Section: Water Engineering

Transient responses of a laboratory-scale activated sludge plant to momentary high sludge loads

Pekka Stén[a], John Dahlbacka[b], Tom Lillhonga[b], Steven Ulbricht[c], Sonja Heikkilä[a], Juha Nieminen[a]

[a]Vaasa University of Applied Sciences, Finland
[b]Novia University of Applied Sciences, Finland
[c]Hochschule Wismar, University of Applied Sciences, Technology, Business and Design, Germany

Abstract

The performance of a laboratory-scale activated sludge plant (capacity approx. 1 dm^3 h^{-1}) during various conditions was studied and modeled. The apparatus consists of a primary clarifier, an anoxic selector, an aeration basin and a secondary clarifier. The fully automatized apparatus utilizes the preanoxic configuration, in which nitrate produced in the aerobic zone is recycled back to the anoxic selector which is situated between the primary clarifier and the aeration basin. Under steady-state conditions, using synthetic waste water as the influent, the COD reduction is over 95%, and the nitrogen reduction over 85%. Several spiking experiments at various operational conditions were carried out. In these experiments, in addition to the continuous influent flow, a known volume of concentrated synthetic wastewater was added directly to the aeration basin. The response of the plant was monitored by measuring the COD, as well as the NH_4^+ and NO_3^- concentrations of the effluent. Vibrational spectroscopic methods were also employed. The effluent was monitored with NIR and the N_2O emission with FTIR. After introducing the spike, COD of the effluent increases from a value of approx. 30 mg dm^{-3} (COD of the influent is approx. 300 mg dm^{-3}) to 200 mg dm^{-3} in 2–3 hours and comes down close to the initial value in an additional 5 hours. The behavior of NH_4^+ concentration in the effluent resembles that of COD, however with a delay of a few hours. The pH in the aeration basin is kept almost constant by PLC-controlled addition of limewater. After introducing the spike, the consumption of limewater goes down during 2–3 hours recovering to the initial values in 12 hours. A model based on mass transport and Monod kinetics was also used to interpret the results and to gain additional information about the process.

Keywords: wastewater treatment; activated sludge plant; laboratory-scale; NIR spectrometry; N_2O emission

1. Introduction

This paper describes an interdisciplinary study combining laboratory-scale wastewater treatment, automation, and in-line NIR (near infrared) spectrometry. The aim of the study was to evaluate the performance of our laboratory-scale continuous activated sludge wastewater treatment plant and to generate treated effluent with controlled variability for characterization by NIR spectrometry.

Several spiking experiments with momentary addition of high concentration influent directly to the aeration basin were carried out. A spiking approach has been rarely used [1], [2], but by generating such spikes, effluents of highly varying and controlled quality were provided for the NIR measurements. This wide variation and reference analyses by conventional methods is necessary for the in-depth development of mathematical calibration models for NIR data.

Laboratory-scale activated sludge plants are quite frequently used as research platforms and several studies have been published even in the 2010's. Lorestani *et al*. [3] compared the performance of the activated sludge system and compartmentalized activated sludge system for the biological treatment of a synthetic wastewater approximating that produced by the milk processing industry. Pholchan *et al.* [4] studied in a systematic way the effects of operating variables on microbial diversity and on the performance of laboratory-scale activated sludge reactors. Poutiainen *et al.* [5] studied the effect of varying anox-circulation schemes on the nitrogen removal efficiency and compared the denitrification efficiencies of ethanol and methanol in a post-denitrification unit. The main objective of the research of Esparza-Soto *et al.* [6] was to

quantify and characterize the effluent organic matter by advanced spectroscopic methods under various operational conditions. Szabolcs *et al.* [7] used a lab-scale SBR (sequencing batch reactor) to aid in process modeling and simulation.

Usually the laboratory-scale studies are carried out in batch reactors as done in the above mentioned papers [4], [6], [7]. However, even continuous processes have been studied recently as done by Lorestani *et al.* [3] and Poutiainen *et al.* [5]. The laboratory-scale wastewater treatment plant used in this study is similar to that used by Poutiainen *et al.* [5] and even our apparatus originates from the Environmental Science research group of University of Eastern Finland. Compared to the other set-ups mentioned above, the Finnish set-up is equipped with advanced instrumentation and automation.

Near infrared (NIR) spectroscopy is a powerful tool for industrial quality control and process monitoring [8]. This method can be described as rapid, non-destructive, non-invasive, deep radiation penetration, suitable for in-line use, in combination with little or no need for sample preparation [9]. Furthermore, the instruments can be said to be robust [10], as well as low in maintenance [11]. According to what was apparently the first publication describing the use of NIR spectroscopy for wastewater measurements, the concentration of urea, solids, and oil could be measured with a reasonable accuracy in samples of wastewater discharged from a biodiesel fuel production plant [12]. Later applications include the measurement of glycerol and methanol [13], [14], and perhaps more importantly the chemical oxygen demand (COD) [15–17], as well as the biological oxygen demand (BOD) [18]. In the case of COD, the reported accuracies (expressed as root mean square errors of prediction (RMSEP) [19]) are around 30 mg dm^{-3}. The preliminary results presented here are comparable to these findings, and perhaps of particular interest since only a few examples of actual in-situ measurement applications can be found [10], [20], [21].

