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Modelling soluble microbial products (SMPs) in a dynamic environment

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ABSTRACT

A calibrated ASM$_{2d}$ model of a full scale MBR is modified as to include the soluble microbial products (SMPs) fractions and study their dynamics in full scale. Batch tests were conducted to estimate the SMP kinetics. The biomass associated products (BAPs) kinetics were estimated with results in tune with previous experiments. The utilization associated products (UAP) kinetics estimation was instead complicated by two aspects which regularly occur when spiking readily biodegradable COD: storage phenomena (not accountable in ASM$_{2d}$); the non-uniformity between the polysaccharide fraction, easily biodegradable, and the protein fraction, which proved to be refractory to biodegradation. The procedure for UAP kinetics estimation would thus require further analysis. UAPs were found in full scale markedly predominant compared to the BAPs. The data analysis revealed that the membrane rejection mechanism was identified as SMP loading rate dependent, emphasizing the need of a more careful consideration towards this parameter when working in a dynamic environment. The work discusses the feasibility of the SMP extension studies in dynamic conditions. Fine tuning of the membrane rejection factor, the necessity of more frequent sampling, and experimental determination of the additional kinetics SMP parameters become necessary and burdensome adaptations of the ASM calibrations. However both nutrients removal, sludge production and energy consumption modelling were not improved by including the SMP fraction in the modelling. SMPs did not correlate with fouling rates in this full scale MBR, indicating a strong drawback, since the main drive for these models is thus not accomplished.

Keywords: Membrane bioreactor (MBR); Activated sludge model (ASM); Soluble microbial product (SMP); Full scale; Modelling

1. Introduction

In the last decade, several studies have been carried out to include the soluble microbial products (SMPs) concept in the activated sludge model (ASM) framework. The main drive for this extension was that SMPs were considered up to now a crucial fouling indicator [1]. The correlation between SMP fraction and fouling events was highlighted inter alia by Le-Clech et al. [2] and Rosenberger et al. [3]. A model extension from the general ASM models with the SMPs always implies the introduction of several extra kinetics parameters, i.e., Lu et al. [4] introduces 8 SMP related parameters in the ASM1 platform. Saroj et al. [5] infers that incorporation of SMPs in ASM would enhance the practical identifiability problem which is considered a crucial issue of the ASM calibration process. Jiang et al. [6] tackles this problem by setting up specific batch tests in order to evaluate separately the SMP related parameters.

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The general aim of this work is to analyze the advantages and feasibility of the ASM-SMP approach in full scale modelling according to the most updated research proposals. Several questions will have to be answered: is it possible to determine experimentally reliable SMP kinetic parameters? Does this procedure impact on the modeling exercise complexity? And last but not least, are the SMPs a relevant indicator for fouling prediction?

2. Materials and methods

The MBR of Schilde (Belgium) was built in 2003. The MBR is composed of a sand trap and a drum-sieve to protect the downstream system, a pre-denitrification tank (500 m^3), an aeration basin (500 m^3), and a filtration unit (240 m^3). The filtration unit is composed of 4 Zenon (Zeeweed 500c) MBR filtration tanks having a total membrane surface area of 10,160 m^2 treating in total an average flow of 230 m^3/h, and maximum peak flow of 355 m^3/h. The sludge recycle flow from the filtration to the aerobic compartment is 6 times the influent MBR flow.

The Schilde MBR model was calibrated in year 2006, and validated in the year 2009, with routine daily composite samples of influent and effluent flows. Sample frequency was every 2 weeks in 2006 and every 1–2 weeks in 2009. During both phases, influent and effluent samples were analyzed for BOD_5, COD, suspended solids, total nitrogen, Kjeldahl nitrogen, ammonia, nitrate, orthophosphate and total phosphorus. All samples were analyzed according to Standard Methods [7]. Samples filtration step was performed with a 0.45 μm polyester filter. Mixed liquor suspended solids concentration (MLSS) was calculated by the Standard Methods [7]. Nitrate, KWh of coarse and fine bubble aeration, flows, and dissolved oxygen were monitored with on-line probes.

