pH, temperature, flow rate and mixed liquor volume were continuously recorded with a Dasy-Lab control program.

### 2.2. Effluent

The SMBR was initially filled with activated sludge from the wastewater plant of Toulouse-Ginestous. Then two kinds of effluent were tested: a synthetic effluent and a domestic wastewater.

The synthetic effluent was composed of a cooking sauce (Viandox), diluted with tap water and complemented with nitrogen salt. The composition of the synthetic effluent is indicated in

Table 1  
Membrane characteristics

Pore size ( $\mu\text{m}$ )	0.1
Filtration area <sup>a</sup> ( $\text{m}^2$ )	0.3
Internal/external diameters (mm)	0.4/0.7
Initial permeability ( $\times 10^{-5}$ L/h Pa $\text{m}^2$ )	300

<sup>a</sup> Total external surface of all the fibres.

Table 2  
Synthetic effluent characteristics

Chemical oxygen demand, COD (g/L)	130
Biological oxygen demand, BOD (g/L)	85
Total Kjeldahl nitrogen, TKN (g/L)	8.75
Ammoniac nitrogen, N-NH <sub>4</sub> (g/L)	1.9
Phosphorus, P-PO <sub>4</sub> (g/L)	1.1

Table 2. There were no suspended solids in this effluent. All suspended solids were generated by the biomass activity. The tap water and the complemented substrate (Viandox) were introduced into the reactor separately. The tap water flow rate was controlled by the liquid level in the reactor, whereas the Viandox solution fed the reactor continuously. The Viandox solution flow rate was adjusted to the sludge concentration in order to keep a constant daily carbon loading of 0.4 g COD/g MLSS day.

The wastewater was a domestic wastewater without additional industrial wastewater. The COD of this effluent was systematically determined once or twice each week. COD ranged from 300 to 600 mg/L.

### 2.3. Operating conditions

Filtration was operated at constant flux. Fouling was then directly correlated with the variation in the transmembrane pressure. An increase of the transmembrane pressure absolute value corresponded to a pressure drift due to membrane fouling. Permeate was drawn through the membrane by a volumetric pump under a constant flux of 10 L/(m<sup>2</sup> h). The transmembrane pressure lay between 0 and 1  $\times 10^5$  Pa. For convenience of presentation, all the results are represented using the absolute value of transmembrane pressure (instead of the negative pressure which was recorded).

Fine bubbles were continuously injected into the bottom of the reactor at 50 L/h through a “Flygt” membrane to bring the necessary oxygen to the biomass and to avoid sludge settling. Unless otherwise specified, the sludge retention time (SRT) was 20 days with an MLSS concentration of 8.5 g/L  $\pm$  0.5 g/L.

### 2.4. Fouling control

All the experiments were performed at a constant flow rate of 10 L/(m<sup>2</sup> h) taking previous results carried out in the same device [13] into consideration. Filtration was operated in an intermittent sequence of filtration (15 min)—no suction (5 min), with periodic backflushing (1  $\times 10^5$  Pa, 1 min every 60 min) as presented in Fig. 2 [13]. The hydraulic retention time varied with filtration sequences and ranged from 3.8 to 6.7 h.

To control membrane fouling, deposit on the membrane surface had to be avoided or removed. This can be done by generating shear stress along the fiber or/and in the mixed liquor close to the fiber. For this purpose, coarse bubbles were injected close to the membrane so as to provide enough shear stress in this area (Fig. 3). Preliminary investigations [14] showed that an aeration flow rate of 50 L/h was enough to prevent fouling in 2 days of experiments. The aeration could be injected either in intermittent mode or continuously under the control of the Dasy-

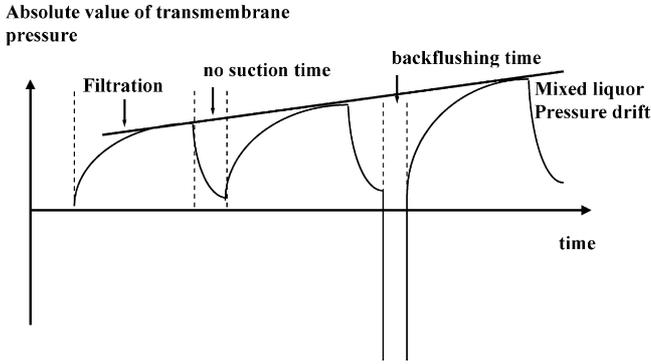


Fig. 2. Transmembrane pressure profile.