2. Materials and methods

2.1. Description of the laboratory-scale wastewater treatment plant

Experiments were carried out at room-temperature in a laboratory-scale continuous-flow activated sludge reactor, which is capable of simultaneous removal of organic matter, phosphorus and nitrogen (Fig. 1). The experimental set-up is delivered by Savonia University of Applied Sciences and is similar to the apparatus described in detail by Poutiainen *et al.* [5].

In this set-up incoming effluent is pumped to the 3.9 dm^3 primary clarifier (which was, however, not used in this study), then flows gravitationally via the anoxic selector (3.2 dm^3) into the aeration basin (8.8 dm^3) where pressurized air is bubbled through air stone diffusers used also in domestic fish tanks. From the aeration basin the mixture of sludge and effluent flows into the secondary clarifier (6.0 dm^3). The clear overflow is discharged and the sludge taken from the bottom of the clarifier is returned to the anoxic selector to maintain a constant concentration of activated sludge in the aeration tank.

In our apparatus phosphorus removal is accomplished by precipitation using iron sulfate (the commercial product Kemwater PIX 105 of Kemira, Finland) which is dosed into the mixed liquor in the anoxic selector by a membrane pump (Grundfoss DME2-18A-PP/V/C-F-3111F). In our experimental set-up removal of nitrogen is realized by a preanoxic configuration, in which nitrate produced in the aerobic zone is recycled back into the anoxic compartment, which is situated between the primary clarifier and the aeration basin.

One peristaltic pump (Watson-Marlow 505 U) with 3–4 pump heads is employed for the influent as well as for the nitrate circulation from the end of the aeration basin to the anoxic selector and circulation of return sludge from the bottom of the secondary clarifier to the anoxic selector. Although all the pump heads are operated at the same rpm, different flow rates are achieved by varying inner diameters (4.8 mm, 8 mm, 6.4 mm) of the Marprene tubings used for ducting each flow.

The start-up sludge is taken from the large-scale wastewater treatment plant of the city of Vaasa. The sludge density and the solid retention time (SRT) are controlled by routing predetermined volumes of activated sludge to waste by another peristaltic pump directly from the aeration basin at predetermined times. The volume of waste sludge needed to maintain the SRT at 3–6 g dm^{-3} varied in this study between 0.7 and 1.5 dm^3/d.
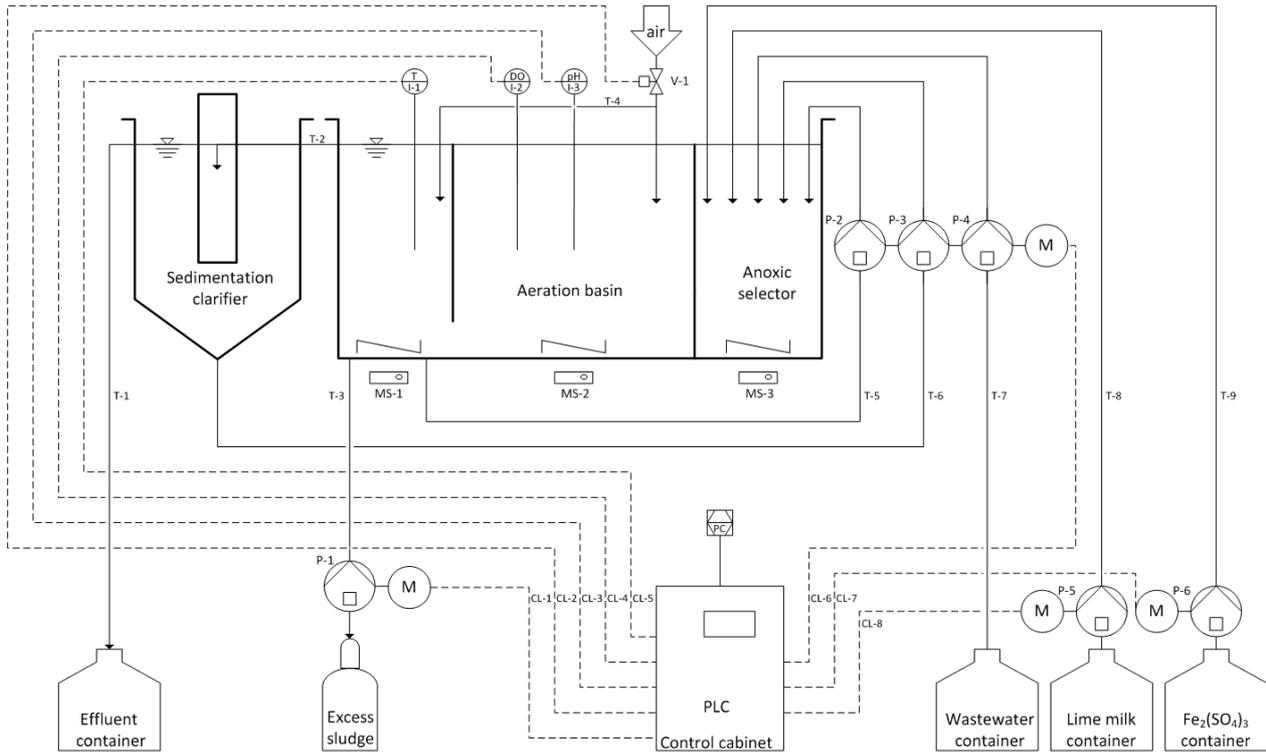
Fig. 1. Schematic diagram of the laboratory-scale wastewater treatment plant