With regards to the influent soluble inerts (Si) measurements, the procedure of Lu et al. [4] was employed: MBR sludge biomass was centrifuged at 3000 rounds per minute (RPM) for 5 min, washed with permeate water and centrifuged again. The biomass was then added to the soluble influent in a concentration of 3 g/l and aerated in an Erlenmeyer flask for about 4 h. Afterwards the sample has been filtrated and the soluble COD (COD_{sol}) measured.

Protein (PT) like SMPs were measured on filtrated samples (0.45 μm polyester filter), according to Lowry et al. [8]. For the calibration Albumin bovine, BSA, (Acros) fraction V, in a concentration range between 0-25 mg/l was used. Polysaccharides (PS) like SMPs were measured on filtrated samples (0.45 μm polyester filter), by the Anthrone method based on Dreywood [9]. For the calibration D(+)– anhydrous glucose (Acros) in a concentration range between 0–100 mg/l was used. Factors are applied for conversion of PT and PS from mg/l in mg COD/l, (respectively 1.5 and 1.0 g COD/g substance), by assuming that bovine serum albumin (BSA) represents PT and glucose represents PS. Conversion factors have been cross-checked in the lab.

Fresh sludge was sampled from the connection between the MBR biology and the filtration tanks. This was immediately poured in the batches ready for experiments. All batch experiments were conducted at room temperature (20°C), and pH was adjusted by NaOH (0.1 M) and HCl (0.1 M) solution.

To determine the biomass associated products (BAP) kinetics, batch experiments were conducted under starvation conditions without substrate addition. The produced SMP, dominated by BAP according to [6], were measured along 15 d. The experiment was repeated twice. COD_{sol}, MLSS, MLVSS, O_2 and SMPs concentration were measured.

To determine the utilization associated products (UAPs), batch experiments were spiked with acetate (HAc) in different concentrations under continuous aeration. The HAc solution is neutralized before being added to the batch. The experiment had 12 h duration. Meanwhile, a reference batch experiment was conducted without HAc addition to obtain the background COD_{sol} and SMP concentration. The net UAP concentration was determined with respect to the reference batch concentration. This method eliminates the BAPs in the UAP batch, as proposed by Jiang et al. [6]. Experimental errors are expressed as mean average deviations.

A model library in MATLAB environment, developed by Aquafin, was used to perform model simulations and parameter estimations. The experimental batch test was reproduced on the ASM2d_SMP model and the kinetics values of the UAP/BAP derived. The model was tested on the validation campaign (20.01.09–31.05.09) of the full scale MBR plant.

3. Results and discussion

3.1. Matrix modifications

SMP models have been developed since the late eighties. The SMP subdivision among UAPs and BAPs species has been widely accepted [10]: (i) UAPs, i.e. SMPs that are associated with substrate metabolism and biomass growth and are produced at a rate proportional to the rate of substrate utilization. (ii) BAPs, i.e. SMPs that are associated with biomass decay and are produced at a rate proportional to the concentration of biomass. Jiang et al. [6] review the ASM2d matrix with some interesting upgrades: amongst others, substrate degradation is accompanied by UAP formation (from 0 to 100 %), and fuap represents this percentage (* in Table 1). According to the authors, UAPs need to be hydrolyzed before being available for metabolic processes. Therefore 3 new hydrolysis steps (aerobic, anoxic, anaerobic) are added in the ASM2d matrix (not shown in this paper). Cell lyses is accompanied by BAP formation (from 0 to 100 %),

Table 1
In line (*), an example of the original Jiang's et al. [6] mode with regards to the "aerobic growth of X_h of S_f". In line (**), the modifications in this work for the very same process

	S_o	S_f	S_{UAP}	X_H	Rate
Aerobic growth of X_h on S_f (*)	$-\frac{1-Y_H-f_{UAP}}{Y_H}$	$-\frac{1}{Y_H}$	$\frac{f_{UAP}}{Y_H}$	1	$\mu_H X_H \cdot$ switches
Aerobic growth of X_h on S_f(**)	$-\frac{1-Y_H\cdot(1-f_{UAP})-f_{UAP}}{Y_H\cdot(1-f_{UAP})}$	$-\frac{1}{Y_H\cdot(1-f_{UAP})}$	$\frac{f_{UAP}}{Y_H\cdot(1-f_{UAP})}$	1	$\mu_H X_H \cdot$ switches

and f_{bap} represents this fraction. Also BAPs would need to be hydrolyzed before being available for metabolic processes. Therefore 3 new hydrolysis steps will be added in the ASM2d matrix.