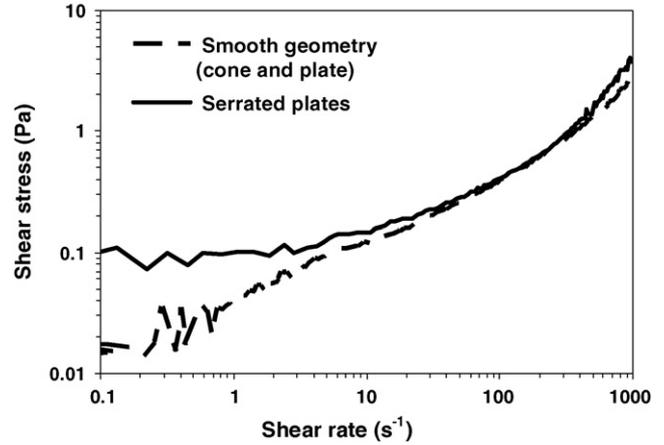


Fig. 4. Rheological experiments with and without serrated plates.

lab control program. Further experiments were then conducted with different injection modes and with a maximum aeration flow rate of 50 L/h.

In a first experiment, aeration was injected continuously at 50 L/h (flux of 166 L/(m<sup>2</sup> h)). In a second experiment, an instantaneous aeration flow rate of 200 L/h was applied in an intermittent mode, for 1 min every 6 min (flux of 111 L/(m<sup>2</sup> h)). In a third experiment an instantaneous aeration flow rate of 370 L/h was applied in an intermittent mode, for 1 min every 12 min (flux of 100 L/(m<sup>2</sup> h)). At the end of each experiment, the fouled membrane was taken out of the bioreactor and replaced by a new one in order to start from the same initial state.

### 2.5. Membrane regeneration

When the membrane became fouled, it was taken out of the reactor for regeneration. A chemical cleaning step was carried out in order to recover as much of the initial permeability as possible. The membrane was successively soaked in different solutions at ambient temperature (chlorine 2 M for 2 h, sodium hydroxide 0.1N for 24 h and oxalic acid for 2 h). With this procedure, most of the initial water permeability was recovered (between 75 and 100%).

### 2.6. Rheometry

Rheological characterizations were conducted on samples taken in the SMBR. Shear flow viscosity and small deforma-

tion oscillation measurements were carried out using a Bohlin C-VOR 200 Rheometer (Malvern Instruments) fitted with either a cone and plate (2°, 60 mm diameter) or serrated parallel plates (60 mm diameter). The latter geometry is used to prevent wall slippage, which is a well-known phenomenon observed in rheometry of suspensions (Coussot and Ancy [15], Bertola et al. [16]). To ensure validity of the measurements, the occurrence of such a slip effect was examined by comparing measurements made with both smooth and serrated geometries. The results are shown in Fig. 4 and confirm that this effect was noticeable for low shear rates (less than 10 s<sup>-1</sup>) and was prevented by the use of serrated geometries. Therefore, measurements were carried out with serrated plates (roughness 150 μm). All were made at a regulated temperature of 23 °C.

Viscosimetry gives information on the liquor behaviour when it is flowing, i.e. on the shear stress (Pa)–shear rate (s<sup>-1</sup>) relation  $\tau_{xy} = \tau_{xy}(\dot{\gamma})$ . Measurements are made by prescribing continuously increasing shear stress or stages of stress. The thixotropic property was investigated by viscosimetric measurements. Thixotropy is the ability of a fluid to change with time. By definition, a fluid is thixotropic if its viscosity decreases with time when a constant shear stress is applied and if it comes back to its original viscosity after a sufficient rest period. This property can be examined by applying increasing shear stress followed by decreasing shear stress. If the

