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Rheological study of sludge of membranes bioreactors for water treatment: protocol, limitations and link with the filtration

Etude rhéologique de boues de bioréacteur à membranes pour le traitement des eaux usées : protocoles, limitations et liens avec la filtration.

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Abstract.

The submerged membranes bio-reactors (sMBR) are more and more visible in the field of waste water treatment. The membrane fouling, that causes a drastic fall of the performances, can be limited by the application of a swarm of bubbles close to the membrane surfaces. This aeration as a major factor of the energetic expense of these processes, lets numerous studies to focus on the understanding of the mechanisms linking the bubbles swarm with fouling.. In this background, the rheological tools appeared to be useful to understand the behaviour of sludge under shear stress. In order to be sure to get significant results, we used three types of rheological measurements: i) the influence of shear rate on shear stress; ii) the influence of upward then downward stages of shear stress on viscosity; iii) the influence of the alternation of two stages of shear stress with implementation times identical to those of sMBR aeration. Besides a viscoplastic behaviour previously observed, a phenomenon of hysteresis appeared. The transparent upper plan used on the rheometer exhibited formations of aggregative structures “in roll”. Sludges of a sMBR using different sequencing of aeration are compared: the developed protocols enabled to differentiate those sludges, with characteristic results depending on mechanical applications. The aim of this work is, eventually, to propose some correlations between usual characteristics of the process and aeration operating parameters, in order to propose an aeration policy improving energetic performances.

Key-words: membranes bioreactor; rheology; shear stress; aeration; sludge;

Résumé

Dans le contexte du traitement de l'eau par bioréacteur à membranes (BaM), les BAM immergées (BAMI) trouvent une place de plus en plus marquée. Le colmatage des membranes, qui entraîne une chute drastique des performances, peut être limité par l'application d'un train de bulle en proche paroi membranaire. Cette aération contribuant pour une part importante au coût énergétique de ces procédés, de nombreuses études s’appliquent à comprendre les mécanismes liant les trains de bulles au colmatage. La rhéologie des boues est complexe. Aussi, pour étudier l’impact hydrodynamique de ces trains de bulles, les outils rhéologiques sont intéressants pour comprendre le comportement des boues sous contraintes. Pour assurer des mesures significatives, nous avons utilisé trois types de mesures rhéologiques: i) l'évolution de la contrainte de cisaillement en fonction du gradient de vitesse; ii) l'évolution de la viscosité

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sur des paliers ascendants puis descendants de contrainte de cisaillement; iii) l'évolution de la viscosité en alternant deux paliers de contrainte des cisaillement dont les temps d’applications sont similaires à ceux des séquences d'aération retrouvées dans le BAMI. En plus d’un comportement viscoplastique précédemment observé, un phénomène d’hystérésis est apparu . L’utilisation d’un plan supérieur du rhéomètre transparent montre la formation de structures agrégatives « en rouleau ». Les boues issues d’un BAMI avec des séquençages de l’aération différents, sont présentées et comparées : l’outil « rhéomètre » a permis de différencier ces boues, par des réponses spécifiques suivant les sollicitations mécaniques. L’objectif de ce travail est, à terme, de proposer des corrélations entre les grandeurs usuelles qualifiant le procédé en fonction des paramètres de l'aération, afin de proposer un mode d’aération adéquat visant les performances énergétiques.

Mots-clés : bioréacteur à membranes; rhéologie; contrainte de cisaillement; aération; boues activées