When ASM2d version matrix was updated as above, a difficulty immediately emerged in the UAP process. In fact, it appeared that the sludge production would have to increase accordingly to increasing fuap percentage. For instance, in the heterotrophic growth on fermentable substrate (S_f) , a unit of S_f would produce Y_H biomass, notwithstanding the amount of substrate diverted into UAPs (* of Table 1). In this way, biomass would be produced immediately from S_f degradation, but also afterwards from UAP degradation, overcoming the maximum Y_H conversion (mg biomass/mg substrate). For example, if 50% substrate would be converted into UAP (f_{uap} = 0.5), the system would produce immediately Y_H biomass plus other $0.5\cdot Y_H$. COD mass balance is thus not valid anymore. In truth, if the UAP concept is correctly implemented, a unit of S_f would at first yield "$Y_H\cdot(1 - f_{uap})$" biomass, and the remaining UAP would produce a maximum of "$f_{uap}\cdot Y_H$" biomass only once converted, i.e., after hydrolysis. Matrix modifications were implemented as such to consider the heterotrophic sludge yield as $Y_H\cdot(1 - f_{uap})$, in place of $Y_H/(1 + f_{uap})$, correction factor applied in [6]. The updated process is shown in Table 1, for the "Aerobic growth of heterotrophs (X_H) on fermentable substrate (S_f)" process. The BAP process was instead not modified.

Another modification was made as to assume that the degradation of UAP and BAP produces intermediate products and biomass but does not lead to the production of new UAP or BAP. This assumption was employed by several research groups [11,12]. Any carbonaceous substrate is partly converted to UAPs according to the fuap percentage. The latter are now converted into a newly made fraction, S_f^*, modelled as the original S_f in ASM_{2d} (a unit of substrate yields Y_H biomass and no UAPs). Differently from S_f, S_f^* can only be produced by UAP hydrolysis.

The last modification regards the role of the autotrophic biomass (X_{aut}) in the UAPs formation. The UAPs production mechanism from the X_{aut} would imply a reduction of the autotrophic sludge yield of $Y_A\cdot(1 - f_{uap})$ in favour of the UAP formation. Consequentially, the autotrophic population would be reduced in favour of the heterotrophic population. This step requires attention when fitting the nitrogen removal. Lu et al. [4] fitted a specific parameter for the UAP formation during autotrophic conditions. However this practice increases the amount of unknown kinetics variables when calibrating the model. The option of running batch tests for the determination of the SMP kinetics of the X_{AUT} has been excluded. We observed instead that the extension of SMPs on the X_{AUT} would not produce a significant UAPs, given their low concentration (250 mg COD/L) compared to the X_{HET} concentration (1800 mg COD/L). The UAPs mechanism was not modelled on the X_{AUT}.

3.2. *Batch experiments for the SMP kinetics determination*

With regard to the UAP batch test: batch experiments with different HAc concentrations were run. A reference batch was conducted without HAc addition, to obtain the background soluble COD and SMP concentration. The highest concentration, 500 mg COD/l, is representative of a high load in full scale. SMPs concentration in the reference batch is constant, and mainly assimilated with BAPs, as justified by the low degradation (red dots in Figs. 1 and 2) . PT-like SMPs exhibits an immediate degradation followed by a very low degradation rate. A concentration of 5 mg/l remains in the system at the end of the test. PS-like SMP exhibit a peak at 1 h and then are completely degraded within 4 h (Fig. 2). PS and PT like SMP peaks do not occur at the same moment but within the same first hour. Net production of PS and PT would then be summed up to yield the net produced UAPs. COD_{sol} is high at t0 and then steadily drops. After 10 h the COD_{sol} of the 3 batches almost coincide again.