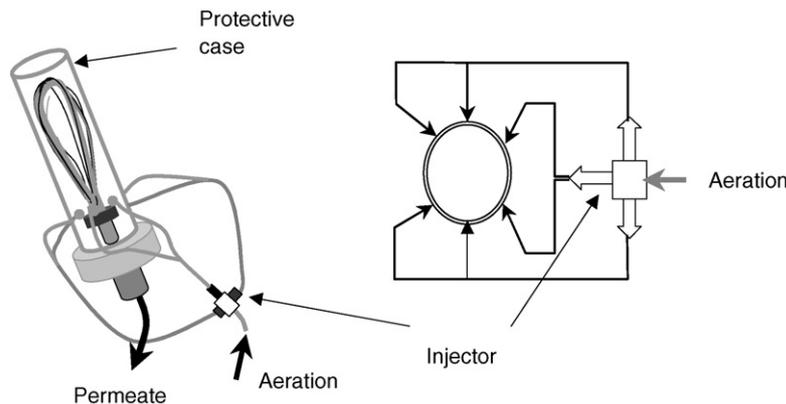


Fig. 3. Coarse bubble injector.

two curves are not superimposed, the fluid can be qualified as thixotropic.

Dynamic oscillatory shear measurements are used to characterize a liquor in small deformations and to detect the elastic domain. They are performed by applying a time dependent strain  $\gamma$  to the fluid sample:

$$\gamma(t) = \Gamma \sin(\omega t)$$

where  $t$  is the time (s);  $\omega$  is the oscillatory frequency (Hz);  $\Gamma$  is the maximum strain and by measuring the resulting shear stress  $\tau_{xy}$  as a function of time:  $\tau_{xy}(t) = \Gamma(G' \sin \omega t + G'' \cos \omega t)$ . The storage modulus  $G'$  is related to the energy that is stored in the sample to resist deformation and the viscous modulus  $G''$  is a measure of the energy that is dissipated when the sample is strained. The loss angle  $\delta$  is defined by  $\tan \delta = G''/G'$  and indicates whether the fluid behaviour is rather elastic (low values of  $\delta$ ) or rather viscous (values of  $\delta$  near  $\pi/2$ ).

The rheometer was also used to simulate intermittent aeration by applying successive stages of stress to the mixed liquor. Once the yield stress of the mixed liquor had been determined, the liquor was submitted successively to shear stress below and above its yield stress. The aeration cycle of 1 min every 6 min was then simulated on the rheometer by alternating 'high' shear stress (above yield stress) for 1 min and low shear stress (less than the yield stress) for 5 min.

### 3. Results

#### 3.1. Preliminary result

Preliminary analyses were carried out in order to determine how to perform the rheological analyses. The thixotropic property was especially investigated. Depending on the experiment, this measurement was carried out for three to six samples of the mixed liquor of the SMBR and results were always similar to those presented in Fig. 5. In this way, no thixotropy was detected in the mixed liquor.

Moreover, two or three measurements successively made on the same sample gave similar results. Measurements were therefore reproducible. Rheological measurements on suspensions

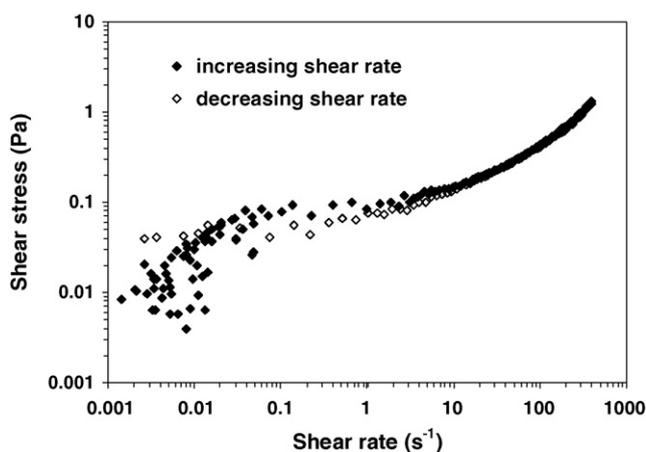


Fig. 5. Analysis of the thixotropy of the mixed liquor.