2.2. Automation and data collection

The system, which was ordered by us from Savonia University of Applied Sciences, includes advanced automation by programmable logic control (PLC). The dissolved oxygen (DO) concentration and pH in the aeration basin are measured (Hach Lange LDO probe and Hach Lange Digital differential pH sensor DPD 1R1.99 connected to Hach Lange SC 200 controller) and used to control aeration rate and dosage of lime water by a membrane pump (Grundfoss DME2-18A-PP/V/C-F-3111F) to keep the dissolved oxygen concentration and pH at pre-set values. The original feed-back control system as ordered by us has been slightly modified in our laboratory and the PLC has been interfaced with a computer system for continuous data collection as depicted in Fig. 2.
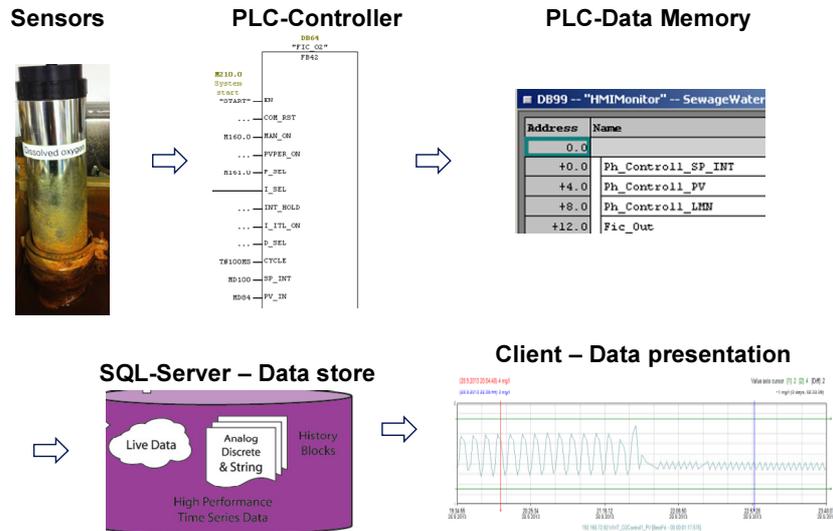

Fig. 2. The logical data flow in the data logging system

The data logging and analyzing system is based on fast industrial SQL-server and the Historian package from Invensys. The experiment data is gathered from the twelve measuring points (dissolved oxygen concentration and pH along with several pump speed and valve position parameters) at 100 ms interval. Each measurement is logged when it changes more than 1%. The total number of the logged measurements during the experimental campaigns in this project exceeds 100 000 000. The average data storing rate is 6.5 measurements / second. To reduce the number of the data at the analyzing phase several methods are available. The time weighted average method gives one measurement to represent each hour. The validity of the hourly data can be verified by retrieving interpolated measurement and also minimum- and maximum values for each one minute interval.

2.3. Operational conditions and plant monitoring, Experiments under steady-state conditions

The aim of the experiments was to evaluate the performance of a laboratory-scale continuous activated sludge wastewater treatment plant and to generate treated effluent with controlled variability for characterization by NIR spectroscopy.

Two series of tests were conducted in this study. The performance was evaluated by COD and nitrogen removal efficiency under steady state conditions at rather conventional values of the operating parameters at two different solid retention times (SRTs). The influent flow was 0.83 dm^3/h, anox-circulation was 1.71 dm^3/h (i.e. 206% of the influent) and return sludge circulation 1.29 dm^3/h (155%). Hence, the hydraulic retention time (HRT) in the main tank (anoxic zone 3.2 dm^3 + aerobic zone 8.8 dm^3) was 14.5 h and in the final clarifier (6.0 dm^3) 7.2 h.

The sludge age and density were controlled by routing activated sludge to waste directly from the aeration basin. The volume of waste sludge needed to maintain the sludge age at 12 days was 1.0 dm^3/d and at 8 days 1.5 dm^3/d resulting in sludge densities of 4.3 g dm^{-3} and 3.3 g dm^{-3}. Dissolved oxygen concentration and pH in the aeration basin were kept at 2.5 mg dm^{-3}and 7.7 using the PLC.

2.4. Operational conditions and plant monitoring, Spiking experiments

In the other experimental series several spiking experiments under various operational conditions were carried out. In these experiments, in addition to the continuous influent flow, a known volume of concentrated synthetic wastewater was added directly to the aeration basin resulting in a momentary high organic loading. In the experiment discussed in detail below, the influent COD was 360 mg dm^{-3} and the flow 1.11 dm^3/h. Under these conditions the plant was subjected to a spike of 300 ml concentrated synthetic waste water having a COD value 29 000 mg dm^{-3}. Hence a COD load of 8.7 g was added which should be compared to the COD load of 12 dm^3 (the volume of the anoxic and aeration basins) of influent, i.e. 12 dm^3·360 mg dm^{-3} = 4.3 g.

In this series three process and operational parameters were varied: dissolved oxygen concentration in the aeration basin varied between 1.5 and 3.5 mg dm^{-3}, hydraulic retention time varied between 9.6 and 14.5 hours and sludge density between 3 and 6 g dm^{-3}.

The response of the plant was monitored by sampling the effluent at regular intervals (0.5 or 1 h) for COD and NH$_4^+$ analyses. The samples were stored under refrigeration and analyzed within a day of collection after centrifugation. In addition to sampling, the effluent was continuously monitored with NIR spectrometry. During some of the spiking experiments samples of the exhaust gas stream were periodically analyzed for N$_2$O with FTIR spectrometry.