According to the procedure defined in the methodology, the batch test results must be modelled on the $ASM_{2d\ SMP}$ model. The model will have to fit first the CODsol when degrading the HAc, and secondarily the SMPs concentration. The kinetic values of the UAP would be consequentially derived. MLSS concentration was

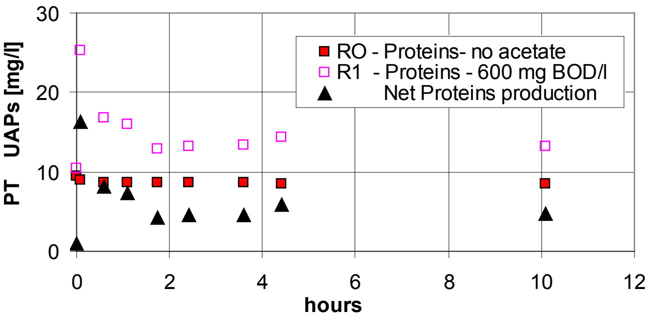

Fig. 1. PT like SMPs in the batch test spiked with 600 mg COD/L HAc, and in the reference batch test.

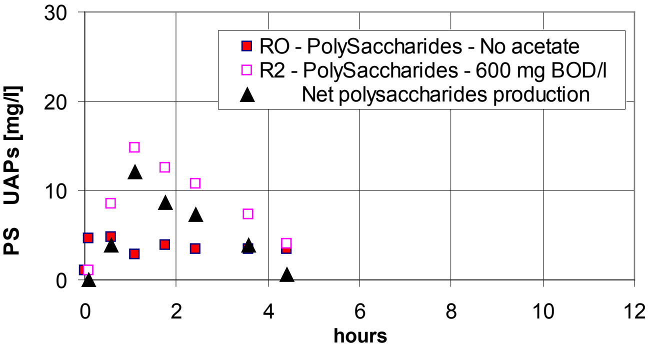

Fig. 2. PS like SMPs in the batch test spiked with 600 mg COD/L HAc, and in the reference batch test.

measured in the plant (9 g/l) and biomass fractions were predicted in the ASM_{2d} model.

Due to the high heterotrophic concentration, the μ_H (growth rate of heterotrophs) governs the speed of the carbon source degradation (default value of μ_H in ASM_{2d} is 3–6 for temperature 10–20°C). The μH has been adjusted to fit the COD_{sol} concentration. But as in Fig. 3, the COD_{sol} could not drop sufficiently, and the modelled COD_{sol} remains far higher than the experimental COD_{sol}. Guidasola et al. [13] pointed out that ASM1 is not capable to model properly the feast conditions when influent is characterized by RBCOD. The data analysis showed that COD_{sol} fate in the batch could not be properly modelled, and that the ASM_{2d} is not able to reproduce this batch condition. The problem could be overcome by considering that a part of the HAc is immediately used for growth, and a part is simultaneously stored. The net experimental SMPs production backs up this assumption. If HAc would have been entirely degraded, the SMPs concentration would have been much higher than the 25 mg/L measured (the fraction of produced UAPs according to literature is generally about 0.3 mg UAPs/mg substrate, meaning that 180 mg COD/l of UAPs should be produced from a HAc spike of 500 mg COD/l).

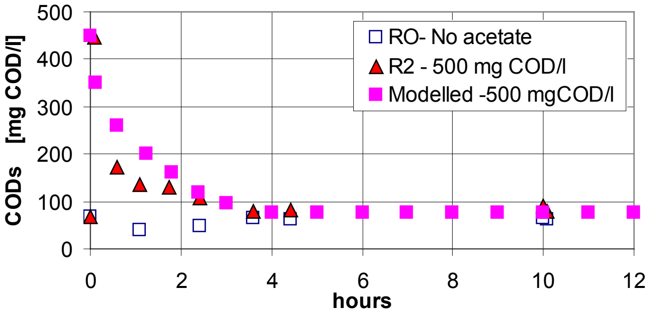

Fig. 3. Experimental and modelled soluble COD fate in the batch reactor.

The UAP kinetics appears different during the experiment. In the second part of the experiment (after the second hour in Fig. 1), the PT fraction is more recalcitrant to degradation. As already highlighted by several authors two phases are distinguishable: growth on substrate and growth on storage products [14]. The problem of the uncertain kinetics determination that rises when comparing first and second phase was avoided by Jiang et al. [6] with ignoring the second phase. However, this is risky since it may lead to an overestimation of the net UAPs formation. Due to (i) storage phenomena occurring with RBCOD spiked, and (ii) different degradation rates between the UAP fractions, the protocol described in the methodology cannot yield valuable data and UAP kinetics data will be simply fitted. The batch protocol should be updated as to overcome these problems.