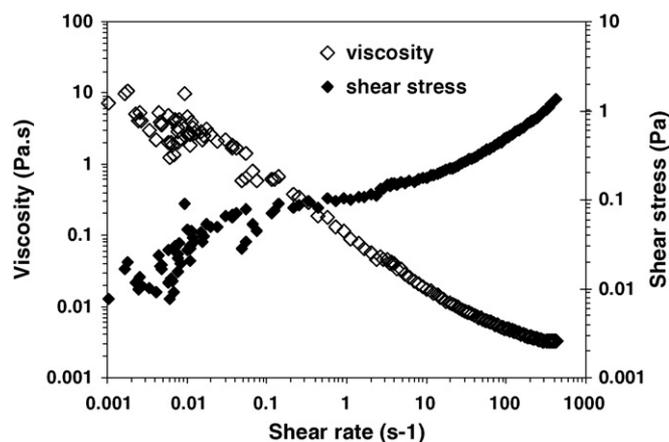


Fig. 6. Mixed liquor rheological analysis at increasing shear stresses.

are generally preceded by a stage of shearing in order to bring the fluid into a reference state and obtain reproducible results. This stage proved not to be necessary, so the mixed liquors were characterized without any previous shearing, except for the installation of the sample in the rheometer and homogenization by gentle reversal of the flask to avoid natural settlement.

#### 3.2. Rheological characterization of the SMBR mixed liquor

##### 3.2.1. Experiments performed with samples from SMBR fed with the synthetic effluent

First, experiments were performed with an activated sludge fed by a synthetic effluent. The biological conditions were fixed at an SRT of 50 days with an MLSS concentration of 5 g/L. A typical rheogram obtained for this kind of mixed liquor is presented in Fig. 6. The viscosity shows significant variations corresponding to a ratio of 3000:1 for the extreme values of the shear rate. Moreover it can be seen that a weak variation of the shear stress around 0.1 Pa led to a strong increase in the shear rate (from  $10^{-1}$  to  $10^1$  s $^{-1}$ ), which means that the mixed liquor had viscoplastic properties.

To check this observation, experiments were performed in oscillation mode. Although less accurate, this method is also used for yield stress determination. The results of these measurements are presented in Fig. 7, where two areas can be distinguished. The first one occurs under 0.05 Pa, where the loss angle is less than 20°. The second one is observed for shear stresses higher than 0.1 Pa and the loss angle is around 65°. The transition region spreads from roughly 0.5–1 Pa. This result points out that the mixed liquor has elastic behaviour when it is subjected to a shear stress lower than 0.05 Pa and viscous behaviour for stresses higher than 0.1 Pa. The mixed liquor can be qualified as viscoplastic with a yield stress around 0.1 Pa.

##### 3.2.2. Experiments performed with samples from SMBR fed with domestic wastewater

Considering the previous results, it is reasonable to wonder about the origin of such behaviour: is this behaviour linked to the specific mixed liquor used, which was a synthetic one, or

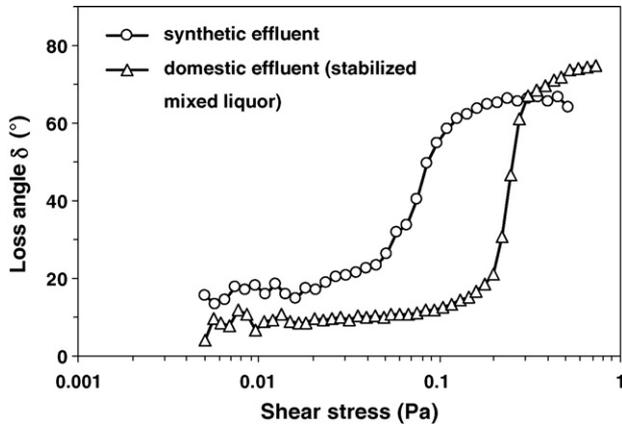


Fig. 7. Mixed liquor rheological analysis in oscillation mode.

is it typical of the SMBR process? To answer this question, a similar experiment was carried out on the same pilot plant but fed with a domestic effluent.