2.5. Synthetic sewage

As natural sewage is prone to strong temporal variations in strength, especially in spring due to the huge amount of melt water, we decided to use synthetic wastewater to minimize the variability in the influent. Several recipes for synthetic wastewater have been published. Despite criticism [22], we decided to use the traditional peptone- and meat extract-based synthetic sewage closely resembling the recipe described in the OECD protocol 209 [23]. A concentrate was prepared by weighing the following components in 1 liter of water: 16 g peptone, 11 g meat extract, 3 g urea, 0.7 g NaCl, 0.4 g CaCl$_2$ ·2 H$_2$O, 0.2 g MgSO$_4$ · 7H$_2$O, 2.8 g KH$_2$PO$_4$ · 3 H$_2$O.

Table 1.Characterization of the influent used in steady state experiments

Parameter	
BOD$_7$	310 mg dm^{-3}
COD	440 mg dm^{-3}
N (total)	70 mg dm^{-3}
PO$_4^{3-}$	8.9 mg dm^{-3}
alkalinity	1.4 mmol dm^{-3}

This concentrate was stored frozen in plastic bottles containing 300–600 ml batches, which were melted, diluted in cold tap water and taken directly into use as the influent. In the steady state experiments, a one day's worth of dilutant of 300 ml in 20 dm^3 of tap water was prepared. The characteristics of the dilutant are tabulated in Table 1. The 20 dm^3 -canister containing the influent was kept in an insulated cold box at a temperature below 10 °C. The COD and total nitrogen concentration remained constant during this period but a minor increase in the ammonium concentration was observed.

2.6. Analyses

The performance of the system was controlled from influent and effluent samples. The COD was determined in accord with the Finnish standard method SFS 5504 [24] with a closed reflux kit (Low range 0–150 mg dm^{-3} COD, Hach, Co) followed by titration with 0.05 M $(NH_4)_2Fe_2(SO_4)_2$ solution using ferroin as the end-point indicator. The sludge concentration of the mixed liquor was measured after filtration through Whatman GFA filters and drying for 2 h at 105 °C in accord with the Finnish standard SFS-EN 872 [25]. BOD was measured using WTW's OxiTop system.

Ammonium was determined spectrophotometrically by the phenolhypochlorite method, also in accord with the Finnish standard SFS 3032 [26]. Nitrate was determined by an ion chromatograph (Dionex ICS 1100), according to the manufacturer's instructions [27]. Portable Hach Lange spectrophotometer was used for analysis of total nitrogen according to the manufacturer's instructions. Iron was analyzed by ICP in accord with the standards SFS-EN ISO 17294-1 [28] and 17294-2 [29] as well as phosphorus from a few samples. Alkalinity was, likewise determined in accord with the Finnish standard method SFS 3005 [30] by titrating with HCl to a potentiometric end point of pH 4.5.

In addition to the in-line measurements described earlier, an optical-based portable WTW 3410 multimeter was used for measuring the dissolved oxygen concentration and a portable Mettler-Toledo MP 120 for measuring pH. A portable FTIR gas analyzer, Gasmet™ DX4000, was used to monitor N_2O emissions during the spikes.

2.7. NIR spectrometry

The NIR spectra were collected with a GetSpec spectrometer, model #: NIR-256L-1.7T1 (Sentronic GmbH, Gostritzer Str. 61-63, D-01217 Dresden, Germany). This diode array instrument has an Indium-Gallium-Arsenide (InGaAs) detector with 256 elements. The spectral range is 900–1700 nm. The spectrometer was equipped with a dip probe with variable path length (getSpec.com, Gostritzer Str. 61-63, D-01217 Dresden, Germany), which was set to 1 mm, and an AvaLight-HAL tungsten halogen light source (Avantes BV, Oude Apeldoornseweg 28, The Netherlands). The spectra were collected as absorbance spectra (averaged for 512 scans) from 905 to 1682 nm, at a data resolution of 3 nm using the Spec32 v. 1.5.6.8 software as interface. The partial least squares (PLS) [31] models were calculated using the PLS_Toolbox v. 7.3.1 (Eigenvector Research, Inc. 3905 West Eaglerock Drive Wenatchee, WA 98801, USA), together with MATLAB R2011b (The MathWorks AB, Kista, Sweden). Cross validations were performed as 10-fold random splits.

2.8. Modeling

The model based on the Monod equation and mass balances consisted of 9 differential equations, four to describe the COD concentration in the four basins of the plant, four to describe the biologically inactive organic matter in the basins, and one to describe sludge density. In the case of sludge density, the Monod equation was used to model the specific growth rate of the sludge. The concentration of the limiting substrate for growth was specified as the COD concentration subtracted from the concentration of biologically inactive organic, i.e. essentially a computed BOD concentration.

3. Results and discussion

3.1. Steady-state experiments

The performance of the plant under steady-state conditions was tested at two different SRTs. The analysis results given in Table 2 are average values of several measurements taken during four days of operation under the same conditions. Removal of organic matter works well at both SRTs as indicated by high COD and BOD reduction of 95–96% and 99%.

The concentration of total nitrogen in the synthetic sewage dilutant used in this study is typical for that of domestic sewage. However, in the synthetic sewage nitrogen is in the form of urea or organic nitrogen in contrast to ammonium in domestic sewage reaching the treatment plants. By comparing the effluent total nitrogen concentration to the concentration of nitrogen in the form of nitrate in the effluent, it is obvious that nitrogen in the influent is easily converted to ammonium, which is further easily oxidized to nitrate as indicated by the marginal concentration of ammonium in the effluent. Oxygen concentration in the anoxic selector remained very low, usually below the detection limit of the portable DO meter. This was the case also in an experiment, where the anox-circulation rate was doubled, which however resulted in a decrease in total nitrogen removal efficiency. Some denitrification seems to take place even in the final clarifier as indicated by the decrease in DO (0.4 mg dm^{-3}) and increase in pH which was 8.2 in the effluent compared to 7.7 in the aeration basin.