With regards to the BAP batch test: two parallel experiments were conducted in order to verify the results reproducibility. The operational conditions were controlled as described in the material and methods section. O_2 increased during the experiment (3–6 mg/l), as a result of the active biomass reduction. This was confirmed by a decrease of the MLVSS fraction, from 70 up to 40%, meaning that the lysed cells were transformed in inert and inorganic material. Both reactors behaved similarly. SMP concentration increases over time in both reactors and eventually saturating, since biomass lyses impedes further degradation. Both fractions (COD_{sol} and SMP) tend to increase in concentration. Overall results are displayed in Fig. 4, where an average value and standard errors of the two parallel reactors are computed. COD_{sol} of the settled sludge supernatant is subtracted from the SMP concentration of the settled sludge supernatant, in order to give inert and RBCOD fractions.

ASM_{2d} was extended according to the processes described above, implemented in Matlab and used to model the experiment, within the same experimental conditions. BAP test was fitted with an f_{BAP} of 0.05 and an hydrolysis rate $K_{h,BAP}$ of 1×10^{-6} d^{-1} (Figs. 5, 6). These values are comparable with Jiang et al. [6] figures, where an f_{BAP} of 0.0215 and an hydrolysis rate $K_{h,BAP}$ of 7.41×10^{-7} d^{-1} was found.

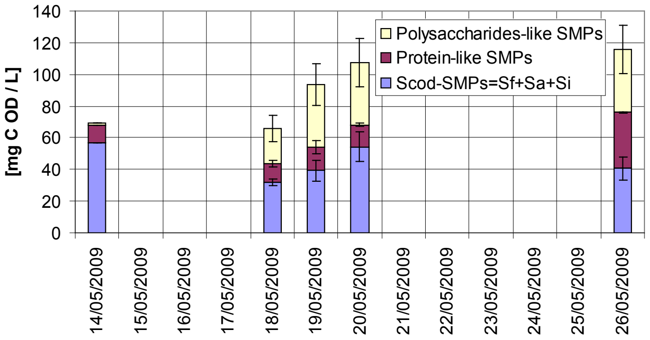

Fig. 4. Average SMPs and mean deviation error in batch 1 and 2.

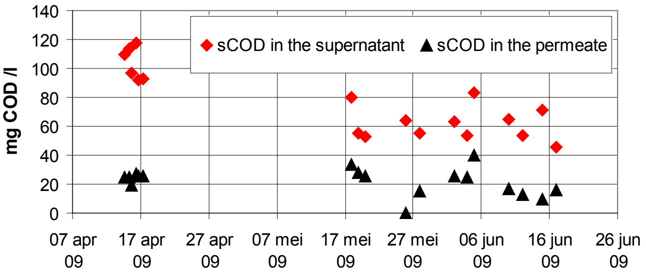

Fig. 7. Soluble COD (COD$_{sol}$) concentration measured in the supernatant and in the permeate.

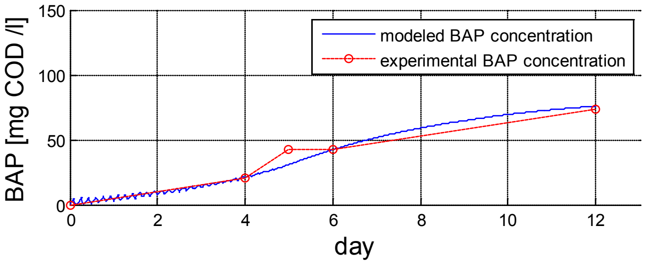

Fig. 5. Experimental and modelled BAP concentration in the batch reactor.

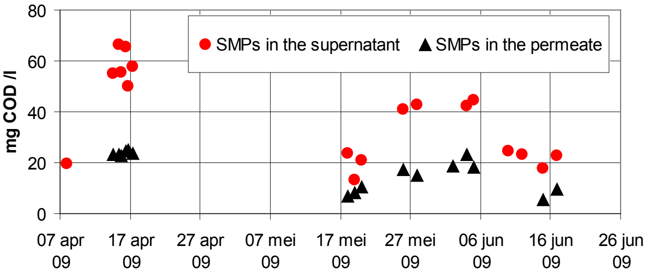

Fig. 8. SMP concentration measured in the supernatant and in the permeate.