Analyses were conducted with the activated sludge in three different biological phases: (i) the original activated sludge taken from the wastewater plant, (ii) the mixed liquor in an intermediate state 12 days after the activated sludge was introduced into the SMBR, and (iii) the stabilized mixed liquor at an SRT of 20 days. The rheological results of the viscosimetric analyses are reported in Fig. 8. The rheological mixed liquor properties changed with the biological phases. The mixed liquor in its development phase and the original activated sludge had viscoplastic properties but, in the case of the stabilized mixed liquor, the effect of viscoplasticity was stronger. In this case, the yield stress was indeed the highest. This was confirmed by dynamic oscillatory measurements which are presented in Fig. 7. They confirm a yield stress of roughly 0.3 Pa for the stabilized mixed liquor.

The suspended solid concentration may explain this result. Tixier [17] has demonstrated that activated sludge with a high suspended solid concentration has pseudoplastic behaviour. In the original and intermediate mixed liquor, the suspended solid concentration did not reach 4 g/L whereas, for the stabilized

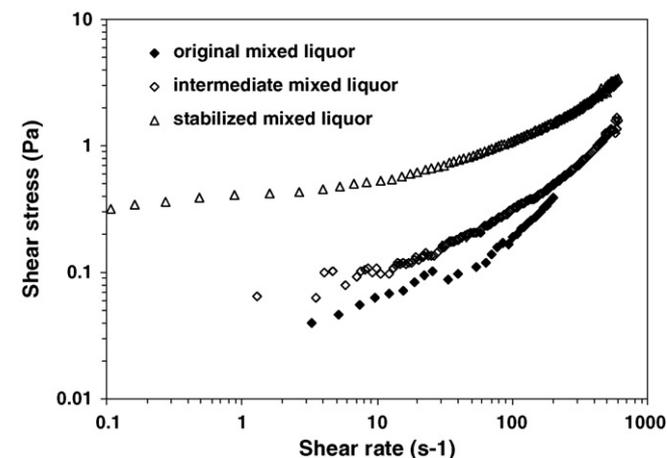


Fig. 8. Mixed liquor rheological analyses of domestic wastewater. Comparison of original, intermediate and stabilized mixed liquor.

mixed liquor, the suspended solid concentration reached 9 g/L. At such a concentration, SMBR mixed liquor may produce a high quantity of exopolymers which are recognized as viscoelastic compounds. It can therefore be assumed that the stabilized mixed liquor contained more exopolymers than the other samples.

These results confirm that the mixed liquor viscoplasticity is an intrinsic property of the SMBR process and not a property linked to the kind of effluent.

### 3.2.3. Rheological experiments by stages of stress

The last way to investigate the behaviour of the mixed liquor was to apply successive stages of stress to the sample. When a shear stress was applied or removed abruptly, a transitory state occurred where the structure and viscosity of the fluid were variable before stabilizing (Tixier [17]). Experiments were performed as follows: stages of increasing shear stress of 0.05, 0.1, 0.5, 1 and 2 Pa were applied, followed by symmetrical stages of decreasing shear stress (Fig. 9). These experiments were performed with the synthetic effluent.

For small shear stresses (less than 0.1 Pa), the viscosity of the mixed liquor reached 100 Pa.s. These instantaneous viscosities are high and indicate that, in this part, the mixed liquor was solicited in its elastic domain, i.e. below the yield stress. Then, increasing shear stress led to a large decrease of the viscosity down to a stabilized value around  $3 \times 10^{-3}$  Pa.s for the shear stresses of 1 and 2 Pa.

When the shear stress is decreasing ( $t > 800$  s in Fig. 9) the phenomenon seems to be reversible. The mixed liquor may have the ability to restructure itself. We focused particularly on the first step of the stress stages (0.05 and 0.1 Pa). An antagonist effect can be seen in Fig. 9. At a shear stress of 0.1 Pa, the viscosity can be seen to decrease at first but then an increase in the viscosity is noticeable although the applied shear stress is still constant. Initially, the increase of shear stress from 0.05 to 0.1 Pa leads to a large, rapid decrease in the viscosity (from 20 to 0.07 Pa.s). This decrease in the viscosity can be explained by a destructuring or disorganization of the mixed liquor around the yield stress of 0.1 Pa. Then the viscosity reaches the value obtained when a shear stress of 0.05 Pa is applied.