Table 2. The performance of the laboratory-scale wastewater treatment plant at two sludge ages.

Parameter	Experimental series I	Experimental series II
Sludge age	12 d	8 d
Sludge density	4.3 g dm^{-3}	3.3 g dm^{-3}
Wasting rate	1.0 dm^3/d	1.5 dm^3/d
COD (influent)	440 mg dm^{-3}	440 mg dm^{-3}
COD (effluent)	21 mg dm^{-3}	17 mg dm^{-3}
COD removal efficiency	95%	96%
BOD$_7$(influen)	310 mg dm^{-3}	310 mg dm^{-3}
BOD$_7$(effluent)	3 mg dm^{-3}	3 mg dm^{-3}
BOD removal efficiency	99%	99%
N$_{tot}$ (influent)	70 mg dm^{-3}	70 mg dm^{-3}
NO$_3^-$–N (effluent)	11 mg dm^{-3}	7.4 mg dm^{-3}
NH$_4^+$–N (effluent)	not analyzed	0.07 mg dm^{-3}
N$_{tot}$ (effluent)	not analyzed	7.5 mg dm^{-3}
Nitrogen removal efficiency	84%	89%
P$_{tot}$ (influent)	8.9 mg dm^{-3}	8.9 mg dm^{-3}
P$_{tot}$ (effluent)	not analyzed	0.14 mg dm^{-3}
P removal efficiency		98%
alkalinity (influent)	1.5 mmol dm^{-3}	1.5 mmol dm^{-3}
alkalinity (effluent)	3.4 mmol dm^{-3}	3.3 mmol dm^{-3}
suspended solids (effluent)	10 mg dm^{-3}	2 mg dm^{-3}

On the whole, under such conditions the system performs well with almost perfect BOD removal, a COD removal efficiency of about 95% and nitrogen removal efficiency of about 85%. The system performed somewhat better at a shorter sludge age which implies a lower sludge density. It should be noted that the concentration of suspended solids in the effluent dropped to the very low level of 2 mg dm^{-3}.

3.2. Spiking experiments

The synthetic wastewater contains considerable amounts of urea. After the addition of a spike the ammonium concentration in the effluent increases rapidly, apparently due to the ammonification of urea (Fig. 3a). At steady state conditions, the concentration of ammonium is essentially zero, but as can be seen in Fig. 3a, the spiking resulted in an ammonium concentration of 40 mg dm^{-3}at 4 h for this particular spike. The increase in ammonium concentration was also highly (negatively) correlated with a decrease in the nitrate concentration in the effluent (Fig. 3a). This was interpreted as an indication that the high momentary load had a negative impact on the efficiency of the ammonia-oxidising bacteria. The spike also resulted in an increase in pH resulting in an decrease in the flow rate of limewater (Fig. 3b).

