On-line monitoring equipment for wastewater treatment processes: state of the art

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Abstract A (non-exhaustive) survey of new and existing technologies for the monitoring of wastewater treatment plants is presented. Emphasis is given to the way these sensors can provide insight in the ongoing (bio-) processes. Three different uses for sensors can be found: for monitoring (operator support), in automatic control systems and as tools for plant auditing/optimization/modelling by consultants. From this, sensors have been classified in two basic types: (i) reliable, simple and low maintenance sensors for day-to-day monitoring and control and (ii) advanced, higher maintenance sensors that are used in auditing, model calibration and optimisation. The paper is organized according to the typical unit processes of biological wastewater treatment systems: anaerobic digestion, activated sludge, nutrient removal and sedimentation. Attention is drawn to a number of practical problems associated with the use of sophisticated sensors in the harsh (dirty) conditions of wastewater treatment processes. The use of autocalibration and built-in sensor checks, cleaning systems and reliable sample preparation units is illustrated. The paper ends with a discussion of the applicability of the different sensors.

Keywords Activated sludge; anaerobic digestion; nutrient removal; on-line monitoring equipment; sedimentation

Introduction

Monitoring and control of wastewater treatment plants rely on four building blocks: 1) insight into the process as summarized in a proper process model; 2) sensors that provide on-line data; 3) adequate monitoring and control strategies and 4) actuators that implement the controller output. Although the chain itself has strengthened considerably during the last decades – with significant breakthroughs in control theory and ever increasing sensor capabilities – the wastewater treatment problem itself evolved as well. Demands on water quality became more and more stringent, requiring more advanced treatment systems able to comply with (tighter) standards not only for organic carbon, but also for nitrogen and phosphorus (nutrient) levels. Hence, increasingly complex treatment systems must be run and yield ever increasing effluent water quality.

A common perception is that sensors represent the weakest link for implementing on-line process control of wastewater treatment plants (Harremoës *et al*., 1993). However, the performance and reliability of many on-line sensors (e.g. nutrient sensors, respirometers) have improved remarkably during the last decade and can today be used directly in many different control strategies (Jeppsson *et al*., 2002). The probably most fundamental barrier for the widespread acceptance of new sensors is that existing wastewater treatment plants were not designed for their use in real-time control systems. This is clearly exemplified in the lack of flexible and controllable actuators. Moreover, the fact that plant design was done in such a way that the effluent quality could be guaranteed without advanced control strategies (that rely on the new monitoring equipment) has resulted in over-dimensioned plants. Hence, although at this stage effluent criteria may still be reached by these systems, the implementation of some new sensor technology seems unavoidable as criteria become more stringent or increasing waste loads must be treated. For outdated treatment plants that need considerable upgrading investments, the use of new monitoring equipment should be regarded as a valuable alternative to increased reactor volumes.

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When trying to classify the numerous sensor systems available in the industry, three classification systems are mentioned here. The first classification is based on the functional application of the sensors. As mentioned above, sensors are used in automatic control systems, but very often they are used (only) for monitoring purposes, i.e. providing information to the operators about the state of the plant that supports them in day-to-day operation. Next to these 2 uses, sensors are also quite often applied by consultants to audit treatment plants, and eventually to calibrate models that are subsequently used for process optimization or evaluation of upgrade scenarios (Petersen *et al.*, 2002a).

A second classification directly follows from this, but focuses on the complexity of monitoring systems. In essence, sensors can be classified into, on the one hand, simple, reliable and low maintenance sensors that are used in day-to-day monitoring and automatic control systems and, on the other hand, advanced, maintenance-intensive sensors that are typically found in auditing and model calibration activities.

In this paper some evolving technologies are reviewed and classified according to the unit process they are intended for: anaerobic digestion, activated sludge, nutrient removal and sedimentation. The potential use and the practical experience gained so far will be presented. Attention will be drawn to the basic problems using sensors (Harremoës *et al.*, 1993): reliability, fouling, calibration and for some of them, sampling techniques.

General

Central to all wastewater treatment plants are flowing water, solids and gases. The physical properties of the three phases are worth monitoring and since these measurements are not specific for any biological process, these variables are treated separately in this section. Sensors measuring characteristics specific to one or more unit processes in a treatment plant will be discussed in the corresponding sections.

Temperature

This is a classic measurement, typically with a thermistor. It is a rather important variable for anaerobic digesters where temperature control is often implemented.

Pressure

Pressure measurements are traditional on wastewater treatment plants as well, especially for alarm functions in aeration and anaerobic digesters.

Liquid level

Common principles used to monitor water levels are: floats with an internal electric switch; conductivity switches; (differential) pressure transducers; capacitance measurements and ultrasonic level detection. The first two techniques are only useful for on/off level detection and mostly serve alarm functions. Differential pressure and ultrasonic equipment give a continuous signal, the latter being more precise but also sensitive to foam.

Flow of liquid/gas

Instruments for the monitoring of gas and liquid flows are ubiquitous in wastewater treatment. Harremoës *et al.* (1993) give an extensive overview of liquid flow measurement techniques and point to the importance of proper installation for guaranteed accuracy. Measurements are based on the change in water level (see above) as a result of an obstacle in the water flow path (Venturi principle). In addition, electromagnetic sensors are applied. For gas flow measurements recurrence is made to rotameters and, less common, thermal mass flow meters.

pH

It is normal practice to install pH electrodes in a treatment plant. Immersion of these probes in "sticky" sludge has encouraged the development of different cleaning strategies: hydraulic (water spray), mechanical (brush), chemical (rinsing with cleaning agent) or ultrasonic cleaning. With these techniques longer periods without maintenance can be attained. Harremoës *et al.* (1993) state that poor or no automatic cleaning may cause problems. Self-diagnosis has been integrated in advanced systems. In the simpler implementations, sensors are duplicated and their readings compared. More sophisticated set-ups include automated checks of the impedance of the diaphragm and the glass electrode, while tests performed during (automatic) calibration may point to other sensor deficiencies.

Although pH is a variable that is important in all biological processes, its value is especially critical in anaerobic digestion and nitrification where important quantities of protons are released, eventually leading to acidification and process failure. Hence, its measurement and control are important. However, in the case of wastewaters with high buffering capacity, pH measurements may be rather insensitive to indicate process changes and are therefore not advisable for process supervision and control. In such cases they may be replaced with bicarbonate measuring systems (see below).

Conductivity

Sensors measuring conductivity are used to monitor influent composition changes (Teichgräber, 1993) and are also at the basis of control strategies for chemical phosphorus removal (Lauer *et al.*, 1993). Subject to fouling as well, these sensors should be equipped with cleaning systems. Moreover, as the principle of the measurement requires applying a voltage over the electrodes, an alternating current is essential to eliminate electrode polarization.

Biomass/suspended solids

Probably the most important variable in wastewater treatment processes is the suspended solids concentration (SS). Three measuring principles have found application: optical measurement, ultrasound and dielectric spectrometry.

Scattering of incident light and absorbance by suspended particles has been a traditional off-line biomass estimation method (Kennedy *et al.*, 1992). With the advent of sensitive light detectors, sensors have been developed capable of automating the measurement of optic effects in an illuminated sample. Although it is impossible to make a direct (a priori) calculation of the dry weight concentration from any "optical density" measurement, the systems can provide reasonable estimates provided regular calibration is performed. Different principles have been brought to practice. Figure 1 illustrates the different phenomena occurring when a sample with (sludge) particles is illuminated. Part of the light is absorbed, another part is allowed to pass the sample (transmission), and, finally, light scattering in all directions occurs. Scattering is not homogeneous with the angle: forward scattering is the more pronounced, while backscattering is the least effective. The scattering over 90º gives light intensities between both extremes; its measurement is also known as nephelometry.

Depending on the solids concentration, one or the other light measurement will be the more beneficial. For instance, transmission measurements will provide a small signal to noise ratio in case of low SS levels (0–100 mg/l) as only a very small percentage of the incident light will be absorbed. Hence, scattering techniques are preferred for low solids samples, typically effluent turbidity, while light absorption is favoured in case mixed liquor or return sludge concentrations are to be measured. Backscattering is also rather advantageous in high solids systems where absorption may be too high to allow its

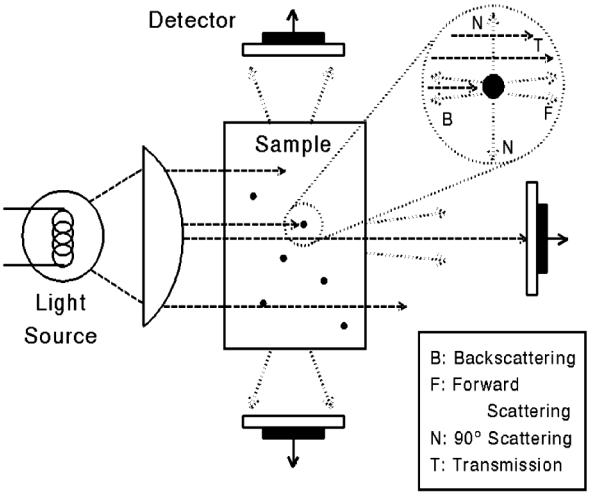


Figure 1 Illustration of the integrated sensor set-up

measurement. Sample dilution may be necessary at higher concentration (Yano *et al*., 1993). Recently, Jones *et al*. (1998) were able to measure wider concentrations ranges by combining multiple angle laser light scattering with chemometric techniques.

Most of the commercially available sensors use a source that emits light in the lower-visible and/or near-infrared range, which has the advantage that most media have a low absorbance in this range (Olsson and Nielsen, 1997). Measurement errors are typically 5–10%, which is the same order of magnitude as the standard dry weight measurement (Andersen and Wagner, 1990).

Problems that need attention are the following. Air bubbles cause interference to the optical signal that may result in error. Interferences from air bubble and agitation can be reduced by passing the medium into an internal measurement chamber where the sample is degassed and by filtering of the bubble-induced spiky data (Hatch and Veilleux, 1995). Fouling of probe tips is a major problem with optical probes. Probes with holes or long "legs" are more prone to blocking than, for instance, backscatter sensors where light source and detector are at the same side of the sample (Andersen and Wagner, 1990). Equipping the sensors with a regularly performed "air check" allows automatic detection of a minute build-up of film on the optics, allowing us to ask for operator intervention (Watts *et al*., 1990). To decrease the fouling problem, different remedies are available. Location of the turbidity monitors in a highly turbulent region and sensors equipped with automatic cleaning devices, e.g. wipers or pistons, will decrease operator attendance time (Thomsen and Nielsen, 1992; Harremoës *et al*., 1993).

The biomass concentrations can also be determined from the difference between the velocity of ultrasonic sound in the suspension and in the microorganism-free solution (Blake-Coleman *et al*., 1986; Zips and Faust, 1989). However, the need for a microorganism-free reference requires either the withdrawal of the sample or *in situ* measurement of the background signal (Olsson and Nielsen, 1997). Owing to the vibrational nature of the device, it is, in principle, self-cleaning. Problems with this method include interference from air bubbles and temperature fluctuations.

Measurement of dielectric properties of biological cells has been used to determine viable biomass (Davey *et al*., 1993; Spierings, 1998; November and Van Impe, 2001). The application of an electric field to a suspension of cells in an aqueous solution results in movement of ions in the solution and within the cells, leading to charge separation or polarization across the cell membrane. The extent of this field-induced polarization can be

measured by the capacitance of the suspension. With the assumption that the physiological state of the microorganisms is unchanged, this capacitance measurement can be used to monitor biomass content (Davey *et al*., 1993). The effect of the medium conductance must be calibrated before the cell concentration can be determined. A major advantage of this technique is the possibility of measuring only the metabolically active biomass as opposed to total biomass. However, interferences from changes in the wastewater capacitance and changes in cell membrane structure, composition and permeability can affect the capacitance.

Anaerobic digestion

The anaerobic digestion process is characterized by the complete mineralization of organic material into gaseous products such as H_2, CH_4, CO_2 and H_2S. The production of this biogas occurs in a two-step process where methanogenesis depends on the intermediates produced in the preceding acidification stage. Both processes must be geared to one another to prevent accumulation of the (volatile fatty) acids produced in the first step. Imbalance will eventually lead to process failure due to the inhibitory effects of these products at high concentrations and the pH-drop they induce (Anderson and Yang, 1992b). Measurement of the intermediates and the final gaseous products is therefore of great interest in the control of anaerobic digesters.

Gaseous products (H_2, CH_4, CO_2 and H_2S)

Although a biogas flow measurement will give an indication of the overall activity of the reactor and has been used frequently as such, more specific techniques have been developed that allow us to monitor the gas composition. A typical lab-scale method consists of a combination of flow measurements in a set-up where one or more of the constituents are trapped in a washing bottle. The ratio of the flows before and after the bottle is representative of the gas composition. For instance, an alkaline washing bottle will trap all CO_2 and H_2S and lets CH_4 pass.

More specific gas analysers monitor the content of a component directly. Typically, an infrared absorption measurement is used to determine carbon dioxide and methane, while specific hydrogen analysers have been developed based on electrochemical cells (Mathiot *et al*., 1992; Pauss *et al*., 1993). Escoffier *et al*. (1992) trap H_2S before the entrance of the biogas into the hydrogen monitor. Hydrogen sulphide measurement in the gas phase may be performed by monitoring the reaction of sulphide with a Pb-strip. Subsequently, the black PbS that is produced is quantified by automatic colorimetry.