1. Introduction

The submerged membranes bio-reactors (sMBR) leave more and more their mark in the field of waste water treatment. The membrane fouling, that causes a drastic fall of the performances, can be limited by the application of a swarm of bubble close to the membranes. Studies, striving to minimize the impact of this aeration on the energetic performances, have shown the impact of its sequencing. However, since the parameters are numerous, finding the optimal sequencing takes exponential time and understanding mechanisms would help to find optimal sequencing for each sMBR configuration. Most of suspensions, particularly activated sludges, are very subtle to characterize, first due to the fragility and the size of the flocculated structures which play a key role in the flow properties (Van Kaam et al, 2008). The rheological properties cannot be reduced to the viscosity value; the whole rheogram should be considered (shear stress vs shear rate). Some difficulties due to the possible settlement during measurement may appear. And finally the possible thixotropy of the activated sludges, which is the evolution of viscosity with time while the shear stress remains constant, should be considered (Tixier et al, 2003). The past of the sample may be determining for its rheological response. Thus a sample with no past shear will have strongly flocculated particles and its viscosity will be high. On the contrary, if a first shear is applied, the most fragile structures would be broken and the viscosity would be low. The aim of this work consists in two steps; the first aimed at defining protocols that are able to characterize rheological behaviour of sludge, that is to say the response of biological sludge being submitted to mechanical shear stress by the mean of the “tool” rheometer, the second step consisted in evaluating by the same mean the rheological characteristics of sludge from sMBR obtained under two different aeration conditions and to link by this way mechanical stresses in the process and in the rheometer.

2. Materials and methods

2.1 The membrane bioreactor

In order to be able to neglect the influence of other parameters, stable biological sludges from a “sludge production pilot” are taken to feed the second “trial pilot”, where sludge is submitted to various aeration conditions. Both pilots are fed with urban waste water. The first pilot, of a reactive volume of 100 L, operates in fixed conditions for sludge retention time (SRT = 20 d), permeate flux (10 $L.h^{-1}.m^{-2}$) and aeration in order to ensure stable biological functioning. The second pilot, of 15 L, has an externalized submerged membrane following Polymem configuration (Lorain et al, 2007). The biological conditions of the sludge are as follow: F/M = 0.1 à 0.13 $kgCOD.kg^{-1}SS$, SS = 3.5 +/- 1.5 $g.L^{-1}$, d(0,5) = 95 µm +/- 10 µm. Aeration and filtration cycles are synchronised: A time of filtration without aeration is followed by a relaxation time with aeration. These stages of filtration/aeration have been done following different parameters that are presented in table 1.

Table 1: Synchronized filtration and aeration cycles used in the second sMBR pilot

	Experiment 1		Experiment 2	
	Stage 1	Stage 2	Stage 1	Stage 2
Time (s)	100	210	300	10
Filtration flux $l.h^{-1}.m^{-2}$	10	0	10	0
Aeration flow-rate $Nm^3.h^{-1}$	0	0.15	0	1.60
SAD_m $Nm^3.h^{-1}.m^{-2}$	0	0.684	0	7.1

2.2 Rheometry

The rheological characterisations are done on samples from the second sMBR. They are done on a rheometer C-VOR 200 (Malvern Instruments) fitted with serrated parallel plates (ϕ 60mm, rough spot 150µm). The serrated is in order to prevent the effects of segregation in the fluid close to the wall and the higher shear of a thin layer of fluid with less flocs in this zone (Coussot et al, 1999). The upper serrated plate has been selectively replaced by a smooth pate in plexiglass to be able to visualise the flow during the measurements. Measurements have been done with a constant temperature of 23°C. Three rheological protocols have been used: the first one, a rheogram, is a measurement with a shear stress rising and descent during 480 s between the two extreme values. The second one consists in upward then downward stages of shear stress. At last, the third is based on the alternation of two stages of shear stress, one with high shear stress, the other with low shear stress. Figure 1 presents a typical rheogram giving the evolution of the shear stress function of the shear rate for a sample of sludge of sMBR. Two zones with different behaviours may be observed: a first zone for low shear stress values where the behaviour is elasto-plastic , and a zone of viscous behaviour below a shear yield point.