A superposition of the increasing and decreasing steps leads to Fig. 10, where it is noteworthy that the curves obtained in

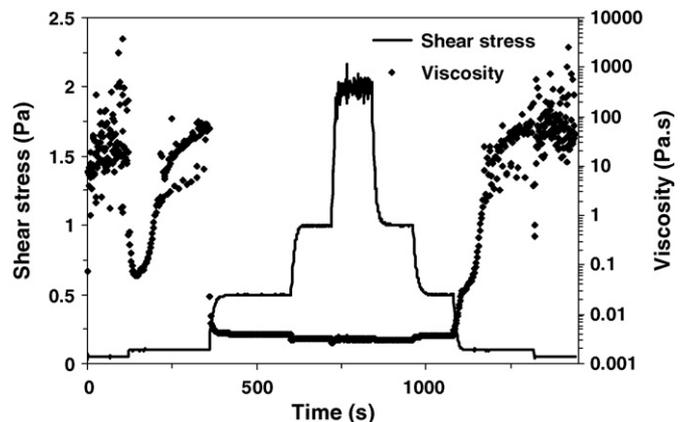


Fig. 9. Mixed liquor rheological behaviour during stages of stress.

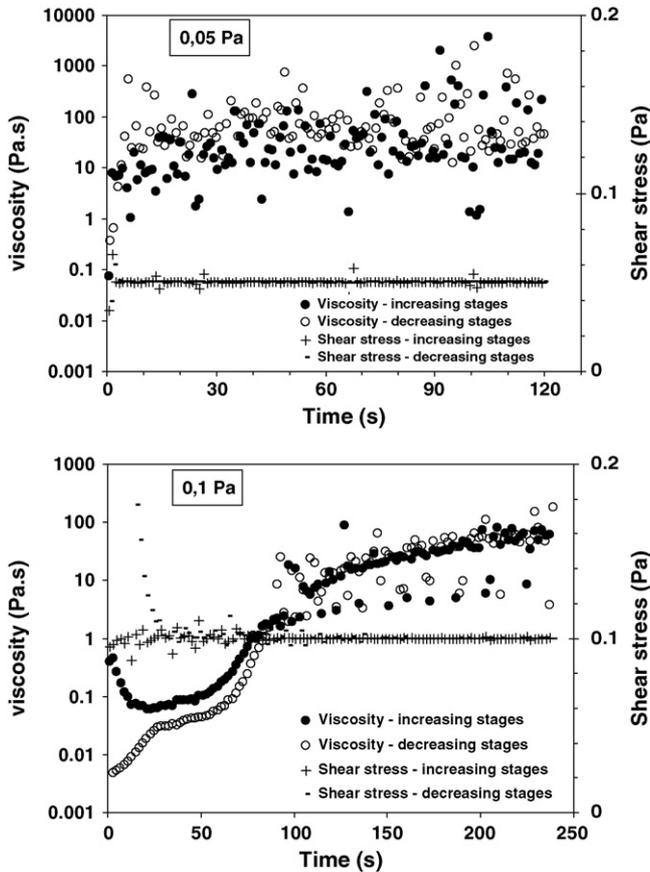


Fig. 10. Comparison of 0.05 and 0.1 Pa shear stress stages of Fig. 9.

increasing stages of stress are almost superposable on the curves obtained in decreasing stages of stress, especially in the restructuring step.

These results are important and useful for process management. It is well known that, the more the mixed liquor flocculates, the more slowly the membrane fouls. A minimization of the shear stress in the reactor is then indicated to keep the best possible mixed liquor flocculation. In the case of the SBR, the main shear stress is provided by the aeration. But this shear stress is also necessary to prevent membrane fouling. A good compromise has to be found for this parameter to satisfy these antagonistic requirements. The effect of intermittent aeration was investigated for this reason.

It can be assumed that intermittent aeration would be better than a continuous one to prevent membrane fouling. In such a situation, the time without aeration would be favourable to a restructuring of the mixed liquor since the shear stress would be lower during this time.