A major problem associated with any monitoring system based on gas analysis is that it is not straightforward to predict the corresponding concentrations in the liquid phase which, after all, represent the environment of the organisms. Pauss *et al*. (1993) mention that often the dissolved hydrogen concentration is calculated from the biogas composition under the assumption of equilibrium between gas and liquid phase (Henry's law):

$$[H_2]^* = K_H p_{H_2}$$

where $[H_2]^*$: dissolved hydrogen concentration in equilibrium with the gaseous partial pressure (mole/l)
p_{H_2}: gaseous H_2 partial pressure (Pa)
K_H: Henry's constant (mole/l.Pa)

To calculate the liquid concentration correctly, however, mass transfer dynamics should also be considered:

$$\frac{d[H_2]}{dt} = k_L a([H_2]^* - [H_2]) + r_{H_2}$$

where $k_L a$: mass transfer coefficient (/h)
$[H_2]$: dissolved hydrogen concentration
r_{H_2}: hydrogen production rate (mole/l.h)

This equation expresses that a discrepancy will exist between the actual dissolved hydrogen concentration $[H_2]$ and one calculated under the equilibrium assumption $[H_2]^*$. Moreover, this discrepancy will be more pronounced as the production rate increases and the mass transfer coefficient decreases. Pauss and Guiot (1993) give hydrogen mass transfer coefficients for digestors ranging between 0.04 and 0.4 h^{-1}. These low mass transfer efficiencies lead to supersaturation of the liquid ($[H_2]>[H_2]^*$). The authors observed that hundred-fold underestimations of the dissolved hydrogen concentration were obtained when calculated from biogas composition measurements. Therefore, gas composition measurements should only be used with great care, especially under dynamic conditions. Immersible sensors have been developed to measure the dissolved hydrogen concentration directly. Pauss *et al.* (1993) evaluated these sensors and reported their reliable use and long-term stability. Here too, fuel cells are the heart of the sensor.

The measurement of dissolved carbon dioxide is described in the next section. As far as is known to the authors, no direct on-line measurement of hydrogen sulphide or methane in the liquid phase has been reported.

Alkalinity/titrimeter

The incentive to measure the dissolved carbon dioxide and bicarbonate content of the mixed liquor originates from the fact that an imbalance of anaerobic digesters (due to accumulation of volatile fatty acids) cannot easily be detected on the basis of pH measurements, especially when the alkalinity of the mixed liquor is high (Rozzi, 1991; Hawkes *et al.*, 1992). Indeed, in such systems, alkalinity must be destroyed to a large extent before pH drops significantly. Since the alkalinity is mainly due to the bicarbonate buffer, it has been proposed since the early sixties that its measurement can be used in control strategies for anaerobic digesters (McCarty, 1964). However, only in the previous decade automated bicarbonate monitors have been developed and applied in practice (Di Pinto *et al.*, 1990; Hawkes *et al.*, 1993; Guwy *et al.*, 1994, Bouvier *et al.*, 2002).

Two basic principles have been used to assess bicarbonate alkalinity. First, titrimetry can be applied. It consists of titrating the sample down to a pH of 5.1 in a first step, followed by a further titration down to pH 3.5. This two-step titration allows to determine the bicarbonate content with a correction for the volatile fatty acids present (Anderson and Yang, 1992a; Kapp, 1992). However, interferences with other weak acid/base organic couples cannot be excluded. As an alternative, titration and back-titration methods have been proposed (Powell and Archer, 1989). Such set-ups are less prone to these interferences since the back-titration provides a CO_2-free blank. An advanced on-line titrimetric sensor was developed for the simultaneous monitoring of different compounds of interest, such as ammonia, bicarbonate and VFAs (Van Vooren *et al.*, 1996; Van Vooren *et al.*, 2001). In contrast to the titrimetric methods mentioned above, a titration is carried out over the whole pH-range (3–11). Next, the buffer capacity, i.e. the amount of acid/base needed per unit of pH change, is calculated (Figure 2). These data are then subjected to model-based interpretation, providing estimates of the concentrations of the different compounds of interest. The response time is approximately 30 minutes and sensitivity is in the ppm-range.

The second method for on-line alkalinity determination is based on quantifying the gaseous carbon dioxide evolving from the sample as it is acidified. The volume of gas may

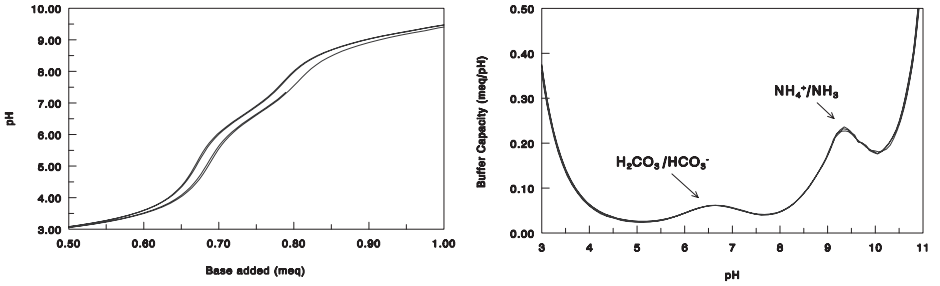


Figure 2 Titrimetric method for the determination of, among others, bicarbonate alkalinity in anaerobic digester samples. The right panel shows the buffer capacity obtained from the four repeat titration curves in the left panel (Van Vooren *et al.*, 2001)

be measured in two different ways. Di Pinto *et al.* (1990) measure the overpressure in a closed constant volume vessel, while Hawkes *et al.* (1993) measure the produced gas volume with a sensitive gas flow meter. The latter principle allows continuous measurement while the former method and the titration methods require intermittent sampling.

Calorimetry

All biological activity is characterized by the production of heat. Athermal or even endothermic growth processes are unthinkable as they would violate the second law of thermodynamics (Von Stockar and Marison, 1989). Measurement of heat production in so-called calorimeters therefore provides direct insight into the biological processes. Moreover, since heat dissipation is a universal feature, calorimetry can be applied to any bioprocess. Calorimeters have essentially followed the developments in temperature measurements. Different set-ups have been devised but it is beyond the scope of this paper to detail their design (Jolicoeur and Beaubien, 1986; Von Stockar and Marison, 1989; Vandenhove, 1998; Franco and Lema, 2001). For on-line monitoring of wastewater treatment processes preference should be given to flow calorimeters. Such devices are installed on a bypass fast loop and do not require special adaptations of the reactor the calorimeter is attached to. As an alternative, the heat balance of the reactor can be applied to calculate the biological heat production (Van Kleeff *et al.*, 1993). Here, all heat flows in and out of the reactor must be known, including for instance heat transfer through reactor walls, heat loss with the (gas and liquid) mass flows across the reactor boundaries, mechanical heat input and heat exchange with the temperature control system. Systematic overviews of such heat balances are given in Messenger *et al.* (1990) and Van Kleeff *et al.* (1993). The latter authors also focus attention on the problems inherent to this type of "whole reactor calorimetry": changes in heat transfer coefficients as a result of wall growth or changing hydrodynamic conditions and the precautions to be taken when the heat balance is used under dynamic conditions.

Fluorescence

A number of essential intermediates in biological reactions are characterized by fluorescence at particular wavelengths. A first group of fluorophores consists of the reduced forms of NAD(P)H.

These electron carriers are widespread among living organisms and since the early eighties attempts have been made to determine their level in microbial cultures (Sonnleitner *et al.*, 1992). Most applications have been found in aerobic systems (see below), but Peck and Chynoweth (1992) report on experiments in which NADH fluorescence allows us to detect digester instability due to substrate overload and inhibitory

compounds. Moreover, it was found that this variable among different variables tested (VFA, pH, biogas flow) gave the first indication of imbalance.

The second fluorescing intracellular compound is the electron carrier coenzyme F_{420} that is unique to methanogens. Although some discussion still remains whether F_{420} is a good indicator of methanogenic activity (Colleran *et al*., 1992), probes have been developed to allow its quantification (Peck and Chynoweth, 1992). At this stage further study is required for a proper interpretation of the F_{420} signal, eventually leading to a direct measurement of the physiological state of methanogens.

Fluorosensors are built around two optical fibres, one bringing the excitation light into the culture, the other carrying the fluorescence light to the detector. For NAD(P)H measurement excitation and fluorescence wavelengths are 351 and 460 nm respectively. For F_{420} monitoring the respective wavelengths are 406 and 465 nm (Peck and Chynoweth, 1992). Again, since these sensors are built as immersion probes, they will be subjected to fouling of the optical surfaces and probe design should take this into account (Peck and Chynoweth, 1992). Often these fluorosensors have been advertised as biomass sensors. It is, however, important to note that the fluorescence measured is indicative of the metabolic state of the culture and can only be used to determine biomass concentrations if the physiological state of the culture remains constant (Sonnleitner *et al*., 1992).

Volatile fatty acids (VFA)

Volatile fatty acids are the most important intermediates in the anaerobic digestion process. Moreover, since their accumulation may lead to process failure due to the pH-drop they induce (Anderson and Yang, 1992b), VFA concentrations have been monitored for a long time as process performance indicators. However, few on-line sensors have been implemented. The most advanced instrumentation consists of a gas chromatograph or high-pressure liquid chromatograph coupled to a sample preparation unit (Zumbusch *et al*., 1994; Banister and Pretorius, 1998) and a full-scale application for this method has been reported (Pind *et al*., 2002).

Steyer *et al*. (2002) use a Fourier Transform Infra-Red (FT-IR) spectrometer in the mid infrared range as one on-line multi-parameter sensor (i.e., one unique equipment to provide on-line measurements of COD, TOC, VFA, PA and TA) with an ultra-filtration membrane unit. FT-IR spectrometry is based on the fact that each compound has an unique absorbance pattern, in terms of band shape and band position in the infrared absorption spectrum. By comparing a sample spectrum with the reference spectra for compounds of interest, the composition of the sample can be identified using the Beer–Lambert law. An example of on-line implementation of this instrumentation technique is shown in Figure 3. Step changes of the influent flow rate at $t \approx 55$ and 75 h and change of the input concentration at $t \approx 100$ h were applied. On-line measurements were compared to those provided by a TOC analyser and titrimetric sensor installed on the same fixed-bed digester. As can be seen, the measurements are very close to those of more widely accepted sensors. The FT-IR spectrometer does not require any chemical to be added and needs only very low maintenance. It provides on-line multi-parameter measurements from one unique equipment. However, one of major drawback of FT-IR spectroscopy is that a big calibration effort is necessary for each component that is targeted for measurements.

More robust techniques are based on titrimetry. To eliminate the interference with the bicarbonate buffer, either two-step titration or titration and back titration have been proposed (Anderson and Yang, 1992a). Both methods provide information on both the bicarbonate and VFA content of the sample. The ratio of these variables gives an idea of the relative amount of buffer capacity which is still left to neutralize VFA and can be used to control anaerobic sludge digestion (Rozzi, 1991).

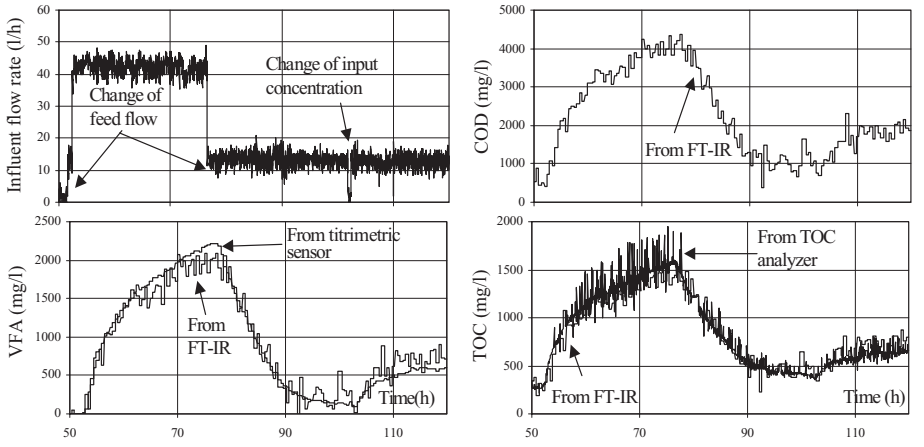


Figure 3 An example of on-line measurements after a step change in the influent flow rate (Steyer *et al.*, 2002)

Rozzi *et al.* (1997a) use a biosensor to determine VFA concentrations in digested anaerobic liquor. A sample of the digested effluent is mixed with denitrifying microorganisms in the presence of excess nitrate. The amount of VFA is indirectly measured by the acid equivalent which is needed to neutralize the denitrification reaction.

Biosensors

Closely related to the above VFA-analyser, the MAIA (Methanogenic Activity and Inhibition Analyzer) titration biosensor is a pH-stat device where the alkalinity produced by acetoclastic methanogens is neutralized by acid addition (Rozzi *et al.*, 2001). By measuring the titrant flow rate the activity of the acetoclastic methanogens is calculated. The main operating problems concern a rigorous experimental procedure and the determination of initial equilibrium conditions.

The RANTOX biosensor is designed to detect upcoming organic overloads and/or toxic loads based on the response of the acetoclastic methanogenic microorganisms to periodic pulses of acetate (Rozzi *et al.*, 1997b). The basic principles of the sensor's operation are similar to the RODTOX respirometric biosensor, already applied to aerobic wastewater treatment plants (Vanrolleghem *et al.*, 1994). Biomass subjected to repeated organic overloads provides repeated peaks of biogas production and allows us to detect the presence of inhibitors in the feed through the change in level and profiles of the biogas production peaks.