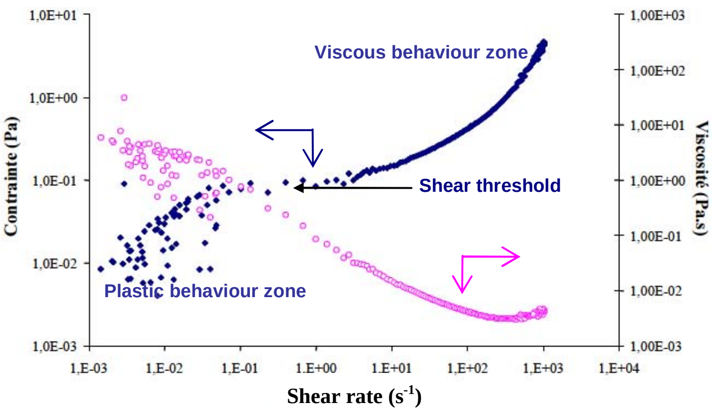

Figure 1. Rheogram characteristic of activated sludge from BRM

The second type of measurements consists in submtting the sludge to upward then downward stages of stress. This experiment consists of increasing rise in shear stress by stages of 100 s: 0.05, 0.1, 0.2, 0.4 and 0.8 Pa followed by a symmetric descent. When shear is applied or stopped brutally, a transitory regime appears showing a restructuring of the sample. The shear stress and the viscosity resulting from it are thus going to evolve until a stabilisation [1, 2]. The last series of measurements study the behaviour of the sludge under sequencing of aeration by imitating its shear with the rheometer: a shear stress of 1 Pa is imposed for 10 seconds (order of magnitude of shear stress during the aeration (Matrinelli et al, 2010)) followed by a landing of 300 seconds to 0,005 Pa and the evolution of the viscosity according to time is followed on several cycles. The measures are made on samples taken after 1 day experiment in the bioreactor.

3. Results and discussion

3.1. Rheograms

The figure 2 shows the evolution of the shear stress according to the shear rate for samples from the experiments 1 and 2 corresponding to different aeration sequencings. Both sludge present a plastic behaviour in the very low shear rates, then, secondly, a viscous behaviour. The transition between the plastic behaviour and the viscous one occurs beyond a shear stress threshold around 0.03 Pa in both cases. The determination of this last parameter remains subtle since for low values of shear, the low measured values do not show an excellent reproducibility due to the fact that they are on the lowest measurable torques by the rheometer. For the sludge resulting from the experiment 1, we observe a marked tixotropy with a rough increase of the shear stress in approximately 1 s^{-1}. We observe quite frequently, tixotrope behaviour for suspensions of asymmetric particles, and it can be explained by two hypotheses. The first one consists in admitting that the structural units dispersed in the fluid tend to align themselves gradually in the direction of the flow as the shear rate increases, promoting the flow of the various layers of liquids [2, 4]. The second hypothesis consists in considering a modification of structure of the liquid attributable either to a break of connections or to a deflocculating of particles (Coussot et al, 1999). The observation of a sudden increase of the shear stress in a practically constant shear rate is rarer but was observed by Wheeler et al. (Wheeler et al, 1998) and explained by a restructuring inferred by the shear and linked to instabilities of flow. The behaviour of the sludge is visualized though the upper plate in Plexiglas during a response to an upward/downward shear stress slope of 600 s. Pictures are realized at the various moments of the measurement. An example of the obtained results is proposed on the figure 3. This sludge rheogram presented a non linear variation of the shear stress with the shear rate and a viscoplastic behaviour similar to those already observed. However, the curve of rise (in blue on the figure 3) presented very important oscillations between 0.1 and 1 s^{-1}. Photos realized during this measurement have shown that, on this range of shear rate, the fluid was not macroscopically homogeneous but that large-dimension radial structures were developed. The fluid seemed exempt from flocs between these structures (time t = 220 s to t = 430 s). For strong shear rate, these structures disrupted and the aggregates disintegrated. The fluid seemed to find a homogeneity (time t = 660 s). Finally, during the phase of decrease of the shear stress, these structures re-formed (approximately in time t = 970 s) and they lasted till the end of the measurement. This phase did not come along with curve oscillations as during the rise. These phases of macro-structuring inferred by the shear rate are more or less important depending of the sludges. They are linked to the content in SS and to the nature of the sludge, in particular in the presence of probable filamentous who favoured the aggregation and the rolling-up of the flocs by the shear.