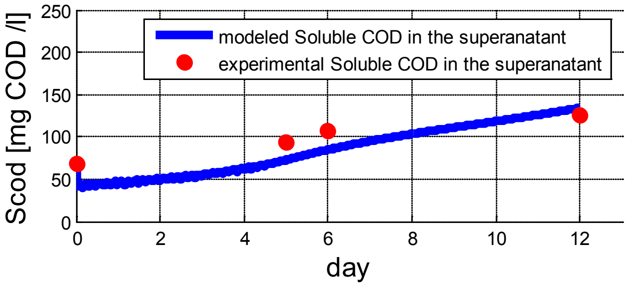

Fig. 6. Experimental and modelled soluble COD concentration fate in the batch reactor.

3.3. SMP sampling campaign in dynamic conditions

The ASM models extended with the SMPs concept are typically differentiated by the need of characterizing two fractions: influent Si and SMP fractions. In an ASM–SMP model, the influent Si cannot be anymore estimated by the effluent COD since effluent MPs have to be excluded and properly modelled. The procedure of Lu et al. [4] was employed to determine the influent Si.

With regards to the SMP measurements, a sampling campaign has been performed during the period 10.04.09–15.06.09. COD$_{sol}$ and SMPs have been quantified in supernatant and permeate (Figs. 7 and 8). In both cases SMPs were always found as expected smaller than the COD concentration. It is important to stress that, while the permeate sample is a flow proportional sample, the supernatant is a grab sample (the sludge water filtration impedes the collection of flow proportional samples). This implies that the supernatant will be much more dependent on the peak loads than the permeate samples.

If the SMP in the permeate is divided by the SMP in the supernatant, and plotted against the relative total daily influent flow, a correlation ($R^2 = 0.67$) appears between the fraction of non retainable SMPs and the total influent flow (Fig. 9). A similar correlation can be seen for the COD$_{sol}$ of the permeate and the supernatant (Fig. 9). In full scale, the higher the total flow, the higher the dilution, exception provided for the first flushes events. But a daily constant flow is diverted to the MBR process, since the limited membrane area restrains the MBR hydraulic dynamics. Thus higher influent flows generally imply lower loads to the MBR lane. The experimental results in Fig. 9 show a dependency among the influent load (*Y* axes) and the SMP removal (*X* axes). This finding will be discussed in the next section.

3.4. Model comparison

The ASM-SMP model has been implemented with the following procedure: (i) The ASM$_{2d}$ model [15,16] has been updated with the extended matrix; (ii) Influent file, influent converter parameters, removal rates of particulate matter in the pre-treatment, ASM$_{2d}$ kinetics parameters, dosed HAc and controls have not been modi-

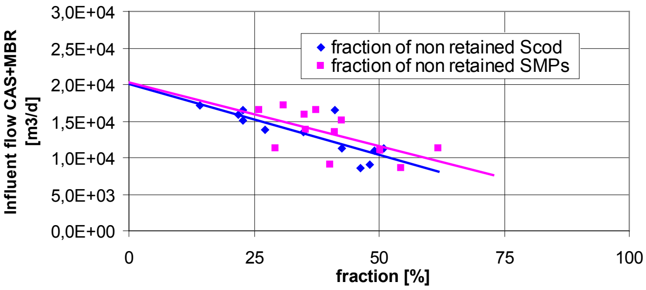

Fig. 9. Influent flow vs. “fraction of non retained COD_{sol}” and “fraction of non retained SMPs”.

fied. (iii) The effluent file has been modified in order to yield a Si concentration according to the experimental work. As a result of the lower Si concentration, the sludge production has to be fitted again; (iv) BAP specific kinetics parameters derived in the batch tests are inserted in the model; (v) UAP specific kinetics parameter will be inserted (as a trial and error procedure) in order to fit the SMPs in the permeate; (vi) SMP dynamic is completed by fine-tuning the retention factors at high-low flows; (vii) In the case of deviation, the real total aeration energy would be matched by fitting the specific aeration energy (in kg O_2 per KWh blown).

Since the matrix has been set in order not to include UAP mechanisms in the nitrification processes, ammonia effluent does not report significantly different results (Fig. 10). Moreover the slowly biodegradable content added in this procedure is not sufficient to yield significantly different denitrification rates. Fitting of the nitrate effluent is overall very good. Cumulative dose of HAc was also fitted since crucial when modelling the denitrification rates.