Activated sludge

Dissolved oxygen (DO)

The key role that oxygen plays in activated sludge processes and the associated aeration costs that can account for up to 40% of the running costs (Healey, 1989), means that sensors for this variable are probably the most widespread instruments in wastewater treatment plants. Oxygen measurement is based on the electrochemical reaction of oxygen diffusing from the liquid through a gas-permeable membrane in an amperometric or polarographic measuring cell (Lee and Tsao, 1979). The DO probes are considered reliable and accurate but care must be taken for proper location and fouling prevention (Stephenson *et al.*, 1981; Watts *et al.*, 1990; Harremoës *et al.*, 1993). Automated cleaning systems have become rather common. As an additional improvement, Watts *et al.* (1990) have proposed a sensor equipped with a cleaning system with automated air-check, allowing self-calibration.

While the DO electrode has been used extensively in control systems of the aeration, yielding important cost savings, the information obtained has also been used to monitor the central process of any activated sludge system. Holmberg *et al.* (1989) determine the oxygen uptake rates (OUR) in an aeration tank with a method that is based on the "dual-controller" concept (Aström and Hägglund, 1984). Essentially the DO control is modified such that an oscillation around a setpoint is enforced by proper excitation of the aeration system. Using an oxygen balance for each oscillation period allows us to estimate both the mass transfer characteristics and the biological oxygen uptake in the aeration tank.

Demuynck *et al.* (1994) illustrate the use of oxygen profiles from a sequencing batch reactor for determining OUR and nitrification control strategies. With on–off control of the aeration (setpoint = 2 mg/l, dead-band = 0.5 mg/l), a DO-profile can be obtained as in Figure 4. The frequency of the resulting DO oscillation is a good indicator of the oxygen uptake rate, i.e. high frequencies correspond with high oxygen uptake rates since then the aeration is switched on more frequently to supply the necessary oxygen. In addition, an actual value of the OUR can be obtained from the available data: during the periods in which the aeration is off, the oxygen uptake rate is equal to the slope of the DO-data. These OUR-values are also given in the figure. In the experiment depicted in Figure 4, one clearly observes that the on–off frequency significantly drops after 90 minutes. Further evidence showed that this was the time when nitrification had completed. This simple to obtain information can be used in nitrification control (Demuynck *et al.*, 1994).

Respirometry

Respirometry is the measurement and interpretation of the respiration rate of activated sludge, and is defined as the amount of oxygen per unit of volume and time that is consumed by the microorganisms in the activated sludge. It is a frequently used tool for the characterization of wastewater and activated sludge kinetics. Several respirometric principles were developed in the past, and one can classify them into a number of basic measurement principles depending on two criteria: 1) the phase where oxygen is measured (gas or liquid), and 2) the flow regime of both gas and liquid phase, which can be either flowing or static (Spanjers *et al.*, 1998).

Most measured variables do not need additional information to be interpreted but this is not the case for respirometry. A respiration rate value or a percentage inhibition deduced from respiration rate measurements cannot be interpreted without additional information about some measurement attributes. Indeed, a respirometer is a reactor in itself where different components are brought together to perform what may called an "In-Sensor Experiment" and in which the experimental conditions generally have a very large influence on the measurement results. The IWA Task Group (Spanjers *et al.*, 1998) has

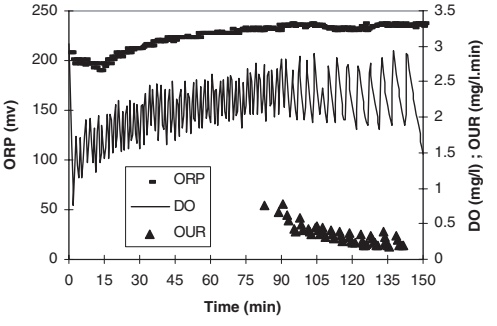
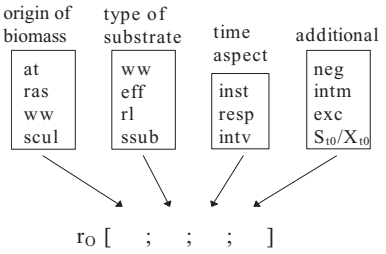


Figure 4 DO and deduced oxygen uptake rate data from a SBR for nitrogen removal. Aeration is regulated with an on–off controller (Demuynck *et al.*, 1994)



at	activated sludge tank	inst	instantaneous
at*k*	k^{th} compartment of plug flow tank	resp	respirogram
ras	return activated sludge	intv	interval
ww	wastewater	neg	negligible
scul	specific culture	intm	intermediate
eff	effluent	exc	excess substrate
rl	return liquor	S_{t0}/X_{t0}	initial substrate to biomass ratio
ssub	specific substrate		

Figure 5 Nomenclature of respiration rate (Spanjers *et al*., 1998)

found that at least three attributes must be specified to interpret respiration rate measurements: (1) biomass source, (2) type of substrate and (3) time aspect (Figure 5).

The result of a respiration rate measurement frequently is converted to a deduced variable that is more appropriate for interpreting results in a particular context. Many deduced variables have been proposed. In Table 1 a selection of deduced variables is presented, together with the respiration rate(s) from which the variable is deduced, the other measured variables used in the calculations, and the calculation methods have been proposed. No detailed explanation of the underlying mathematics will be given here, though.

Biological oxygen demand (BOD)

Traditionally, the biodegradable component of wastewater is measured by the standard, off-line, method of biochemical oxygen demand (BOD_5). The BOD_5 is a measure of the amount of dissolved oxygen required for the biochemical oxidation of the organic solutes in 5 days from the time when the test sample is seeded with a microbial system. However, the BOD_5 test is inadequate for automated monitoring and control because of the time required to complete the test and the difficulty in achieving consistently accurate measurements. Therefore, the on-line measurement of the load of the wastewater is based on the on-line short term BOD (BOD_{st}) estimation. Two types of on-line BOD_{st} methods are currently used: respirometric methods and microbial probes.

The respirometric method uses either batch or continuous respirometers in which the BOD_{st} is calculated from a respirogram resulting from the addition of a wastewater sample or through the oxygen mass balance over the respiration chamber. Reviews on the state of the art of BOD_{st} estimation were given by Spanjers *et al*. (1993). A respirometric sensor (RODTOX; a flowing gas-static liquid respirometer) is presented that is capable of monitoring the BOD_{st} and potential toxicity of wastewaters (Vanrolleghem *et al*., 1994; Kong *et al*., 1996). The RODTOX consists of a constantly aerated, completely mixed batch reactor containing 10 litre of sludge. From the respirograms obtained after injections of a pulse of calibration substrates and wastewater the RODTOX sensor calculates BOD_{st}. The sensor has an inherently large dynamic range for BOD_{st} measurements (0.01–500 g BOD_{st}/dm^3) and BOD_{st} values can be obtained every 30 minutes. As an illustration of the highly

Table 1 Selection of deduced variables obtained from respiration rate measurements (Spanjers *et al.*, 1998). For explanation of nomenclature, see Figure 5. The wildcard "*" means that different biomass sources, substrate types or time aspects are possible

Deduced variable	Respiration rates (and other measurements)	Method
R	$r_O[*,*,*]$ (and X)	arithmetic
BOD_{st}	$r_O[*,*,resp]$	integration
	$r_O[*,ww,inst]$	arithmetic
$\%I$	$r_O[*,ww,inst,exc]$	comparison
t.t.e.	$r_O[*,ww,resp]$	comparison
X	$r_O[*,-,inst]$	arithmetic
	$r_O[*,ssub,inst,exc]$	arithmetic
	$r_O[*,ssub,resp]$	parameter estimation
B_X	$r_O[at,ww,inst]$, $r_O[ras,-,inst]$ (and Q_{in}, Q_{ras})	arithmetic
$R_{NH,max}$	$r_O[at,*,resp]$, $r_O[at,ssub,resp]$	arithmetic
	$r_O[at,ssub,resp,exc]$	parameter estimation
ASM parameters	$r_O[*,ww,resp]$ (and X)	parameter estimation
ASM components	$r_O[*,ww,resp]$	parameter estimation
	$r_O[at,ww,inst,exc]$, $r_O[at,ww,inst]$, $r_O[at,-,intv]$	arithmetic

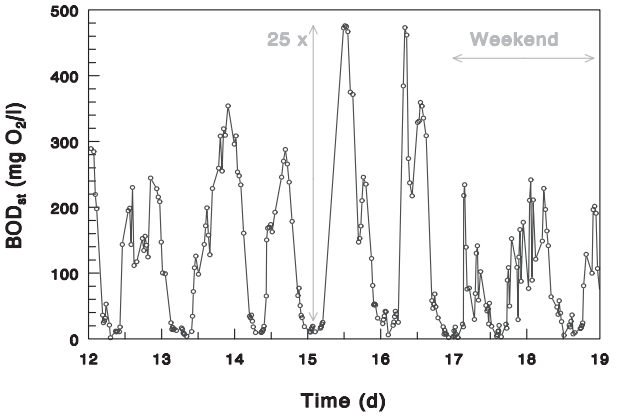


Figure 6 Typical weekly pattern of influent waste concentration measured with a RODTOX respirometer at a hospital wastewater treatment plant in Gent, Belgium

dynamic load variations that a plant is subject to, Figure 6 gives the BOD_{st} evolution at a hospital wastewater treatment plant in Gent (Belgium) as measured with such a RODTOX device.

The microbial probe consists of immobilized cells, a membrane and a dissolved oxygen electrode. The signal of the probe is a measure of the activity of the cells, which is, in turn, a measure of the substrate concentration in the wastewater. Microbial BOD sensing is best with a microbial system having no selectivity and high assimilability for a wide range of organic solutes or containing many different microorganisms such as in the activated sludge (Qian and Tan, 1998). Microbial BOD sensors with activated sludge have been developed (Karube *et al.*, 1977; Strand and Carlson, 1984; An *et al.*, 1998), but these sensors show poor response stability and reproducibility compared with single microorganism based biosensors (Riedel *et al.*, 1988; Preininger *et al.*, 1994). Qian and Tan (1998) develop a dead-cell BOD sensor using thermally killed cells for fabricating the biofilm attached to a dissolved oxygen probe. The dead cells can be prepared and stored dry at room temperature for prolonged periods prior to their use for the biofilm preparation. This is an important criterion for the bioactive material to become commercially acceptable for biosensor preparation (Rawson *et al.*, 1989).

Microbial BOD sensors require careful maintenance and storage to maintain their efficacy. Most microbial BOD sensors have a short lifetime, from a few days to a few months, and require low storage temperature of 4–10°C (Strand and Carlson, 1984; Riedel *et al*., 1988; Preininger *et al*., 1994). Because of the biochemical reactions involved in BOD sensing, their application is also limited by their specificity with respect to the substrate, pH, temperature and their susceptibility to poisoning and deactivation.

Chemical oxygen demand (COD)

One of the most intensively monitored variables in wastewater treatment plant is the chemical oxygen demand. Using standardized laboratory analyses, the performance of most plants with respect to their carbon removal efficiency is determined. A few attempts have been made to automate the two-step laboratory procedure which consists of: 1) two hour digestion in bichromate solution and 2) back-titration or colorimetric quantification of the residual oxidant. Two types of implementations have resulted, batch systems and flow-through continuous COD monitors.

In the different set-ups that have evolved, several modifications have been made to the original methods in view of their on-line use. Digestion is shortened (typically 0.5 h instead of 2 h) to improve sample throughput and response time (Korenaga *et al*., 1990, Meredith, 1990). More important changes concern the digestion method applied. Meredith (1990), for instance, proposes not to use bichromate as an oxidizing agent, but to use hydrogen peroxide coupled to UV light to produce ozone *in situ*. After oxidation the surplus oxygen is monitored by means of a DO probe, giving the COD-content of the sample.

One of the main limitations of the COD test is its inability to differentiate between biodegradable and biologically inert organic matter on its own (Bourgeois *et al*., 2001). The use of chemicals such as chromium and strong acid produce liquid hazardous waste which requires disposal. Experience with a number of these sensors in the authors' laboratory has shown that sensors using chemical oxidation are subject to important clogging problems due to the formation of crystals of the oxidation chemicals on the one hand, and the presence of particulate matter in the sample on the other hand. Clogging problems were also noticed by Korenaga *et al*. (1990) and Meredith (1990). Hence, it must be advised to apply only prefiltered samples.

Total organic carbon (TOC)

The central principle of a TOC measurement is to convert organic carbon to CO_2 and measure this product in the evolving gas phase, typically with an infrared off-gas analyser. Two principles exist for the conversion to carbon dioxide: in the first, a high temperature (650–800°C) catalytic conversion is imposed, while in the other case persulphate is added to the sample where UV light promotes the oxidation of organic matter at moderate temperatures. Both methods have drawbacks. For the former method salts can produce a melt on the catalytic surface inhibiting its proper operation. For the persulphate method, incomplete oxidation can occur when the pH of the sample is too low or when turbidity in the sample decreases penetration of the UV light (Van Vooren and Willems, 1993). Potassium persulphate crystals can plague operation and care must be taken to remove humidity from the gas stream prior to introduction in the gas analyser. For both types particulate matter is to be avoided because the retention time in the reaction chamber is insufficient for complete combustion. Moreover, clogging may be a problem. Prefiltration of the samples seems therefore essential for proper operation.