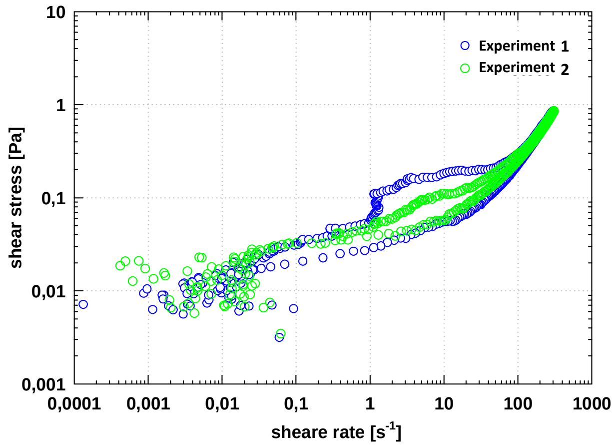

Figure 2. Rheogram obtained for experiment 1 (○) and 2 (○)

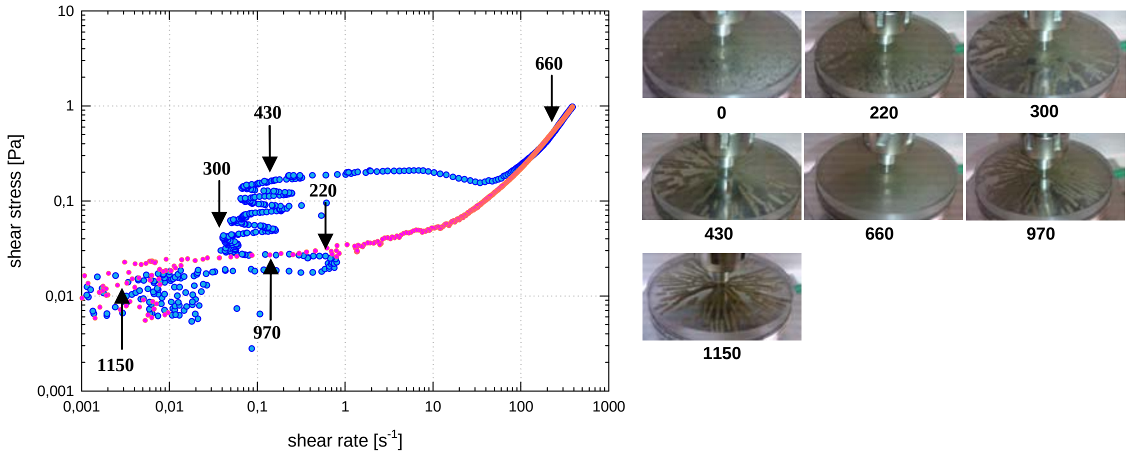

Figure 3. Rheogram of a SBRM sludge with a visualisation through an upper plate in plexiglass (●: upward slope, ●: downward slope.)

3.2. Upward/downward stages.

As it can be seen on Figure 4 for low values of shear stress, the rheometer measured values of instantaneous viscosity in the order of 0.1 to 10 Pa.s. These high values belong to the zone of plastic behaviour, below the threshold flow. When the shear stress was increased to 0.4 and then to 0.8 Pa, a sudden drop of the viscosity until a value of 3. 10^{-3} Pa.s was noticed. When the shear stress was decreased in the reverse way, the restructuring of the material began but was very slow. If we focus now on the samples of the experiment 2 (green curve), the values of viscosity with low shear stresses are higher in the order of 10 to 100 Pa.s also proving that they were in the zone of plastic behaviour. In a similar way, if the shear stress was increased to 0.4 and to 0.8 Pa, the viscosity suddenly felt until a value of 1.10^{-2} Pa.s, value higher than for the sludge of the protocol l. When shear stress has been decreased in the reverse way, we notice that the phenomenon is reversible. These two sludges, demonstrating different properties of restructuring, were probably of different structures.