The average reduction of Si (from 25 mg COD/l to 17 mg COD/l) will correspond with a slightly higher particulate inert content Xi (since the total inert content does not change). In reality the Xi variation did not have a marked influence on the sludge production. Hence, the K_{bod} (0.27) was not modified. The introduction of SMPs did not reflect in a significantly different energy consumption.

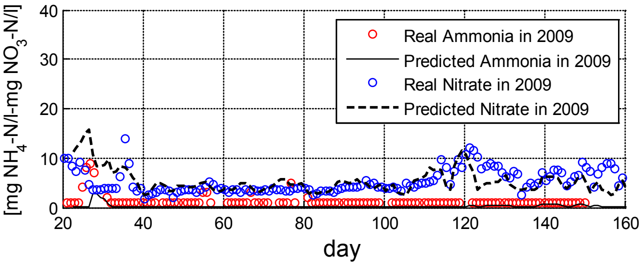

Fig. 10. Ammonia and nitrate predicted and measured in the MBR effluent.

Thus nutrients removal, sludge production and energy fitting did not receive further benefit from the SMP mod- The fractions of non retainable SMP and COD have been modelled as linear function of the incoming flow, according to experimental results and fine-tuned by a trial and error procedure. In terms of COD effluent, fluctuations are fitted, due to the modelled fraction of non retainable COD, and the model predictions are excellent (Fig. 11).

In terms of SMPs, several points must be commented: (i) BAP have been determined by inserting the specific parameters obtained during the experimental part, (fbap of 0.05 and $K_{h,bap}$ of 1×10^{-6} d^{-1}). The impact of the BAP fraction is minimal, and higher values of fbap do not lead to increased significant SMPs concentrations. It appears that most of the SMPs will have to be produced through the UAP metabolic path, being thus very sensitive to the influent loads; (ii) UAP have been fitted with a trial and error procedure. Results yield a fuap of 0.6 and $K_{h,bap}$ of 1×10^{-6} d^{-1}. SMPs in the permeate have been reasonably fitted along the days 110–150, but a poor fitting has occurred during the days 95–105, where the SMP effluent values were as high as the COD effluent (Fig. 12).

If the BAP concentrations are not so significant, the higher influent loads will necessarily generate higher SMPs, according to the UAPs definition. The previous Fig. 9 would then imply that SMPs are less retainable with increasing loads, i.e. increasing SMP loading rates. This result was already observed by Drews et al. [17]. Recent literature on SMP modelling has instead simplistically considered the COD retained fraction as a constant

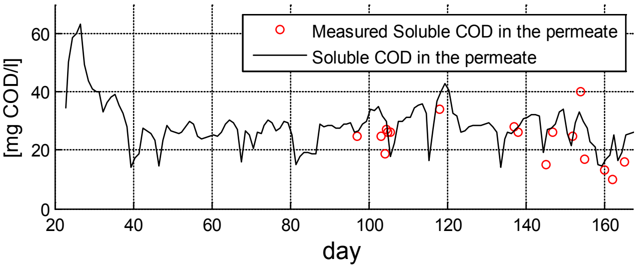

Fig. 11. Predicted and measured permeate soluble COD concentration.

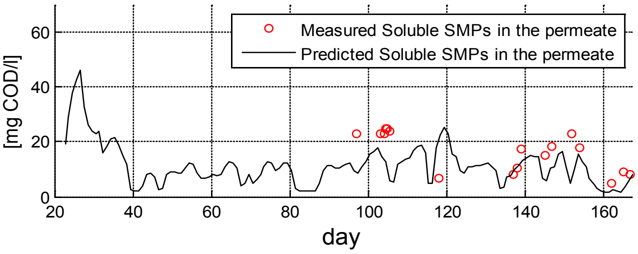

Fig. 12. Predicted and measured permeate SMP concentration.

parameter [18], ranging from 0 to 100%. The difference between this experimental finding and the recent literature (inter alia [19]) can be explained by the non-dynamic conditions of pilot scale academic works: low variability in flows and concentrations will yield quasi constant retention factors.

It must be reported that SMPs were slightly underestimated in the supernatant. These values are very difficult to be matched because the supernatant samples are grab samples (fetched always at 15.00 PM). Grab samples are certainly sensitive to the peak loads which might result in higher SMPs measured concentration. However, a model calibration based on shorter frequencies than a daily base would require a burdensome sampling work for long calibration campaigns in full scale.