Some drawbacks inherent to the method are that the inorganic carbon present in the sample must be stripped first, potentially resulting in the loss of volatile organics. Also, one measures organic carbon (an important variable to characterise the load), but no

information is obtained with respect to the oxidation state of the carbon, which is important for the oxygen demand it represents. In addition, no data on the biodegradability of the wastewater is given, similarly to the COD measurement (Londong, 1992). However, if the composition of the wastewater is rather constant, one can calibrate for these effects. Since the method is fast (5–10 min response time) and reliable if low-salt and particulate-free samples are provided, TOC can be advocated as a good monitoring parameter, especially for effluent quality where other methods are insufficiently sensitive.

UV-absorbance

Many wastewater components absorb UV light. Already in the early fifties measurement of UV absorbance was introduced to assess the quality of an effluent (Dobbs *et al*., 1972). Optical methods have the advantage of being rather inexpensive and not to require reagents or preparation of the sample. Moreover, in the last decade fibre optic technology has made significant progress enabling remote and multi-point measurement (MacCraith *et al*., 1993).

Different studies have illustrated the high correlation between UV absorption and organic matter in wastewater. Dobbs *et al*. (1972) show clear relationships between absorption at 254 nm and the total organic carbon present in effluents and surface waters. They state, however, that turbidity should not exceed a certain limit if the correlation is to be useful and propose the use of filtration units. Matsché and Stumwöhrer (1996) show the good correlation of UV absorption at 260 or 254 nm with COD or TOC. They can reduce the influence of particulate material with the help of the measurement at a second wavelength (e.g. 380 nm). Brookman (1997) investigates the absorbance at 280 nm for farm slurry effluents and found an exponential relationship between absorbance and BOD_5. But this method shows poor sensitivity for values of less than 100 mg/l due to interference of UV absorbance by the presence of particles and toxic metals. Thomas *et al*. (1997) demonstrate the potential of UV spectral measurements with a deconvolution method for the estimation and on-line monitoring of specific parameters such as TOC, COD and BOD. However, the major drawback of UV spectrometry lies in the fouling of optical cell components resulting in a loss of sensitivity and need for frequent recalibration. In addition, not all compounds (e.g. carbohydrates, saturated hydrocarbons) absorb at the specified wavelength and thus cannot be considered with UV spectrophotometry (Bourgeois *et al*., 2001).

Nutrient removal

Nutrient removal systems aim at eliminating nitrogen and phosphorus from the wastewater, either in a biological, a chemical or a combined treatment. Chemical phosphorus removal is based on the addition of precipitants, while biological P-removal is obtained by selecting for a microbial community that stores excess phosphorus. Selection is based on subjecting the sludge to a sequence of anaerobic and aerobic (or anoxic) conditions. Mainstream biological nitrogen removal processes are two-step processes consisting of nitrification of reduced nitrogen under aerobic conditions and denitrification of nitrate to dinitrogen gas in the absence of oxygen, i.e. anoxic conditions.

The complexity of these processes and the interactions between the different unit processes have resulted in rather complex plant configurations with rather high monitoring and control demands. Sensor technology for this application has evolved remarkably in recent years and is largely based on the automation of lab analysis.

Pre-treatment

Although recent developments have been important, most of the commercially available measuring systems still require pre-treated samples to ensure satisfactory operation. Figure

7 gives a schematic diagram of the ultrafiltration units that are frequently used for this purpose. A submerged pump transfers sludge through the membrane filter (typical pore size of 20 μm) and a filtered sample is then led to the in-line sensor.

Ultrafiltration (UF) membranes with a cut-off molecular weight of 20,000 are typically applied. The lifetime of a cross-flow filter is approximately 1 year (Thomsen and Nielsen, 1992) but regular cleaning is necessary. To maintain sample flow to the analysers a set-up with two UF units is preferred. Using built-in filtrate flow meters or pressure drop measurements, automated switching of the active unit and activation of the cleaning program has been implemented. Typically the membranes need to be cleaned every 1 to 4 weeks. Cleaning strategies consist of an air blow or a hypochlorite chemical treatment. Sample preparation has proven to be reliable, but still requires maintenance efforts by the operators (Harremoës *et al*., 1993).

Lynggaard-Jensen *et al*. (1996) developed a semi-micro continuous flow analysis (μCFA) system based on membrane technology. A carrier (clean water) is pumped to the membrane device and flows in a track on one side of the membrane. The other side of the membrane is exposed to the wastewater, and the carrier is enriched with ions passing through the membrane. The enriched carrier is sent to the analysis manifold. The principle has been applied to sensors for ammonium, nitrate and phosphorus respectively. The sensors are all based on the colorimetric method and calibration is carried out automatically. These sensors have been tested for wastewater treatment plant applications and are commercially available on the market (Ingildsen, 2002). One drawback associated with this type of meter is that it is impossible to multiplex the sensor, i.e. couple more than one measuring point to one measuring device. UF units on the contrary allow us to use one meter for several parallel UF units connected to different sampling points.

Automated wet chemistry methods

Since the advent of reliable sample preparation units, a lot of effort has been devoted to the automation of typical laboratory methods for on-line use in wastewater treatment plants. Three implementations exist: batch-wise chemical analysis, continuous flow-through systems based on the flow injection analysis (FIA) principle and sequential injection analysis (SIA). FIA (Ruzicka and Hanssen, 1975) is the most popular on-line measurement option. An interesting feature of FIA is that the analytical reactions need not reach equilibrium to

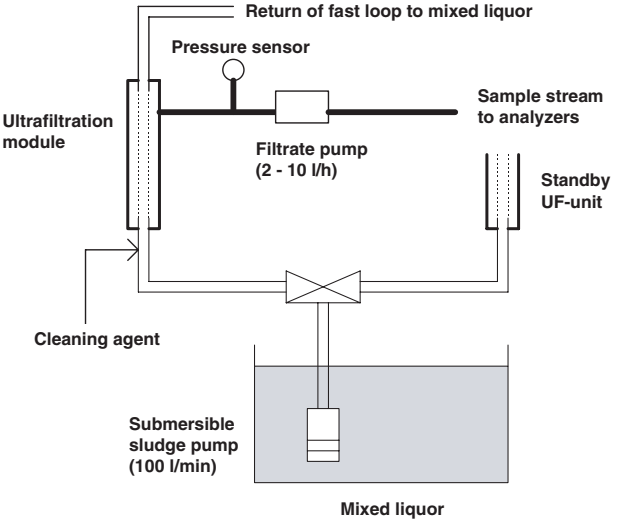


Figure 7 Diagram of a typical ultrafiltration module

obtain reproducible measurements, as the dilution of the sample and the reaction time between injection and detection is reproducible at constant carrier flow rates. However, the selection of a suitable pump (piston, peristaltic, etc.) is important (Van der Pol *et al*., 1996; Vaidyanathan *et al*., 1999).

SIA is an improvement of FIA (Christensen *et al*., 1996; Thomas *et al*., 1997). The main feature of this system is the substitution of the FIA manifold with a single multi-position valve that can be operated with a single pump, which is typically a high-precision piston pump. SIA increases the flexibility of measurement and reduces mechanical complexity at the cost of increased analytical time (Vaidyanathan *et al*., 1999).

In all cases chemicals are added to the sample and after some reaction time the coloured products are quantified colorimetrically. Advantages of the FIA and SIA systems over the batch systems are the small sample size, low reagent use (Pedersen *et al*., 1990; Thomas *et al*., 1997) and high sample throughput (Isaacs *et al*., 1992).

NH_4^+. For colorimetric NH_4^+ determinations, all NH_4^+ is converted into NH_3 before it is transformed into a blue (phenate method) or a yellow (nesslerization method) azo dye. Pedersen *et al*. (1990) use a gas diffusion unit through which ammonia is transported in a weak pH-buffer with a pH-indicator. The ammonia gas causes a pH-increase that is accompanied with a colour change. Thomsen and Nielsen (1992) describe the more traditional method that consists of producing an indophenol blue compound, the intensity of which is proportional to the amount of NH_4^+ present in the sample. However, the high maintenance need (e.g. the gas diffusion membrane has to be replaced once every week, need for a well-trained technician) was a serious drawback for on-line application. Continuous technological improvements have in the mean time resulted in gas diffusion membranes with a lifetime of about 6 months. Colorimetric NH_4^+ analysers have a higher reagent consumption (Thomsen and Nielsen, 1992) and are sensitive to sample temperature variations (Wacheux *et al*., 1996). It is generally accepted that the colorimetric method is less reliable compared to the ISE method (see below, Harremoës *et al*., 1993). Moreover, response times for batch chemical analysis are rather long compared to electrodes.

Wacheux *et al*. (1996) performed a field test to operate five analysers or monitors (three based on ISE and two based on colorimetry) in real conditions, to observe their behaviours and to compare the results with traditional sample analysis. All sensors were installed in parallel at the discharge point of a wastewater treatment plant. Figure 8 shows the daily evolution of NH_4^+ as seen by the various monitors. An offset difference between monitors

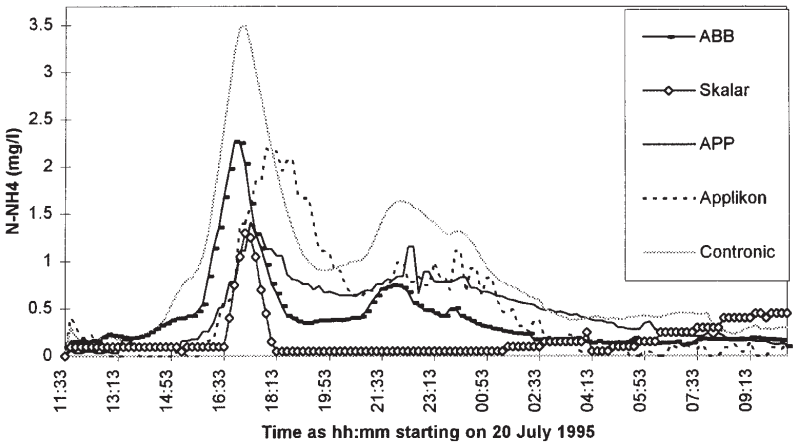


Figure 8 Field test of NH_4^+ monitors on 20 July 1995 (Wacheux *et al*., 1996)

and laboratory analysis was observed. The field test showed that every detail is important when installing a monitor, particularly the filtration unit, the constant level in the reagent reservoirs and the plastic piping which can be the origin of important maintenance efforts.

NO_3^-. Quantifying nitrate concentrations is based on its reduction to nitrite on a Cd-column and the determination of total nitrite (Pedersen *et al*., 1990). Using a separate nitrite-only determination allows us to calculate the nitrate concentration originally present in the sample by subtraction. Harremoës *et al*. (1993) report that complete nitrate reduction often fails, limiting the applicability of this method. A separate colorimetric on-line NO_2^- measurement can be carried out by just bypassing the Cd reduction step from the colorimetric NO_3^- determination method (Pedersen *et al*., 1990; Teichgräber, 1993).

PO_4^{3-}. Two colorimetric methods have been implemented in automated orthophosphate analysers. In the vandomolybdophosphoric acid method, vanadium is used in combination with ammonium molybdate to produce a yellow colour with the phosphate ion. The measuring range is 0.1 to 20 mg/l. In the ascorbic acid method, a molybdenum blue is formed using ascorbic acid. The measuring range is 0.01 to 5 mg/l. These sensors have proven to be accurate (Thomsen and Kisbye, 1996), but operating costs are high due to significant chemical use (Barnard and Crowther, 1993).

Redox

ORP electrodes provide a general indication of the oxidative status of the monitored system. ORP electrodes, in contrast to DO electrodes, also provide information about the biological processes occurring under anoxic and anaerobic conditions. From a technical point of view, the ORP measurement can be considered accurate and unproblematic (Harremoës *et al*., 1993). However, it must be stressed that one should not control processes on the basis of absolute ORP values. The absolute ORP values fluctuate with the actual load and interference with other redox buffers and slow changes on the electrode surface affect the absolute values (Heduit *et al*., 1993; Heduit and Thevenot, 1992).

Interpretation of ORP measurements is based on the detection of breakpoints or "knees" in ORP profiles. The knees indicate the appearance or disappearance of a redox buffer system, comparable to a pH buffer system in acid/base titrations. The most well known ORP breakpoints are the DO breakpoint and the NO_3^- breakpoint (Wareham *et al*., 1993; Demuynck *et al*., 1994). The DO breakpoint indicates the disappearance of NH_4^+ during the aerobic phase (end of nitrification). The NO_3^- breakpoint indicates the disappearance of NO_3^- during the anoxic cycle (end of denitrification). Figure 9 illustrates the occurrence of

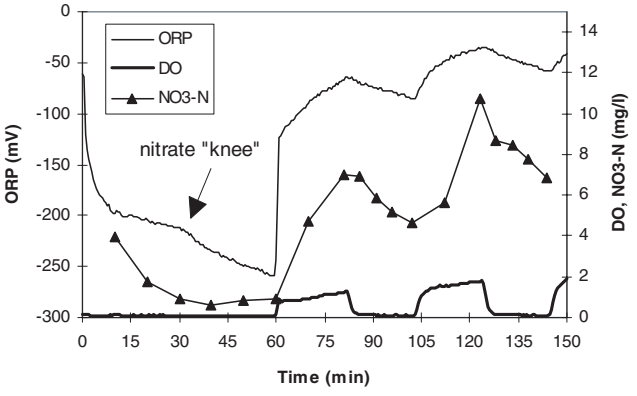


Figure 9 ORP profile with "nitrate knee" (Demuynck *et al*., 1994)

a nitrate "knee" in the ORP profile as nitrate disappears from the mixed liquor. The nitrate knee occurs after 35 minutes in the anoxic phase, indicating that all NO_3^- is denitrified, as evidenced also by the NO_3^- measurements presented in Figure 9. Model-based methods to reliably detect such knees from the raw data were evaluated in Vanrolleghem and Coen (1995). Since it is better to monitor changes in the ORP-profile (slope of the ORP curve), it is essential to impose changing conditions to the process such as in sequencing batch reactors (Wareham *et al*., 1993; Demuynck *et al*., 1994), intermittent aeration systems (Sasaki *et al*., 1993; Wasiak *et al*., 1993) or alternating activated sludge plants (Isaacs *et al*., 1993).