The main hypothesis concerns the adaptation of the sludge to the structuring/destructuring led, on the short term, by the shear rate produced by bubbles close to the membrane. The conditions of experiment 1 leaded a structuring/destructuring of the sludge with the same frequency but on a more important duration with a lower intensity. The structure of these sludges could be more fragile, bringing lower values of the viscosity for high shear stress than sludges of the experiment 2. Indeed these last ones, undergo a structuring/destructuring on a lower duration but with a stronger intensity. The time of restructuring in the MBR is longer and would allow a structuring and a solidification of the biological aggregates. This could explain a higher value for the viscosity for high values of shear stress.

3.3. Alternation of two stages of shear stress.

As shown on Figure 5, the behaviour of the sludge over sequenced shear stress has been studied. When the shear rate was suddenly modified, a transitory regime appeared. We observed on previous measures that the samples responded in a different way depending on the solicitation what we interpreted as a restructuring of the sample. An experiment was realized by imposing shear stress in stages of 10 seconds at 1 Pa (forced strong equivalents to the very airy phase) followed by a stage of 300 seconds to 0.005 Pa, (not aerated phase) and the evolution of the viscosity according to time was followed. We noticed on the figure 5 that during the period when the shear stress is for its maximum (1 Pa) the viscosity of sludges is its minimum (1.10^{-2} Pa.s) what would tend to prove that the sludge is in its most destructured state. On the other hand, when the “low” shear stress is imposed, during 300 seconds, the viscosity tended to grow and could reach values of 1000 Pa.s.

These two figures show well the ability of restructuring of the sludge under intermittent shear rates. Very few differences were observed during the use of this protocol which allows concluding on the limitation of the use of it to determine differences between these two sludges.

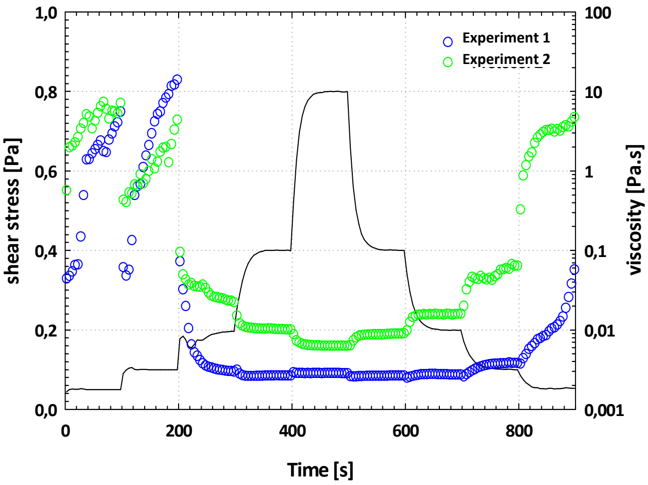

Figure 4. Evolution of the viscosity for sMBR sludges for experiment 1 (○) and 2 (○) function of time for upward and then downward stages of shear stress (-).

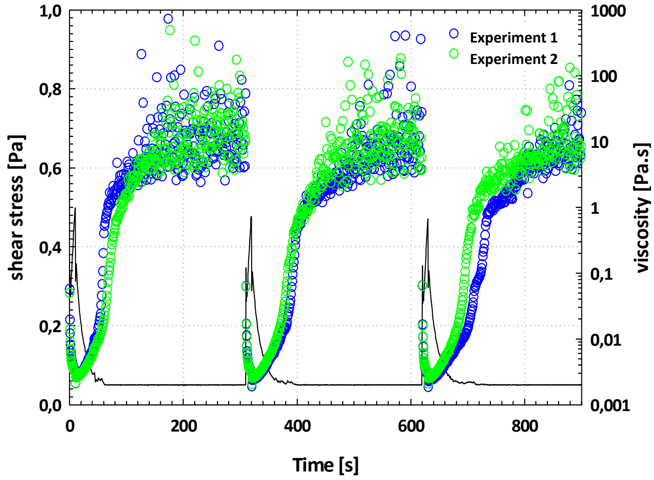

Figure 5. Evolution of the viscosity for sMBR sludges for experiment 1 (○) and 2 (○) function of time for an alternation of stages of1 Pa/ 0.005 Pa shear stress of respectively 10s/300 s duration.