### 3.3. Rheology as a process management tool

#### 3.3.1. Simulation of intermittent aeration by a rheometer

Intermittent aeration was simulated with the rheometer on the mixed liquor as described at the end of the part 2.6. This was done on a sample of mixed liquor in a non-stabilized state with an MLSS concentration of 3.5 g/L. Its yield stress was determined to be around 0.01 Pa. The stress stages were 0.05 Pa for 1 min and

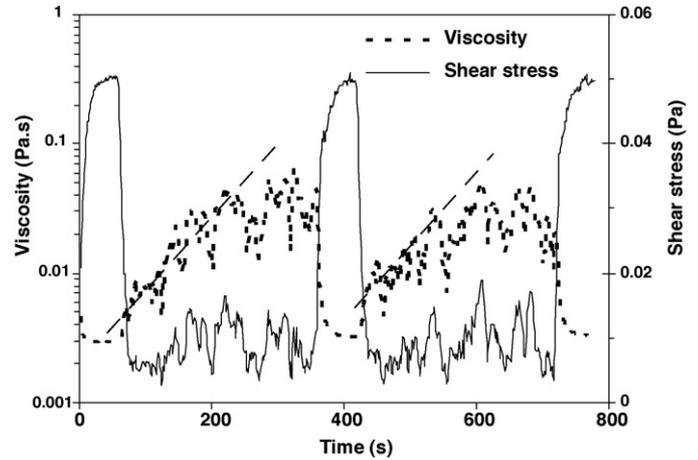


Fig. 11. Mixed liquor rheological analysis at shear stress stages of 0.05 Pa for 1 min and 0.005 Pa for 5 min. The mean increase of viscosity is highlighted by the dashed lines (---).

0.005 Pa for 5 min. From Fig. 11 it can be noted that the viscosity was the lowest ( $3 \times 10^{-3}$  Pa s) when the shear stress was the highest, which may mean that the mixed liquor flocculation was dispersed. But when a lower shear stress was applied, the mixed liquor viscosity tended to increase up to an approximate value of 0.1 Pa s. This simulation of intermittent aeration by rheology points out the ability of mixed liquor to organize itself during periods with low shear stress.

#### 3.3.2. Interest of intermittent aeration in a SBR

If energy costs are to be kept to a minimum, fouling has to be prevented using the lowest effective aeration flow rate. Three experiments were conducted as presented in Section 2.4 for an SRT of 14 days and an MLSS concentration of 5 g/L. Pressure drifts of the three previous experiments are reported in Fig. 12. Globally, it can be seen that pressure drift decreased with a decrease in the aeration frequency. When aeration was injected continuously, a slow pressure drift was recorded for the first 3 days but an abrupt increase was then observed, reaching 493 Pa/h at the end of the experiment. In the case of intermittent aeration, very slow pressure drifts were recorded during the 2 weeks of the experiment. A 58 Pa/h pressure drift was determined when

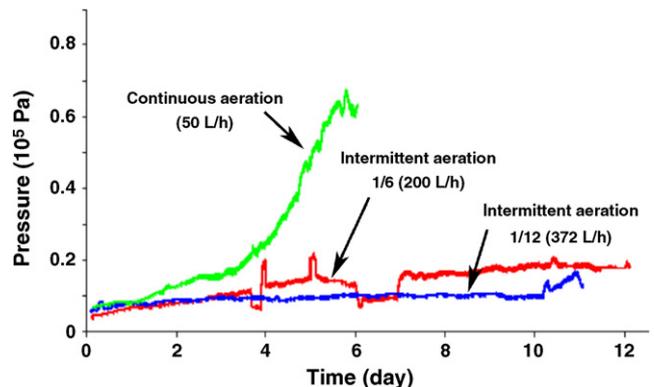


Fig. 12. Pressure drift variation according to aeration mode for a stabilized mixed liquor at a SRT of 14 days.

Table 3  
Settlement index variation and energy cost according to aeration mode

Mode	Flow rate (L/h)	Settlement index	Energy cost (kWh/m)
Continuous	50	None	1.06
1/6 <sup>a</sup>	200	241 (good)	0.71
1/12 <sup>b</sup>	370	86 (very good)	0.37

<sup>a</sup> Intermittent aeration 1 min every 6 min.