Ion-selective electrodes

A number of ion-selective electrodes (ISE) use an electrochemical reaction to monitor the concentration of specific compounds such as Cl^-, Na^+, F^-, CN^-, S^{2-} and, more importantly, NH_4^+ and NO_3^-.

NO_3^-. The nitrate ISE is advantageous for its low chemical consumption and little or no sample pre-treatment, resulting in a short response time (Thomsen and Nielsen, 1992; Barnard and Crowther, 1993). However, the system is sensitive to contamination of the electrode (Wacheux *et al*., 1993; Sikow and Pursiainen, 1995), electrode drift (Wacheux *et al*., 1993) and interference of ions such as HCO_3^- (Sikow and Pursiainen, 1995) and especially Cl^- (APHA, 1992). However, the electrode drift phenomenon of the nitrate probe can be overcome by implementing automatic *in situ* calibration method (Sin *et al*., 2003; Petersen *et al*., 2002b).

NH_4^+. ISEs are the preferred measuring principle for NH_4^+ (Thomsen and Nielsen, 1992; Harremoës *et al*., 1993). The method consists of increasing the sample pH to 11, converting all NH_4^+ into NH_3 which is subsequently quantified by the gas-sensitive electrode. The limited operating problems concern clogging (Aspegren *et al*., 1993), electrode drift (Patry and Takács, 1995), hydroxide poisoning of the electrode (Aspegren *et al*., 1993) and gas bubble retention under the electrode tip (Andersen and Wagner, 1990). The importance of a thermostated measuring cell has also been reported (Thomsen and Nielsen, 1992; Teichgräber, 1993). Response times are typically 15 minutes, including sample pre-treatment (Thomsen and Nielsen, 1992).

UV absorption for NO_3^- determination

The UV absorption of nitrate at 210 nm can be applied for its quantification. The UV absorption NO_3^- analysers are advantageous for their low maintenance need (Thomsen and Nielsen, 1992; Sikow and Pursiainen, 1995) and short response time of about 10 seconds (Wacheux *et al*., 1993). The UV technique is suitable for wastewaters low in organic matter. However, a lot of organic substances also present in wastewater absorb in the UV region, and despite a lot of effort to compensate for this, UV absorption measurements suffer substantially from such interference. Frequent zero calibration is necessary to prevent baseline drift; automatic cleaning and autocalibration have been incorporated in commercial systems (Andersen and Wagner, 1990; Aspegren *et al*., 1993).

Titrimetric sensor

In so-called titrimetric sensors, the stoichiometric conversion of NH_4^+ to produce $2H^+$ ($NH_4^+ + 2O_2 \rightarrow NO_3^- + H_2O + 2H^+$) is used to obtain information about the nitrification process (Beccari *et al*., 1980; Ramadori *et al*., 1980, Aivasidis *et al*., 1992). A good correlation is obtained between the amount of ammonium added to activated sludge and the amount of ammonium measured with the titrimetric sensor, where the latter was obtained

by applying the stoichiometric conversion factor to the measured amount of protons produced during nitrification of the ammonium (Massone *et al.*, 1995). A typical data-set obtained in a titrimetric sensor is depicted in Figure 10. Nitrification starts immediately after addition of the sample and continues for 25 minutes. The initial high base dosing rate is needed to quickly reach the pH setpoint. Interpreting the cumulative base addition curves can be done using a simple slope extrapolation method, assuming that nitrifying 14 mg N will produce 2 meq protons. The NH_4^+-N concentration S_{NH} (mg N/l) and the nitrification rate r (mg N/l·h) are readily calculated according to the following equations:

$$S_{NH} = \frac{2}{14} \cdot (B2 - B1)$$

$$r = \frac{2}{14} \cdot (S1 - S2) \cdot 60$$

where the intercepts $B1$ and $B2$ are expressed in meq/l units, while the slopes $S1$ and $S2$ are expressed in meq/l·min units.

This measuring principle has been developed and applied to the on-line measurement of the nitrification rate in activated sludge (Gernaey *et al.*, 1997), the on-line measurement of the ammonium concentration (Gernaey *et al.*, 1997; Massone *et al.*, 1998) and the detection of toxic effects of wastewater and chemical compounds (Gernaey *et al.*, 1999b; Rozzi *et al.*, 1999). Yuan and Bogaert (2001) applied a titrimetric respirometer to measure nitrifiable nitrogen concentrations.

Compared to existing chemical on-line NH_4^+ analysers, titrimetric sensors need no sample preparation step. Moreover, the titration procedure does not use expensive and environmentally unfriendly chemicals (e.g. EDTA) as most of the on-line NH_4^+ analysers do. A disadvantage of the titrimetric sensor is its variable response time, which is dependent on both the NH_4^+ concentration in the sludge sample and the nitrification rate of the sludge. The readily biodegradable COD present in the wastewater also causes interference to the pH signal induced by CO_2 production from COD oxidation and leads to the assimilation of significant amounts of ammonia into new biomass (Yuan and Bogaert, 2001; Gernaey *et al.*, 2002).

Respirometry

Nitrification processes are characterized by a high oxygen consumption and it is therefore evident that respirometry has been adapted to monitor these processes. The main problem to be solved before nitrification activities can be determined is to separate oxygen

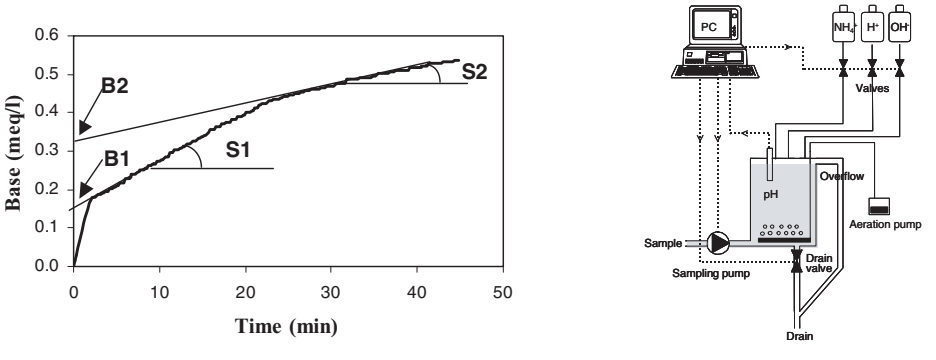


Figure 10 Typical cumulative base addition curve (left) obtained in a titrimetric sensor (right) with activated sludge in endogenous state to which NH_4^+-N (1.3 mg N/l) was added at $t = 0$ before the pH of the sludge was increased to its setpoint (Gernaey *et al.*, 1997)

consumption for carbon oxidation and endogenous metabolism from the oxygen uptake for nitrification. Different methods are applied in practice to obtain this separation.

Batch experiments for the determination of the nitrifying capacity of a sludge are normally performed using an endogenously respiring sludge sample. During the experiment a limited amount of NH_4^+ is injected. The respirogram obtained in this way allows us to estimate kinetic nitrification parameters (Brouwer and Klapwijk, 1995; Spanjers and Vanrolleghem, 1995). A procedure for the simultaneous characterization of carbon oxidation and nitrification was also proposed using an appropriate mixture of a readily biodegradable carbon source and NH_4^+ (Vanrolleghem and Verstraete, 1993). Kong *et al.* (1996) applied this procedure for the simultaneous estimation of the effect of toxic compounds on carbon oxidation and nitrification.

The NITROX (NITRification tOXicity tester) is developed as a respirometric on-line toxicity detection system combining a high sensitivity with a short response time (Gernaey *et al.*, 1997). The measuring principle is that of a closed batch respirometer. It is based on the measurement of the activity of the nitrifiers (OUR_n) as the difference between the total activity (OUR_{tot}) of a mixed culture (nitrifiers and heterotrophs) and the activity of the heterotrophs after inhibiting the nitrification (OUR_{atu}) by the addition of a selective inhibitor, allylthiourea (ATU) (Kroiss *et al.*, 1992; Surmacz-Gorska *et al.*, 1996). The presence of the toxicity will be determined by a decrease of $OUR_n = OUR_{tot} - OUR_{atu}$. The toxicity of a sample is determined by comparing the nitrifying activity obtained from a wastewater sample with unknown toxicity ($OUR_{n,sample}$) with that obtained from a reference cycle ($OUR_{n,ref}$):

$$\%inhibition = \frac{OUR_{n,ref} - OUR_{n,sample}}{OUR_{n,ref}} \times 100$$

Tap water is used as non-toxic reference solution. A reference cycle is normally performed once every two or three hours.

Nitrification is a two-step reaction. Surmacz-Gorska *et al.* (1996) take the above mentioned approach a step further and present a more sophisticated respirometer that allows us to separately monitor the activity of both nitrification steps. The method also provides information about the oxygen uptake for carbon oxidation or endogenous metabolism of the biomass. The measurement is based on the sequential addition of $NaClO_3$ and ATU, specific inhibitors for the NO_2^- and NH_4^+ oxidizing bacteria respectively, to an activated sludge sample. The separate activities can be calculated by subtracting the different OUR values, as illustrated in Figure 11. Dosing both $NaClO_3$ and ATU gives extra value to respirometric methods. When applied to a nitrifying activated sludge system, the method should for instance allow the detection of the presence of NO_2^--N in the mixed liquor. This data could be used to optimize the activated sludge process in such a way that NO_2^--N is converted completely to NO_3^--N, or inversely, to perform nitrogen removal via the cost-effective NO_2^--N route (Hellinga *et al.*, 1998).

Respirometric applications for nitrogen removal processes are not limited to the estimation of nitrification rates only. Model-based interpretation of respirograms, recorded after adding a wastewater sample to activated sludge in the endogenous state, is used to determine the concentration of nitrifiable nitrogen present in the influent of a wastewater treatment plant (Brouwer and Klapwijk, 1995; Spanjers and Vanrolleghem, 1995). Initial substrate concentrations can be calculated by interpreting the area under the respirogram.

A combined respirometric-titrimetric set-up was recently applied to monitor the degradation processes during batch experiments with activated sludge (Gernaey *et al.*, 2001). The respirometer consists of an open aerated vessel and a closed non-aerated respiration

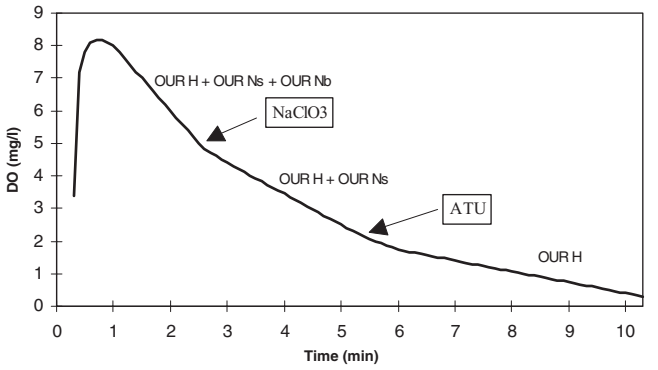
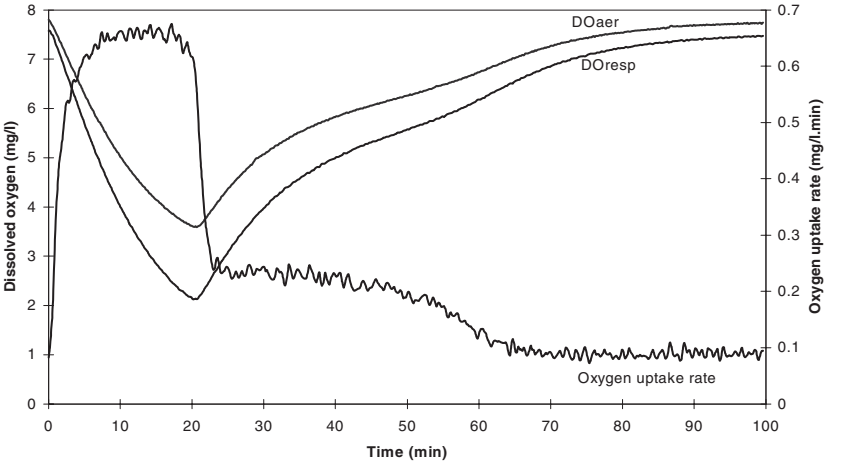
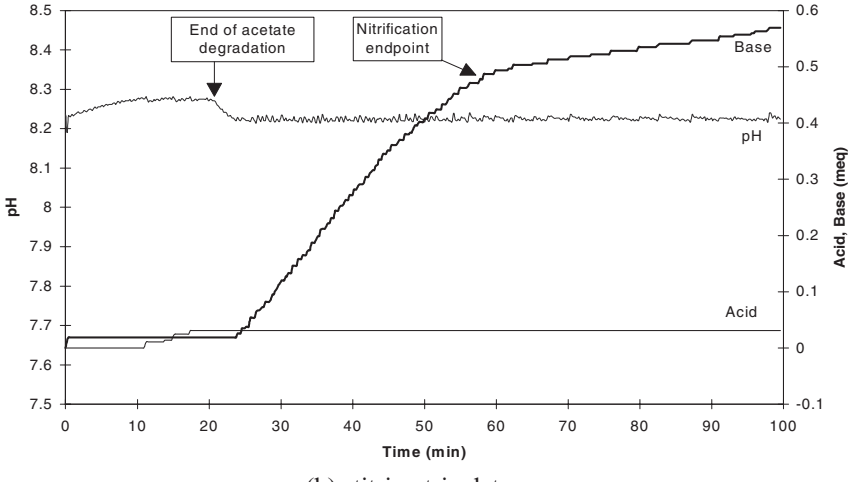