The results from the measurements of the nitrous oxide concentration above the aerated basin during three spikes are shown in Fig. 4. Quantitative results were obtained using the calibration model delivered with the hardware. However, due to a considerable sampling error, the ppm values given in Fig. 4 should be perceived as relative. The sampling error was a result of the collection and the extraction of exhausted gases above the aerated basin. When a sample is extracted through the funnel, surrounding air replaces the extracted gas. In addition, the aeration rate of the basin is also varying. Typically, three consecutive samples were collected. The nitrous oxide concentration in the first sample was always considerably higher than in the following two samples. The data shown in Fig. 4 represents the result from the second of at least two consecutive sampling. As a consequence, the information value of Fig. 4 is somewhat limited. However, seen as trends, the results indicate that the spiking caused an increase in the nitrous oxide concentration, reaching a maximum value somewhere around 8–12 hours after the spike.
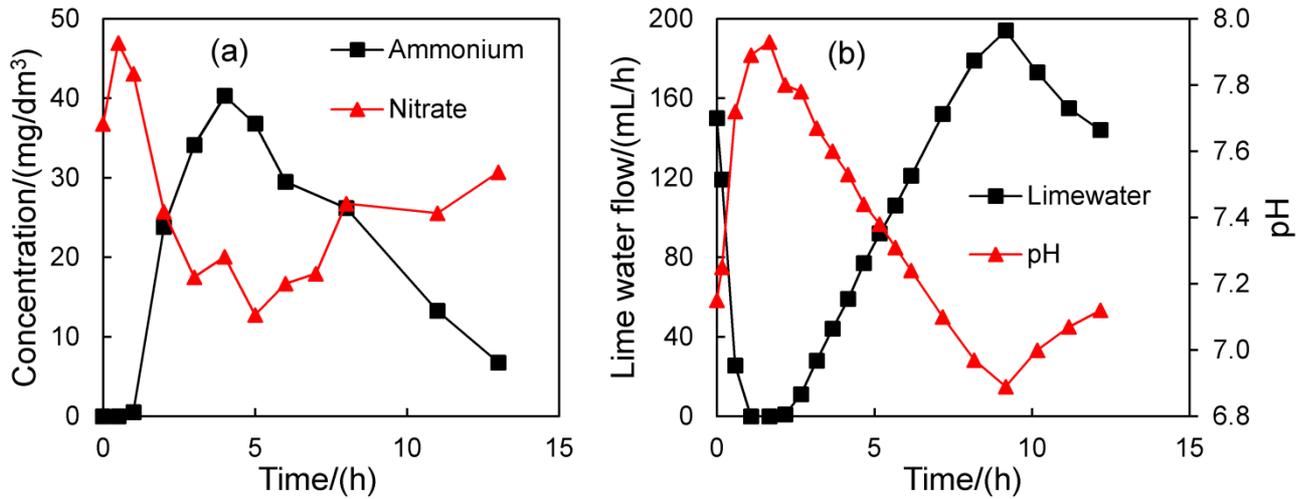
Fig. 3. The ammonium and nitrate concentration in the effluent (a), the pH in the aerated basin plotted together with the limewater consumption (b), vs. time relative to the spike addition
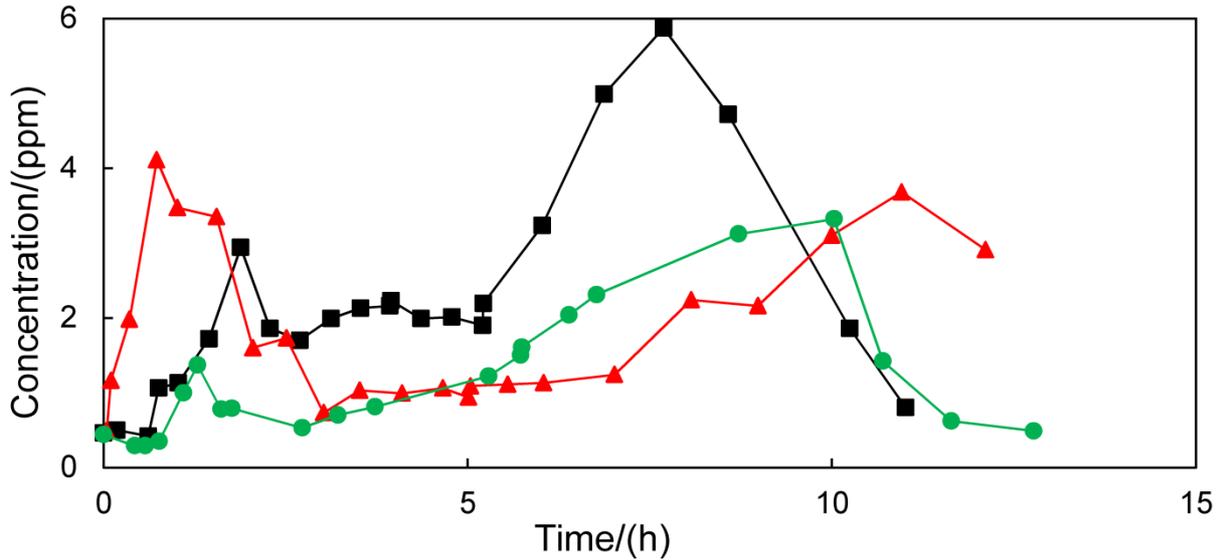

Fig. 4. The nitrous oxide concentration above the aerated basin measured with the Gasmet instrument, vs. time relative to the spike addition

Fig. 5 shows the results from NIR and the Monod equation based modelling of a spike response in the effluent. At this point, the only conclusions that can be drawn from the modelling based on the Monod equation and mass balances, is that it is fairly easy to fit a model of this type to the reference data. However, studying the impact of the operational conditions on the parameters in the Monod equation will hopefully give some new insight into the behaviour of this activated sludge process. Fig. 5 also includes the cross validation predictions of a PLS model based on the NIR measurements. The cross validation error of this model is only 7 mg dm^{-3}. This is very promising, but at the same time it should be stressed that this is a result from cross validation, i.e. not from an external data set or from a separate spike for that matter. The whole collected wavelength region was used in the calibration model, utilising 7 PLS components and a 7 point second order polynomial Savitzky-Golay derivative, followed by auto scaling as spectral pre-processing. A spline function was also applied on the reference measurements in order to obtain an estimate the COD concentration for every NIR spectrum collected.
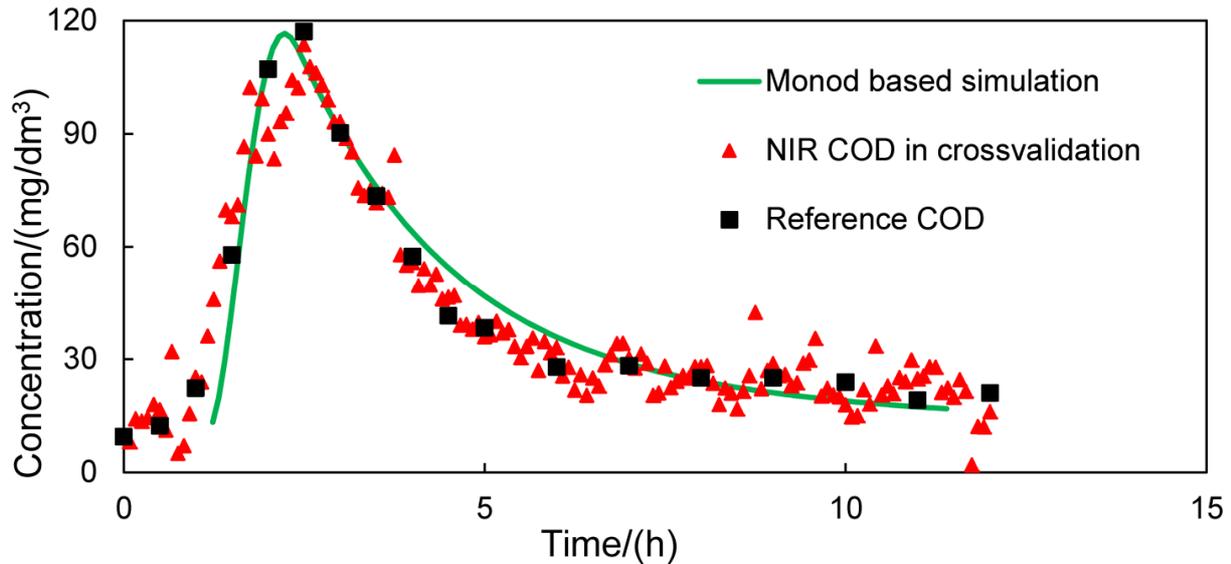
Fig. 5. The COD value in the effluent as determined using the reference method, as cross validation predictions of a PLS model based on the NIR measurements, and with the mathematical model based on the Monod equation, vs. time relative to the spike addition