3.3. Influence of the variation of the sequencing of the aeration, coherent with the filtration, on the evolution of the TMP.

A first analysis of the influence of the sequencing was realized through the comparison of two operating conditions allowing limiting the fouling in a bioreactor to membranes while optimizing its energy consumption. The figure 6 allows comparing the TMP for both realized experiments.

Classically for the experiment 1, after a sudden increase in very beginning of filtration, the trans-membrane pressure (TMP) remained almost constant. For the second experiment, the increase of TMP is more important in the beginning of filtration and the stabilisation having higher slope couldn't be maintained. Both types of sequencing of the aeration led responses in different TMP; The sludge that seemed with more destructuring (experiment 2) is the one that has faster fouling.

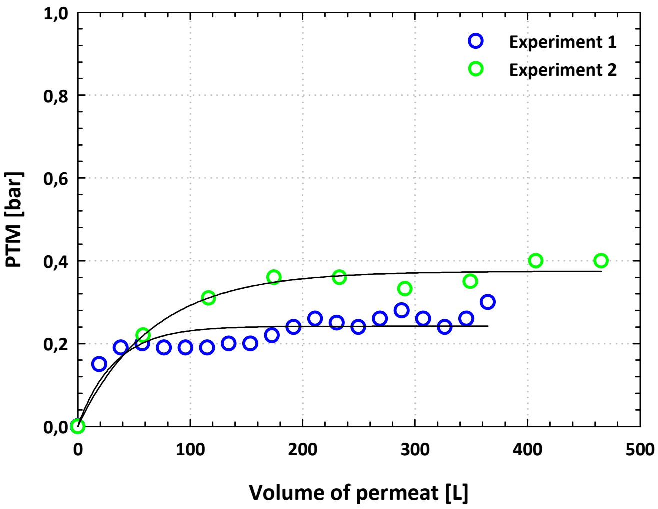

Figure 6. Evolution of the TMP function of time for experiment 1 (○) and 2 (○) (Lp = 150 $L.h^{-1}.m^{-2}.bar^{-1}$)

Conclusion

Two aspects were studied in this paper: the reaction to various policies of sequencing of the aeration and the understanding of the phenomena occurring at the level of the sludge thanks to the rheological tool. This study allowed to set up protocols allowing to assess the behaviour of sludges under constraints. These protocols brought to light problems of structuring under flow of sludges (unhomogeneity in the air-gap etc.). The implementation of a transparent mobile plate showed structures in "rollers" led by a flow until then never observed in the literature for this type of biological aggregates. This information explains the interest of the sequencing of the aeration of industrial processes, and show that it would be interesting to test policies of sequencing with time longer than those used at present so as to leave with the fluid the time to become less viscous. The formation of these structures can be bound to restructuring abilities of the sludges and thus require a quantitative consideration. It is indispensable to define relevant rheological parameters for the characterization as the surface of hysteresis, the constraint threshold of formation of the structures. It is within this framework that interesting perspectives are to be formulated. One of the interesting points is to study the correlation between the various properties observed restructuring and the performances of filtration. Within the use of the rheometer as tool for the evaluation of the fooling potential of sludges, both campaigns of experiment showed that the performances of filtration are modified when sludges show different restructuring properties; the sludge that seemed with more destructuring (experiment 2) is the one that has faster fooling. These have to be connected with the properties of sludges such as concentrations in SS and in polymers, flocs size, etc.

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