Figure 11 Illustration of a respirometric experiment including the addition of 2 selective nitrification inhibitors to sludge sample (Surmacz-Gorska *et al*., 1996). In a first phase, the total OUR is measured. After addition of $NaClO_3$, nitrification step 2 (OUR Nb) is inhibited. Addition of ATU inhibits nitrification step 1 (OUR Ns). The remaining OUR H is the heterotrophic and endogenous OUR



(a) respirometric data



(b) titrimetric data

Figure 12 Example of combined respirometric-titrimetric data obtained after addition of acetate (30 mg COD/l) and ammonium (2 mg N/l) at t = 0 (Gernaey *et al*., 1999a)

chamber. It is operated with two oxygen probes resulting in two sources of information on the oxygen uptake rate; both collected at a high frequency. The respirometer is combined with a titrimetric unit that maintains the pH. The amount of added acid and base serves as a complementary information source on the degradation processes. An example of combined respirometric-titrimetric data collected after addition of a mixture of acetate and NH_4^+-N to activated sludge is given in Figure 12. It is clear from the two oxygen and OUR curves (Figure 12(a)) that the substrate degradation phase can be divided into two parts. First, both acetate and NH_4^+-N are oxidized simultaneously, and second only degradation of NH_4^+-N continues. From the titration data (Figure 12(b)) it can indeed be seen that the pH effects of both processes (acetate degradation and nitrification) almost compensate for each other, and only a small amount of acid has to be added. However, as soon as acetate is degraded, base is added to compensate for the protons produced during the nitrification process. Finally, it can be seen that there is a clear bend in the base curve at the nitrification end-point. In summary, such titrimetric curves can provide important information on the different processes and, combined with respirometry, it becomes a powerful and very information-rich method for characterizing biological wastewater treatment processes.

Recently, Sin *et al*. (2003) developed an integrated sensor to monitor nitrification, denitrification and aerobic carbon source degradation processes in one single set-up. The sensor provides information-rich data of high frequency (every 3 seconds) obtained from respirometric-titrimetric and ISE nitrate measurements.

Fluorescence

Under aerobic conditions the oxygen uptake rate is a very good indicator of sludge activity, but assessment of the metabolic state of the cell under the anoxic and anaerobic conditions in nutrient removal plants exclude application of this reliable measurement. Under such conditions monitoring NADH fluorescence can take over.

The NAD(P)H fluorescence signal is a measure of the intracellular redox state and is of value in determining the metabolic status of the microorganisms. For a given pool of coenzyme NAD(P), the NAD(P)H fluorescence signal reflects the ratio between NAD(P)H and NAD(P). Since a highly reduced state is necessary in the anaerobic zone to provide sufficient reducing power to convert VFAs into PHB (Barnard and Crowther, 1993; Armiger *et al*., 1993), a relatively greater proportion of coenzyme is present as NAD(P)H under anaerobic conditions, compared to either aerobic or anoxic zone. With this concept in mind, Isaacs and Henze (1994) use the NADH fluorescence to detect the end of the denitrification in an alternating activated sludge plant. Isaacs *et al*. (1998) evaluated such *in situ* measurement technique for the estimation of key parameters affecting denitrification in activated sludge processes. The method is based on either fluorescence or redox potential measurements taken in an isolated mini-reactor, the Biological Activity Meter (BAM), situated in the anoxic zone of the plant. Armiger *et al*. (1993) provide evidence suggesting a linear relationship between the VFA loading of a nutrient removal plant and the fluorescence signal in the anaerobic zone. Since VFAs are central in the P-removal process, the authors conclude that this signal is superior to COD or BOD measurements in view of the control of the recycle rates in the nutrient removal plant studied. Moreover, the fluorescence signal is immediate while the other methods have a significant response time.

NOx biosensor

Larsen *et al*. (1997) developed a micro-scale nitrate/nitrite biosensor based on bacterial nitrate/nitrite reduction to nitrous oxide and a subsequent detection of the nitrous oxide produced. The bacteria-containing outer casing of the biosensor is a tapered glass tube (Figure 13). The tip opening is covered by an approximately 10 μm thick ion-permeable

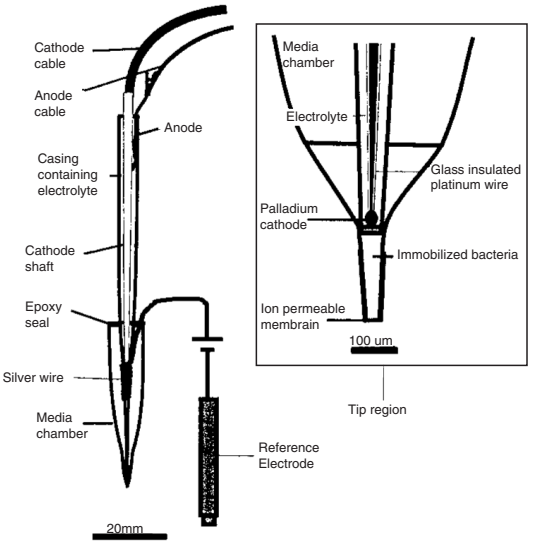


Figure 13 A nitrate/nitrite biosensor. Left: entire sensor. Right: enlarged section of the tip region (Larsen *et al.*, 2000)

membrane. When nitrate/nitrite passes through the membrane it enters a dense mass of denitrifying bacteria that are deficient in nitrous oxide reductase and thus reduce nitrate/nitrite to nitrous oxide. The nitrate oxide produced is quantified by a built-in nitrous oxide microsensor (Revsbech *et al.*, 1988). The response time of the biosensor is between 20 and 50 seconds. The only interfering substances are nitrous oxide, which is normally present in negligible concentrations. The detachment of the ion-permeable membrane in the tip of the biosensor limits the period with continuous monitoring to about five days.

Sedimentation

In view of the fact that a clarifier is the final step in all wastewater treatment plants, it is clear that any failure of this sludge separator has tremendous effects on the effluent quality. It is therefore surprising that relatively little attention is paid to instrumentation of this important unit process. Probably this is due to the lack of fundamental insights in the determining factors, e.g. bulking sludge. Some developments have however been made and some of the resulting sensors are reviewed.

Sludge blanket

Three measuring principles for the localisation of the sludge blanket have been used in practice: ultrasonic absorption and turbidity devices (see above) detect the suspended solids interface as a result of the sudden change in sludge concentration as one penetrates into the sludge blanket. The third method, ultrasonic scanning provides a concentration profile and is regarded as the best measuring principle. Moreover, the measurement is insensitive to sludge clouds above the blanket (Harremoës *et al.*, 1993). The turbidity sensors on a rotating drum are probably the most applied.

The turbidity probe is lowered precisely until the sludge blanket is reached and the distance travelled gives the sludge blanket depth. These measuring systems give reliable results provided proper maintenance of the drum mechanism and sufficient cleaning precautions are taken (Aspegren *et al.*, 1993). One should note that this equipment can also be used to determine a concentration profile as a function of the clarifier depth. As an illustration Figure 14 shows the results of a detailed one-month measuring campaign on a

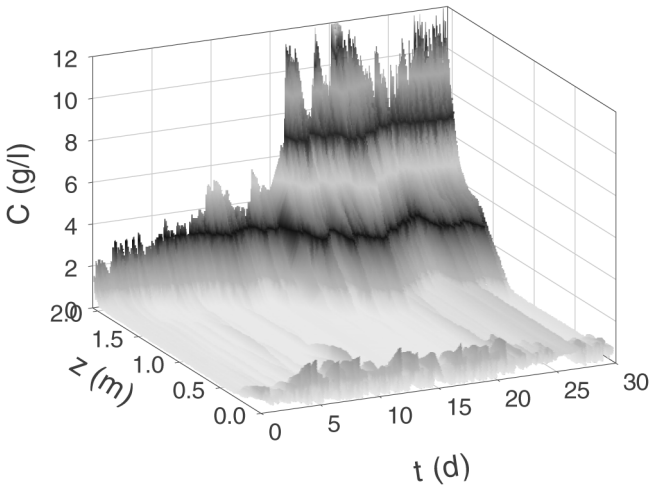


Figure 14 Measured sludge profile versus time (De Clercq *et al.*, 2002b)

full-scale wastewater treatment plant, providing hourly measurements of concentration profiles in the settler (De Clercq *et al.*, 2002b).

A different set-up consists of three turbidimeters located at fixed but different depths in the clarifier. The presence or absence of the sludge blanket at these positions can then be assessed. This is a more reliable set-up since mechanical problems with the rotating drum are eliminated. A control strategy is possible that maintains the sludge blanket around the level indicated by the probe situated at the middle position. The signals of the two other turbidity meters can be used for alarm triggering.

Settlometer

The sludge settling characteristics are often quantified by the sludge volume index (SVI). This parameter is obtained by dividing the volume of settled sludge after 30 minutes of settling by the suspended solids concentration. However, it was proven that the SVI is strongly influenced by the sludge concentration (Dick and Vesilind, 1969). To minimize concentration and wall effects (Sekine *et al.*, 1989), modifications of the procedure have been proposed, such as gentle stirring of the sludge so as to better simulate clarifier conditions (White, 1976). The resulting values are reported as stirred SVI (SSVI). Another modification proposed by Lee *et al.* (1983) is to dilute the sludge to a certain sludge concentration, leading to the diluted SVI value (DSVI). All methods mentioned are labour intensive and their measuring frequency is too low (typically once per day) to track the time-varying sludge settleability.

Recent technological progress has resulted in the development of sensors to measure sludge settling characteristics. The main feature of the sensors is often a central glass cylinder bringing a mixed liquor sample in a batch sedimentation experiment under conditions that approach those in the secondary clarifier. The descent of the sludge blanket interface in batch settling experiments is tracked using light transmission, measured either via a fixed array of light emitting diodes (LED) on one side and photodiodes on the other (Reid and Nason, 1993; Fuchs and Staudinger, 1997) or via a moving LED photodiode couple (Sekine *et al.*, 1989). Following the latter approach, Vanrolleghem *et al.* (1996) introduced a sensor with a stirrer mechanism in the settling cylinder. In this Settlometer the evolution of the blanket height is recorded with a moving optical detection system. From the resulting sludge sedimentation curve, the maximum sedimentation velocity and the sludge volume index (SVI, SSVI or DSVI) can be readily obtained. In addition, a sludge concentration

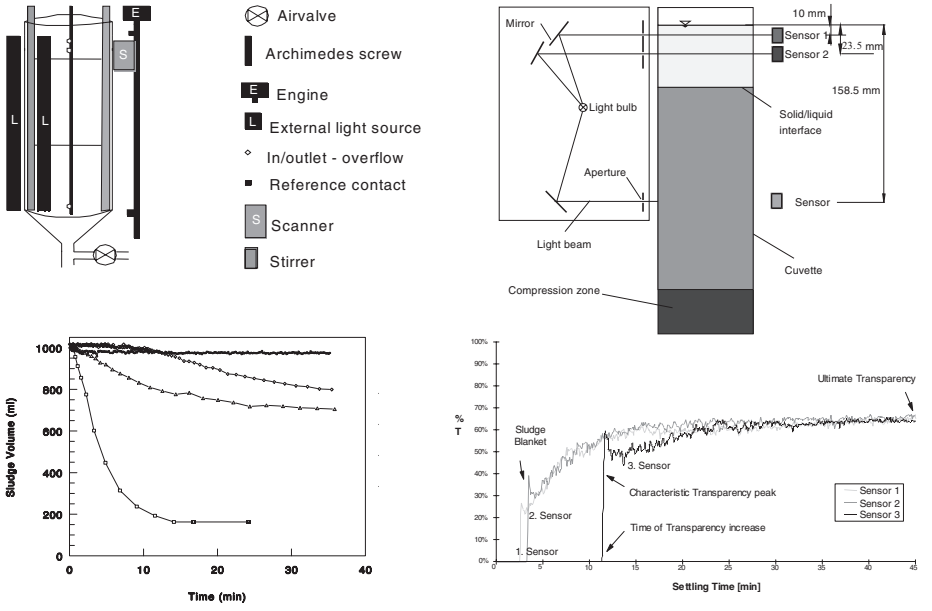


Figure 15 Schematic diagram and data of the Settlometer (Vanrolleghem *et al.*, 1996) illustrating differences in settling (left) and of the Schlumosed (Fuchs and Staudinger, 1997) illustrating the transparency at 3 different locations during a settling experiment (right)

measurement can be installed in the sensor and an automatic dilution system has been tested as well.