With regards to the fouling rates, they have been computed in Fig. 13 (in lmh/bar/$m^3_{permeate}$) through the dataset of Fig. 14. Fouling rates are characterized by relatively constant values with the exception of high fouling rates for low MLSS concentrations (4–8 g/l after sludge inoculation). Fouling propensity of low MLSS concentrations (from day 20 to 40 in Figs. 12–14) has already been reported by Bin et al. [20]. When fouling rates are plotted against the supernatant SMP concentration, the results are very scattered and tend to show no correlation, meaning that SMPs do not trigger higher filtration resistances. This fact indicates a strong drawback in the use of these models since, at least in case of this full scale MBR, the main drive is not accomplished. It is thus strongly recommended to assure such correlation before starting this modelling procedure.

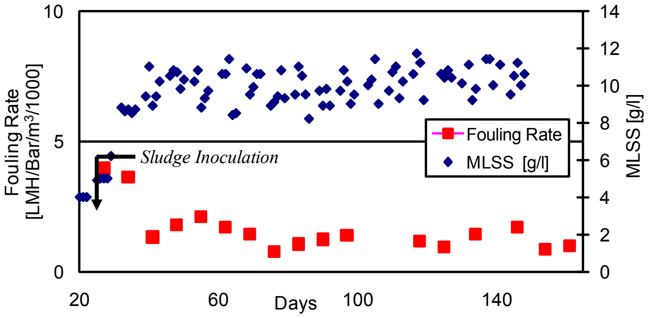

Fig. 13. Fouling rates (20.01.09–20.06.09).

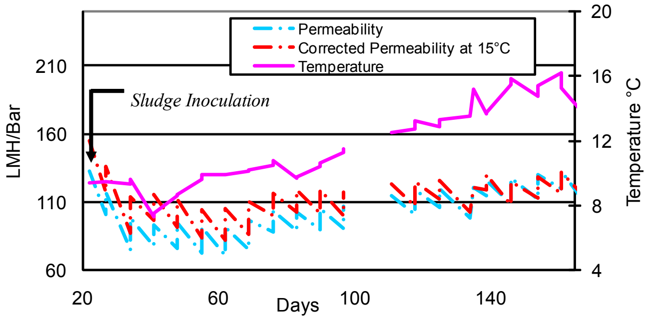

Fig. 14. Fouling history (20.01.09–20.06.09).

4. Conclusions

The work represents a rare application of the most advanced literature on ASM-based models extended for SMP modelling, in a full scale waste water treatment plant. A new matrix has been created based on the ASM_{2d} model. Main conclusions are:

- While the biomass associated products (BAPs) kinetics can be estimated, in tune with previous experiments, the utilization associated products (UAP) kinetics are instead hindered by storage phenomena (accountable only on ASM3) and by the non-uniformity between the polysaccharide fraction, easily biodegradable, and the protein fraction, which is refractory to biodegradation.
- The BAP modelling results in a fbap of 0.05 and $K_{h,bap}$ of 1×10^{-6} d^{-1}. UAP kinetics were fitted by trial and error procedures yielding a fuap of 0.6 and $K_{h,uap}$ of 1×10^{-6} d^{-1}. UAPs were found markedly predominant compared to the BAPs.
- The membrane rejection mechanism was identified as SMP loading rate dependent, showing the need of a more careful consideration towards this parameter when modelling in full scale or in a dynamic environment.
- The extension of an ASM model with SMPs and its experimental work impacts on the modelling exercise complexity. Some of the problems originated by the experimental work cannot be easily solved: (i) UAP kinetics estimation in batches is not accurate. (ii) The SMP batch experiments protocol is a burdensome procedure; (iii) Flow average samples cannot be collected for the sludge water samples, being the results thus very dependent on the daily peak loads. A more frequent sampling campaign would thus be required; (iv) the fine tuning of the membrane rejection factor appears a necessary adaptation. Both nutrients removal, sludge production and energy fitting did not receive further benefit from the SMP modelling.
- SMPs did not correlate with fouling rates in this full scale MBR, indicating a strong drawback, since the main drive for these models is thus not accomplished.

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