4. Conclusions

The results presented are promising, although almost all the information available at this point is of a preliminary nature. Both COD removal and denitrification worked very well during steady state conditions. The Gasmet measurements clearly indicate that a high sludge load triggers nitrous oxide production. When sampling from ambient air the absolute concentration readings are of minor importance. The important information is that the nitrous oxide production is increased after a spike, and that the elevated level prevails for a period comparable with at least one hydraulic retention time. The mathematical modelling of the plant showed that the simple model used could describe the impact of a spike on the COD concentration in the effluent well. This is not something new, but further studies on how the operating parameters affect the parameters in the Monod equation might prove useful. The on-line NIR measurements on the effluent were remarkably accurate at least as in terms of cross validation predictions. Further modelling is needed to fully validate the method, i.e. on-line COD predictions on fully external spikes. In brief, a considerable amount of data was generated during this study and hopefully the analysis of this will bring new insights into the field of activated sludge treatment of wastewater.

Acknowledgements

References

[1] Alinsafi, A.; Adouani, N.; Beline, F.; Lendormi, T.; Limousy, L.; Sire, O. 2008. Nitrite effect on nitrous oxide emission from denitrifying activated sludge, *Process Biochemistry* 43(6): 683–689. http://dx.doi.org/10.1016/j.procbio.2008.02.008

[2] Wareham, D. G.; Hall, K. J.; Mavinic, D. S. 1993. Real-time control of aerobic-anoxic sludge digestion using ORP, *Journal of Environmental Engineering* 119(1): 120–36. http://dx.doi.org/10.1061/(ASCE)0733-9372(1993)119:1(120)

[3] Lorestani, A. A. Z.; Bashiri, H.; Asadi, A.; Bonakdari, H. 2012. Comparison of different fluid dynamics in activated sludge system for the treatment of a stimulated milk processing wastewater: Process analysis and optimization, *Korean Journal of Chemical Engineering* 29(10): 1352–1361. http://dx.doi.org/10.1007/s11814-012-0029-9

[4] Pholchan, M. K.; Baptista, J. C.; Davenport, R. J.; Curtis, T. P. 2010. Systematic study of the effect of operating variables on reactor performance and microbial diversity in laboratory-scale activated sludge reactors, *Water Research* 44(5): 1341–1352. http://dx.doi.org/10.1016/j.watres.2009.11.005

[5] Poutiainen, H.; Laitinen, S.; Pradhan, S.; Pessi, M.; Heinonen-Tanski, H. 2010. Nitrogen reduction in wastewater treatment using different anox-circulation flow rates and ethanol as a carbon source, *Environmental Technology* 31(6): 617–623. http://dx.doi.org/10.1080/09593331003592246

[6] Esparza-Soto, M.; Nunez-Hernandez, S.; Fall, C. 2011. Spectrometric characterization of effluent organic matter of a sequencing batch reactor operated at three sludge retention times, *Water Research* 45(19): 6555–6563. http://dx.doi.org/10.1016/j.watres.2011.09.057

[7] Szabolcs, S.; Botond, R.; Ildiko, M.; Beata, A.; Szabolcs, L.; Niculae, D. R. 2010. Lab scale sequencing batch reactor construction and characterization for dynamic modelling with activated sludge model, *Studia Universitatis Babes-Bolyai, Chemia* 55(Spec. Issue): 119–133.

[8] Siesler, H. W.; Ozaki, Y.; Kawata, S.; Heise, H. M. (Eds.) 2002. *Near-infrared spectroscopy: principles, instruments, applications*. Weinheim: Wiley.