The left panel of Figure 15 shows experimental batch settling curves obtained in this Settlometer with sludges with different settling characteristics. The right panel gives the measuring principle of the Schlumosed developed by Fuchs and Staudinger (1997) and shows illustrative data collected from its 3 diodes located at 3 different positions along the settling column. Noteworthy is the slow increase in transparency indicative of continued flocculation and settling and the highly transparent peak just after passage of the blanket at the diode.

An alternative approach is the continuous settling column developed by Rasmussen and Larsen (1997) to measure the dynamic variations of settling velocity of activated sludge. The method uses a mass balance to determine the settling velocity based on two concentration measurements with a light transmission system.

Image analysis

The increasing capabilities and decreasing investment costs of image analysis systems has led to a surge of development in the field of processing microscopic images (Adams and Thomas, 1988). Interpretation of such images is especially interesting with respect to a better insight into the settling properties of activated sludge. Filamentous bulking is one of the main problems in wastewater treatment today. Monitoring the filamentous character of sludge flocs can contribute to a better understanding of the processes that led to this phenomenon. In the end the goal is to develop strategies for the control of the filamentous growth (Vanrolleghem and Van Impe, 1992). Image analysis of activated sludge, at high magnifications, was used by Watanabe *et al.* (1990) to determine the filamentous state of the sludge. Li and Ganczarczyk (1990) examined the internal structure of activated sludge flocs using image analysis. Grijspeerdt and Verstraete (1997) have developed an on-line setup using low magnification microscopy combined with image analysis to estimate

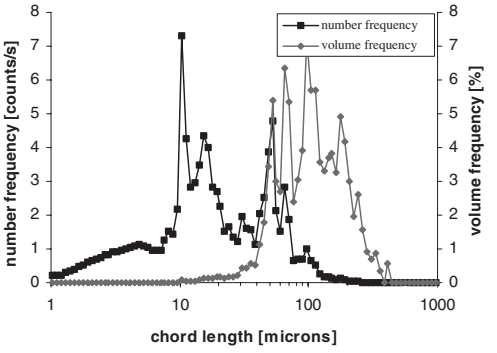


Figure 16 Particle size distribution measured in a secondary clarifier in Oxley Creek (Australia) using a Lasentec FBRM probe (De Clercq *et al.*, 2002a)

settling properties of activated sludge. The morphology of the activated sludge flocs such as the mean equivalent circle diameter and the mean form factor of the sludge flocs is measured and this can be related to traditional settling indices. In addition, a rapid and reliable estimate of the suspended solids concentrations can be obtained. A method to estimate the amount of filamentous bacteria as well as the size and fractal dimension of flocs was developed by da Motta *et al.* (2001). From the captured binary images flocs and filamentous bacteria are discriminated on the basis of their size and gyration diameter. However, a compromising magnification to visualize both the filaments and the flocs must be carefully determined.

Floc size

Floc size and size distribution measurements open opportunities for detecting changes in the floc properties during various treatment steps and provide valuable information about how well the separation processes work. Various methods can be found in literature to measure the size of the activated sludge flocs (Li and Ganczarczyk, 1991; Andreadakis, 1993; Barbusinski and Koscielniak, 1995). A laser light scattering technique has recently been used to obtain on-line floc size and size distribution (Biggs and Lant, 2000; De Clercq *et al.*, 2002a; Govoreanu *et al.*, 2002). Floc sizing is measured using a Malvern Mastersizer (Malvern, UK) sizing instrument that works on the basis of Fraunhofer diffraction theory (Biggs and Lant, 2000; Govoreanu *et al.*, 2002). Diluted samples of activated sludge are introduced into the path of a He-Ne laser. Scattered light is then detected and related to the particle size distribution. Guan *et al.* (1998) utilize the scattering data from the Malvern Mastersizer to monitor the fractal dimension of activated sludge flocs from full-scale wastewater treatment plants. With an alternative method (Lasentec FBRM M500, Lasentec, USA), De Clercq *et al.* (2002a) could measure the sludge particle size distribution *in situ* in a secondary clarifier. The probe is based on the focused beam reflectance method. As particles pass in front of the probe, the laser beam intersects the edge of the particle and backscatters the laser light. The backscatter continues until the focused beam reaches the particle's opposite edge. The backscatters are collected by the optical detector and used to determine a particle size distribution as the chord lengths of the particles. Figure 16 gives typical experimental results, both in number frequencies (i.e. numbers per particle size class) and volume frequencies (i.e. volume of particles per particle size class).

Conclusions

A review of existing and new sensor technology was presented. Developments are many and increasingly sophisticated sensors are proposed in an attempt to provide the necessary

Table 2 State of the art of on-line monitoring equipment for wastewater treatment processes

Physical measurements			Physico-chemical measurements			(Bio-) chemical measurements		
Variable	**Process**	**Range**	**Variable**	**Process**	**Range**	**Variable**	**Process**	**Range**
Temperature	G	∀	pH	G	∀	Respirometry	2, 3	∀
Pressure	G	∀	Conductivity	G	∀	Toxicity	2, 3	∀
Liquid level	G	∀	Oxygen concentration	2, 3	∀	BOD_{st}	2, 3	∀
Flow rates	G	∀	Fluorescence	2, 3	∃	COD	1, 2, 3	O
Suspended solids	G	∃	Redox	1, 3	∀	TOC	1, 2, 3	∀
Sludge blanket	4	∃	NH_4^+ (ISE)	3	∀	NH_4^+	3	∀
Sludge volume	4	∃	NO_3^- (ISE)	3	∃	NO_3^-	3	∀
Settling velocity	4	O	Digester gas	1	∃	Micro-scale NOx	3	∀
Sludge morphology	G	O	(CH_4, H_2S, H_2) CO_2	1, 2, 3	∀	PO_4^{3-}	3	∃
Calorimetry	1, 2, 3	O				Bicarbonate alkalinity	1, 3	∃
UV absorption	G	∃				VFA	1, 3	O

Process: Unit process in wastewater treatment plants where the sensor can be implemented 1: Anaerobic Digestion; 2: Activated Sludge; 3: Nutrient Removal; 4: Sedimentation; G: All processes. Applicability Range: ∀: State of the Technology; ∃: Application in certain cases; O: Requires development work

information on the complex processes required to meet effluent standards. A clear divide is growing between sensors applicable for routine monitoring and use in automatic control systems and advanced on-line monitoring equipment typically used by consultants involved in problem solving (auditing) or model-based optimisation of treatment processes.

In Table 2 the state of the art of sensor technology as presented in this paper is summarized. The sensors are classified into (i) state-of-the-art technology that is found on many plants, (ii) measuring systems that have found their way for particular applications at certain treatment plants whereas on other plants they are not considered useful and (iii) devices that are operational, but are typically requiring further development or need specialized personnel to make proper use of their signals (e.g. by consultants). Further work in academia and industry will lead to further improvements in 1) the reliability of the developed instruments and 2) the applicability of the information they provide in automatic monitoring and control systems for wastewater treatment processes.

Acknowledgements

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References

Adams, H.L. and Thomas, C.R. (1988). The use of image analysis for morphological measurements on filamentous organisms. *Biotech. Bioeng.*, **32**, 707–712.

Aivasidis, A., Hochscherf, H., Rottman, G., Hagen, T., Mertens, M.T., Reiners, G. and Wandrey, C. (1992) Neuere Konzepte zur Prozessüberwachung und -regelung bei der biologischen Stickstoffelimination. *Abwassertechnik*, **5**, 48 –55. (In German).

An, L., Niu, H. and Zeng, H. (1998). A new biosensor for rapid oxygen demand measurement. *Water Environ. Res.*, **70**, 1070–1074.

Andersen, K.L. and Wagner, P. (1990). Testing on-line monitors for ammonia, orthophosphate and suspended solids in activated sludge treatment plants. In: *Instrumentation, Control and Automation of Water and Wastewater Treatment and Transport Systems (Advances in Water Pollution Control 10)*. Ed. Briggs R., Pergamon Press, London, 667–675.

Anderson, G.K. and Yang, G. (1992a). Determination of bicarbonate and total volatile acid concentration in anaerobic digesters using a simple titration. *Water Environ. Res.*, **64**, 53–59.

Anderson, G.K. and Yang, G. (1992b). pH control in anaerobic treatment of industrial wastewater. *J. Environ. Eng.*, **118**, 551–567.

Andreadakis, A.D. (1993). Physical and chemical properties of activated sludge floc. *Wat. Res.*, **27**, 1707–1714.

APHA (1992). *Standard Methods for the Examination of Water and Wastewater*, 18th ed. Washington DC.

Armiger, W.B., Lee, G.J., Schwegler, B.R. Jr. and Mah, T.J. (1993). Control of BNR processes by regulation of the VFA/M ratio. *Wat. Sci. Tech.*, **28**(11–12), 303–314.

Aspegren, H., Nyberg, U. and Andersson, B. (1993). Integration of on-line instruments in the practical operation of the Klagshamn wastewater treatment plant. *Med. Fac. Landbouww. Univ. Gent*, **58**, 2019–2028.

Aström, K.J. and Hägglund, T. (1984). Automatic tuning of simple regulators with specifications on phase and amplitude margins. *Automatica*, **20**, 645–651.

Barbusinski, K. and Koscielniak, H. (1995). Influence of substrate loading intensity on floc size in activated sludge process. *Wat. Res.*, **29**, 1703–1710.

Banister, S.S. and Pretorius, W.A. (1998). Optimisation of primary sludge acidogenic fermentation for biological nutrient removal. *Water SA*, **24**, 35–41.

Barnard, J.L. and Crowther, R. (1993). Instrumentation in biological nutrient removal plants. In: *Preprints of 6th IAWQ Workshop on ICA of Water and Wastewater Treatment and Transport Systems*. June 17–19, Banff, Canada.

Beccari, M., Passino, R., Ramadori, R. and Tandoi, V. (1980). Inhibitory effects on nitrification by typical compounds in coke plant wastewaters. *Env. Tech. Lett.*, **1**, 245–252.

Biggs, D.A. and Lant, P.A. (2000). Activated sludge flocculation: on-line determination of floc size and the effect of shear. *Wat. Res.*, **34**, 2542–2550.

Blake-Coleman, B.C., Clarke, D.J., Calder, M.R. and Moody, S.C. (1986). Determination of reactor biomass by acoustic-resonance densitometry. *Biotech. Bioeng.*, **28**, 1241–1249.

Bourgeois, W., Burgess, J.E. and Stuetz, R.M. (2001). On-line monitoring of wastewater quality: a review. *J. Chem. Tech. Biotech.*, **76**, 337–348.

Bouvier, J.C., Steyer, J.P. and Delgenes, J.P. (2002). On-line titrimetric sensor for the control of VFA and/or alkalinity in anaerobic digestion processes treating industrial vinasses. In: *Proceedings VII Latin American Workshop and Symposium on Anaerobic Digestion*, Merida, Mexico, October 23–25 2002 (in Press).

Brookman, S.K.E. (1997). Estimation of biochemical oxygen demand in slurry and effluent using ultra-violet spectrophotometry. *Wat. Res.*, **31**, 372–374.

Brouwer, H. and Klapwijk, A. (1995). A method based on respirometry to calculate the aerobic nitrification volume of a nitrifying/denitrifying activated sludge plant. In: *Proceedings Symposium "Erfahrungen mit Analysen und prozessmessgeräten in Abwasserreinigungs-anlagen."* November 27, Ostfildern, Germany.

Christensen, L.H., Marcher, J., Schulze, U., Carlsen, M., Min, R.W., Nielsen, J. and Villadsen, J. (1996). Semi-on-line analysis for fast and precise monitoring of bioreaction processes. *Biotech. Bioeng.*, **52**, 237–247.

Colleran, E., Concannon, F., Golden, T., Geoghegan, F., Crumlish, B., Killilea, E., Henry, M. and Coates, J. (1992). Use of methanogenic activity tests to characterize anaerobic biodegradability and determine toxicity thresholds against individual anaerobic trophic groups and species. *Wat. Sci. Tech.*, **25**(7), 31–40.

da Motta, M., Pons, M.N. and Roche, N. (2001). Automated monitoring of activated sludge in a pilot plant using image analysis. *Wat. Sci. Tech.*, **43**(7), 91–96.

Davey, C.L., Davey, H.M., Kell, D.B. and Todd, R.W. (1993). Introduction to the dielectric estimation of cellular biomass in real-time, with special emphasis on measurements at high-volume fractions. *Anal. Chim. Acta.*, **279**, 155–161.

De Clercq, B., Brannock, M., Lant, P.A. and Vanrolleghem, P.A. (2002a). *In situ* particle size characterization on a circular clarifier of a wastewater treatment plant. In: *Preprints of International IWA Conference on Automation in Water Quality Monitoring, AutMoNet 2002*. May 21–22, Vienna, Austria.

De Clercq, J., Devisscher, M., Boonen, I., Vanrolleghem, P.A. and Defrancq, J. (2002b). A new one-dimensional clarifier model verification using full-scale experimental data. In: *Proceedings 3rd IWA World Water Congress*. April 7–12, Melbourne, Australia.