<sup>b</sup> Intermittent aeration 1 min every 12 min.

aeration was injected for 1 min every 6 min and only 39 Pa/h when the frequency of aeration was 1 min in 12.

In parallel with these experiments, the settlement index of mixed liquor was followed to investigate the influence of the aeration mode on the mixed liquor settlement. A small settlement index indicates a water with mature flocs whereas a high settlement index indicates mixed liquor with uniform dispersion of flocs in the suspension. The results reported in Table 3 reveal that, in the range of frequencies explored, the more intermittent the aeration, the better the settlement index. In the case of a continuous aeration, the mixed liquor was not allowed to settle whereas the intermittent aeration every 12 min led to good flocculation of the mixed liquor with a low settlement index.

These results point out that mixed liquor flocculation is less damaged by high but short intermittent aeration than by continuous or frequently intermittent aeration. These results confirm the previous experiments performed with the rheometer (Section 3.3.1): injecting intermittent aeration allowed mixed liquor to restructure. To conclude on the efficiency of an intermittent aeration mode, membrane fouling has to be taken into consideration for these different modes. It is then obvious that intermittent aeration effectively prevents membrane fouling.

Another way to evaluate the benefit of intermittent aeration is to determine the energy cost of the different aeration modes. Aeration energy consumption ( $E_a$ ) can be calculated according to Eq. (1).

$$E_a = \frac{Q_a \cdot \Delta P_a}{V_{H_2O}} t \quad (1)$$

With  $Q_a$  aeration flow rate ( $m^3/s$ );  $\Delta P_a$  pressure loss (Pa), equal to  $10^5$  Pa for all the experiments;  $V_{H_2O}$  volume of waste water treated ( $m^3$ ); and  $t$  running time (h), depending on the state of membrane fouling. The results are presented in Table 3. The energy cost is divided by three. This calculation only takes the aeration energy cost into account and disregards the permeate pumping energy since the average pressure drift decreases with the introduction of intermittent aeration. It is then obvious that an intermittent aeration mode provides considerable cost savings.

#### 4. Conclusion

An original approach using a rheological study to minimize the fouling in SMBR has been elaborated. First, a study of the rheological properties of the mixed liquor showing viscoplastic behaviour led to the hypothesis that the mixed liquor had a different structure depending on the applied shear stress. A comparison with activated sludge mixed liquor showed that this

property was specific to SMBR mixed liquor. Then, a study using increasing and decreasing shear stress stages and another using alternating shear stresses reinforced this assumption. The former study showed that intermittent use of aeration could be interesting. The study of the settlement index and of the pressure drift in an SMBR with different aeration modes showed that the intermittent mode was much more efficient than continuous aeration from a fouling point of view. From a theoretical point of view, this analysis would be improved by a determination of the mean shear stress in the process and its comparison with the yield shear stress. From the process point of view, the next steps will be to study the influence of the frequency of water suction and of intermittent aeration.

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#### Nomenclature

BOD	biological oxygen demand (mg O <sub>2</sub> /L)
COD	chemical oxygen demand (mg O <sub>2</sub> /L)
$E_a$	aeration energetic consumption (Wh/m <sup>3</sup> )
$G'$	storage modulus (Pa)
$G''$	viscous modulus (Pa)
MLSS	mixed liquor suspended solid (g/L)
$\Delta P_a$	pressure loss (Pa)
$Q_a$	aeration flow rate (m <sup>3</sup> /s or L/h)
SRT	sludge retention time (h)
$t$	running time (s or h or day)
TKN	total Kjeldahl nitrogen, (g/L)
$V_{H_2O}$	volume of waste water treated (m <sup>3</sup> )

#### Greek letters

$\delta$	loss angle (°)
$\gamma, \Gamma$	strain, strain amplitude
$\dot{\gamma}$	shear rate (s <sup>-1</sup> )
$\tau_{xy}$	shear stress (Pa)
$\omega$	oscillatory frequency (Hz)

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