[9] Pasquini, C. 2003. Near infrared spectroscopy: fundamentals, practical aspects and analytical applications, *Journal of the Brazilian Chemical Society* 14(2): 198-219. http://dx.doi.org/10.1590/S0103-50532003000200006

[10] Pons, M. N.; Bonté, S. L.; Potier, O. 2004. Spectral analysis and fingerprinting for biomedia characterisation, *Journal of Biotechnology* 113(1): 211–230. http://dx.doi.org/10.1016/j.jbiotec.2004.03.028

[11] Dias, A. M. A.; Moita, I.; Páscoa, R.; Alves, M. M.; Lopes, J. A.; Ferreira, E. C. 2008. Activated sludge process monitoring through in situ near-infrared spectral analysis, *Water Science and Technology* 57: 1643–1650. http://dx.doi.org/10.2166/wst.2008.147

[12] Suehara, K. I.; Owari, K.; Kohda, J.; Nakano, Y.; Yano, T. 2007. Rapid and simple determination of oil and urea concentrations and solids content to monitor biodegradation conditions of wastewater discharged from a biodiesel fuel production plant, *Journal of Near Infrared Spectroscopy* 15(2): 89–96. http://dx.doi.org/10.1255/jnirs.721

[13] Kohda, J.; Ooshita, K.; Nakano, Y.; Yano, T. 2008. Measurement of the glycerol concentration during the microbial treatment of the wastewater from the biodiesel fuel production plant using near infrared spectroscopy, *Journal of Near Infrared Spectroscopy* 16(3): 199–204. http://dx.doi.org/10.1255/jnirs.804

[14] Kawai, S.; Kohda, J.; Nakano, Y.; Yano, T. 2009. Predicting methanol and glycerol concentrations in microbial treated wastewater discharged from a biodiesel fuel production process using near infrared spectroscopy, *Journal of Near Infrared Spectroscopy* 17(1): 51–58. http://dx.doi.org/10.1255/jnirs.825

[15] Pan, T.; Chen, W. W.; Chen, Z. H.; Xie, J. 2011. Waveband selection for NIR spectroscopy. Analysis of wastewater COD, *Key Engineering Materials* 480: 393–396. http://dx.doi.org/10.4028/www.scientific.net/KEM.480-481.393

[16] Pan, T.; Chen, W. W.; Huang, W. J.; Qu, R. T. 2012. Model optimization for near-infrared spectroscopy. Analysis of chemical oxygen demand of wastewater, *Key Engineering Materials* 500: 832–837. http://dx.doi.org/10.4028/www.scientific.net/KEM.500.832

[17] Jin, C.; Cen, H.; Liu, F.; He, Y. 2009. Study on an advanced treatment of domestic wastewater by bio-filtration and water-quality measurement system, in. *Proc. of the fifth International Conference on Natural Computation ICNC'09*. IEEE, Vol. 3, 644–648.

[18] Yang, Q.; Liu, Z.; Yang, J. 2009. Simultaneous determination of chemical oxygen demand (COD) and biological oxygen demand (BOD$_5$) in wastewater by near-infrared spectrometry, *Journal of Water Resource and Protection* 4: 286–289. http://dx.doi.org/10.4236/jwarp.2009.14035

[19] Næs, T.; Isaksson, T.; Fearn, T.; Davies, T. 2002. *A user friendly guide to multivariate calibration and classification*. Chichester, UK.: NIR Publications.

[20] Páscoa, R.; Lopes, J.; Lima, J. 2008. In situ near infrared monitoring of activated dairy sludge wastewater treatment processes, *Journal of Near Infrared Spectroscopy* 16: 409. http://dx.doi.org/10.1255/jnirs.803

[21] Sarraguça, M. C.; Paulo, A.; Alves, M. M.; Dias, A. M.; Lopes, J. A.; Ferreira, E. C. 2009. Quantitative monitoring of an activated sludge reactor using on-line UV-visible and near-infrared spectroscopy, *Analytical and bioanalytical chemistry* 395(4): 1159–1166. http://dx.doi.org/10.1007/s00216-009-3042-z

[22] Boeije, G.; Corstanje, R.; Rottiers, A.; Schowanek, D. 1999. Adaptation of the CAS test system and synthetic sewage for biological nutrient removal. Part I: Development of a new synthetic sewage, *Chemosphere* 38(4): 699–709. http://dx.doi.org/10.1016/S0045-6535(98)00311-7

[23] OECD Guideline for Testing of Chemicals 1984. *Activated sludge, Respiration inhibition test 209*. Paris: Organization for Economic Cooperation and Development.

[24] SFS 5504. 1988. *Determination of chemical oxygen demand (COD$_{Cr}$) in water with the closed tube method. Oxidation with dichromate*. Helsinki: Finnish Standards Association.

[25] SFS-EN 872. 2005. *Water quality. Determination of suspended solids. Method by filtration through glass fibre filters*, Helsinki: Finnish Standards Association.

[26] SFS 3032. 1976. *Determination of ammonia nitrogen of water*. Helsinki: Finnish Standards Association.

[27] Dionex Corporation 2010. *Product manual for IonPac AG22, IonPac AS22, IonPac AG22-Fast, IonPac AS22-Fast. Fast analysis of the common inorganic anions using an optimized flow rate (AS22)*. 20.

[28] SFS-EN ISO 17294-1 (eng.). 2006. *Water quality. Application of inductively coupled plasma mass spectrometry (ICP-MS). Part 1: General guidelines*. Helsinki: Finnish Standards Association.

[29] SFS-EN ISO 17294-2 (eng.) 2005. *Water quality. Application of inductively coupled plasma mass spectrometry (ICP-MS). Part 2: Determination of 62 elements*. Helsinki: Finnish Standards Association.

[30] SFS 3005. 1981. *Alkalinity and acidity in water. Potentiometric titration*. Helsinki: Finnish Standards Association.

[31] Geladi, P.; Kowlaski, B. 1986. Partial least square regression: A tutorial, *Analytica Chimica Acta*, 185(1): 1–17. http://dx.doi.org/10.1016/0003-2670(86)80028-9