Demuynck, C., Vanrolleghem, P.A., Mingneau, C., Liessens, J. and Verstraete, W. (1994). NDBEPR process optimization in SBRs: Reduction of external carbon source and oxygen supply. *Wat. Sci. Tech.*, **30**(4), 169–179.

Di Pinto, A.C., Limoni, N., Passino, R., Rozzi, A. and Tomei, M.C. (1990). Anaerobic process control by automated bicarbonate monitoring. In: *Instrumentation, Control and Automation of Water and Wastewater Treatment and Transport Systems (Advances in Water Pollution Control 10)*. Ed. Briggs, R., Pergamon Press, London. 51–58.

Dick, R.I. and Vesilind, P.A. (1969). The sludge volume index-what is it? *J. Water. Pollut. Control Fed.*, **41**, 1285–1291.

Dobbs, R.A., Wise, R.H. and Dean, R.B. (1972). The use of ultra-violet absorbance for monitoring the total organic carbon of water and wastewater. *Wat. Res.*, **6**, 1173–1180.

Escoffier, Y., Ehlinger, F., Coudert, J.P., Leyris, J.P. and Moletta, R. (1992). On-line monitoring and control of a fluidised bed anaerobic digester. In: *Proceedings 6th Forum Applied Biotechnology. Med. Fac. Landbouww. Univ. Gent*, **57**, 1607–1614.

Franco, E.M. and Lema, J.M. (2001). Calorimetry in investigating biological anaerobic digestion: hydrolytic and acidogenic processes. In: *Preprints of 9th World Conference on Anaerobic Digestion, AD 2001*. September 2–6, Antwerpen, Belgium, 311–314.

Fuchs, A. and Staudinger, G. (1997). Sedimentation and clarification properties as characteristics of biological sludges. In: *Proceedings of 11th Forum Applied Biotechnology, Med. Fac. Landbouww. Univ. Gent*, **62**(4b), 1625–1632.

Gernaey, K., Bogaert, H., Massone, A., Vanrolleghem, P.A. and Verstraete, W. (1997). On-line nitrification monitoring in activated sludge with a titrimetric sensor. *Environ. Sci. Tech.*, **31**. 2350–2355.

Gernaey, K., Petersen, B., Ottoy, J.-P. and Vanrolleghem, P.A. (1999a). Biosensing activated sludge. *WQI* (May–June), 16–21.

Gernaey, K., Maffei, D., Vanrolleghem, P.A. and Verstraete, W. (1999b). A new pH-based procedure to model toxic effects on nitrifiers in activated sludge. *J. Chem. Tech. Biotech.*, **74**, 679–687.

Gernaey, K., Petersen, B., Nopens, I., Comeau, Y. and Vanrolleghem, P.A. (2002). Modelling aerobic carbon source degradation processes using titrimetric and combined respirometric- titrimetric data: I. Experimental data and model structure. *Biotech. Bioeng.*, (in press).

Gernaey, K., Petersen, B., Ottoy, J.–P. and Vanrolleghem, P.A. (2001). Activated sludge monitoring with combined respirometric-titrimetric measurement. *Wat. Res.*, **35**, 1280–1294.

Govoreanu, R., Seghers, D., Nopens, I., De Clercq, B., Saveyn, H., Capalozza, C., Van der Meeren, P., Verstraete, W., Top, E. and Vanrolleghem, P.A. (2002). Linking floc structure and settling properties to activated sludge population dynamics in an SBR. In: *Proceedings 3rd IWA World Water Congress*. April 7–12, Melbourne, Australia.

Grijspeerdt, K. and Verstraete, W. (1997). Image analysis to estimate the settleability and concentration of activated sludge. *Wat. Res.*, **31**, 1126–1134.

Guan, J., Waite, T.D. and Amal, R. (1998). Rapid structure characterization of bacterial aggregates, *Environ. Sci. Tech.*, **32**, 3735–3742.

Guwy, A.J., Hawkes, D.L., Hawkes, F.R. and Rozzi, A.G. (1994). Characterization of a prototype industrial on-line analyzer for bicarbonate/carbonate monitoring. *Biotech. Bioeng.*, **44**, 1325–1330.

Harremoës, P., Capodaglio, A.G., Hellström, B.G., Henze, M., Jensen, K.N., Lynggaard-Jensen, A., Otterpohl, R. and Soeberg, H. (1993). Wastewater treatment plants under transient loading – performance, modelling and control. *Wat. Sci. Tech.*, **27**(12), 71–115.

Hatch, R.T. and Veilleux, B.G. (1995). Monitoring of Saccharomyces cerevisiae in commercial baker's yeast fermentation. *Biotech. Bioeng.*, **46**, 371–374.

Hawkes, F.R., Guwy, A.J., Rozzi, A.G. and Hawkes, D.L. (1993). A new instrument for on-line measurement of bicarbonate alkalinity. *Wat. Res.*, **27**, 167–170.

Hawkes, F.R., Rozzi, A., Black, K., Guwy, A. and Hawkes, D.L. (1992). The stability of anaerobic digesters operating on a food-processing wastewater. *Wat. Sci. Tech.*, **25**(7), 73–82.

Healey, M.J. (1989). Improvements in the activated sludge process in the U.K. and U.S. *J. Water Pollut. Control Fed.*, **61**, 447–451.

Heduit, A., Martin, B., Duchamp, I. and Thevenot, D.R. (1993). Comparison of gold and platinum electrode responses in activated sludge. *Wat. Sci. Tech.*, **28**(11–12), 473–480.

Heduit, A. and Thevenot, D.P. (1992). Elements in the interpretation of platinum electrode potentials in biological treatment. *Wat. Sci. Tech.*, **26**(5–6), 1335–1344.

Hellinga, C., Schellen, A.A.J.C., Mulder, J.W., van Loosdrecht, M.C.M. and Heijnen, J.J. (1998). The Sharon process: an innovative method for nitrogen removal from ammonium-rich wastewater. *Wat. Sci. Tech.*, **37**(9), 135–142.

Holmberg, U., Olsson, G. and Andersson, B. (1989). Simultaneous DO control and respiration estimation. *Wat. Sci. Tech.*, **21**(10/11), 1185–1195.

Ingildsen, P. (2002). Realising full-scale control in wastewater treatment systems using *in situ* nutrient sensors. PhD thesis, Dept. of Industrial Electrical Engineering and Automation, Lund University, Sweden.

Isaacs, S.H. and Henze, M. (1994). Fluorescence monitoring of an alternating activated sludge process. *Wat. Sci. Tech.*, **30**(4), 229–238.

Isaacs, S.H., Henze, M., Soeberg, H. and Kümmel, M. (1993). Effect of supplemental carbon source on phosphate removal in an alternating activated sludge process. *Wat. Sci. Tech.*, **28**(11–12), 499–512.

Isaacs, S.H., Mah, T. and Maneshin, S.K. (1998). Automatic monitoring of denitrification rates and capacities in activated sludge processes using fluorescence or redox potential. *Wat. Sci. Tech.*, **37**(12), 121–129.

Isaacs, S.H., Zhao, H., Soeberg, H. and Kümmel, M. (1992). On the monitoring and control of a biological nutrient removal process. In: *Proceedings Workshop Modelling, Monitoring and Control of the Activated Sludge Process. Med. Fac. Landbouww. Rijksuniv. Gent*, **57**, 2199–2208.

Jeppsson, U., Alex, J., Pons, M.N., Spanjers, H. and Vanrolleghem, P.A. (2002). Status and future trends of ICA in wastewater treatment – A European perspective. *Wat. Sci. Tech.*, **45**(4–5), 485–494.

Jolicoeur, C. and Beaubien, A. (1986). Microcalorimetric studies of microbial metabolism and inhibition: bases for in vitro toxicity evaluation. In: *Toxicity Testing using Microorganisms*, Vol I. Eds. Dutka B. and Bitton G., CRC Press, Florida. 115–151.

Jones, A., Young, D., Taylor, J., Kell, D.B. and Rowland, J.J. (1998). Quantification of microbial productivity via multi-angle light scattering and supervised learning. *Biotech. Bioeng.*, **59**, 131–143.

Kapp, H. (1992). On-line Messung der organischen Saüren. *Korrespondenz Abwasser*, **39**, 999–1004.

Karube, I., Matsunaga, T., Mitsuda, S. and Suzuki, S. (1977). Microbial electrode BOD sensors. *Biotech. Bioeng.*, **19**, 1535–1547.

Kennedy, M.J., Thakur, M.S., Wang, D.I.C. and Stephanopoulos, G.N. (1992). Estimating cell concentration in the presence of suspended solids: A light scatter technique. *Biotech. Bioeng.*, **40**, 875–888.

Kong, Z., Vaerewijck, M. and Verstraete, W. (1996). On-line stBOD measurement and toxicity control of wastewaters with a respirographic biosensor. *Environ. Tech.*, **17**, 399–406.

Korenaga, T., Takahashi, T., Moriwake, T. and Sanuki, S. (1990). Water quality monitoring system using a flow-through sensing device. In: *Instrumentation, Control and Automation of Water and Wastewater Treatment and Transport Systems (Advances in Water Pollution Control 10)*. Ed. Briggs R., Pergamon Press, London. 625–631.

Kroiss, H., Schweighofer, P., Frey, W. and Matsche, N. (1992). Nitrification inhibition – a source identification method for combined municipal and/or industrial wastewater treatment plants. *Wat. Sci. Tech.*, **26**(5–6), 1135–1146.

Larsen, L.H., Damgaard, L.R., Kjær, T., Stenstrøm, T., Lynggaard-Jensen, A. and Revsbech, N.P. (2000). Fast responding biosensor for on-line determination of nitrate/nitrite in activated sludge. *Wat. Res.*, **34**, 2463–2468.

Larsen, L.H., Kjær, T. and Revsbech, N.P. (1997). A microscale NO_3^- biosensor for environmental applications. *Anal. Chem.*, **69**, 3527–3531.

Lauer, L., Hou, R. and Welcz, S. (1993). Process parameters for the improvement of the phosphate elimination in sewage treatment plants. In: *Proceedings TI-KVIV Studiedag Optimalisatie van Waterzuiveringsinstallaties door Proceskontrole en -sturing*. October 8, Gent, Belgium.

Lee, S.E., Koopman, B., Bode, H. and Jenkins, D. (1983). Evaluation of alternative sludge settleability indices. *Wat. Res.*, **17**, 1424–1426.

Lee, Y.H. and Tsao, G.T. (1979). Dissolved oxygen electrodes. *Adv. Biochem. Eng.*, **13**, 35–86.

Li, D.-H. and Ganczarczyk, J. (1990). Structure of activated sludge flocs. *Biotech. Bioeng.*, **35**, 57–65.

Li, D.-H. and Ganczarczyk, J. (1991). Size distribution of activated sludge flocs. *J. Water Pollut. Control Fed.*, **63**, 806–814.

Londong, J. (1992). Strategies for optimized nitrate reduction with primary denitrification. *Wat. Sci. Tech.*, **26**(5–6), 1087–1096.

Lynggaard-Jensen, A., Eisum, N.H., Rasmussen, I., Svankjær Jocobsen, H. and Stenstrøm, T. (1996). Description and test of a new generation of nutrient sensors. *Wat. Sci. Tech.*, **33**(1), 25–35.

MacCraith, B., Grattan, K.T.V., Connolly, D., Briggs, R., Boyle, W.J.O. and Avis, M. (1993). Cross comparison of techniques for the monitoring of total organic carbon (TOC) in water resources and supplies. *Wat. Sci. Tech.*, **28**(11–12), 457–463.

Massone, A., Antonelli, M. and Rozzi, A. (1996). The denicon: a novel biosensor to control denitrification in biological wastewater treatment plants. *Med. Fac. Landbouww. Univ. Gent*, **60**, 2361–2368.

Massone, A., Gernaey, K., Rozzi, A. and Verstraete, W. (1998). Measurement of ammonium concentration and nitrification rate by a new titrometric biosensor. *Water Environ. Res.*, **70**, 343–350.

Massone, A., Gernaey, K., Rozzi, A., Willems, P. and Verstraete, W. (1995). Ammonium concentration measurements using a titrimetric biosensor. *Med. Fac. Landbouww. Univ. Gent*, **60**, 2361–2368.

Mathiot, S., Escoffier, Y., Ehlinger, F., Couderc, J.P., Leyris, J.P. and Moletta, R. (1992). Control parameter variations in an anaerobic fluidised bed reactor subjected to organic shockloads. *Wat. Sci. Tech.*, **25**(7), 93–101.

Matsché, N. and Stumwöhrer, K. (1996). UV absorption as control-parameter for biological treatment plants. *Wat. Sci. Tech.*, **33**(12), 211–218.

McCarty, P.L. (1964). Anaerobic waste treatment fundamentals. Part two. Environmental requirements and control. *Publ. Wks*, **95**, 123–126.

Meredith, W.D. (1990). Recent innovations in instrumentation for sewage treatment plant monitoring and control. *Instrumentation, Control and Automation of Water and Wastewater Treatment and Transport Systems (Advances in Water Pollution Control 10)*. Ed. Briggs R., Pergamon Press, London. 699–703.

Messenger, J.R., De Villiers, H.A. and Ekama, G.A. (1990). Oxygen utilization rate as a control parameter for the aerobic stage in dual digestion. *Wat. Sci. Tech.*, **22**(12), 217–227.

November, E.J. and Van Impe, J.F. (2001). On-line viable biomass measurement and estimation of the specific growth rate of activated sludge from municipal wastewater treatment. *Wat. Sci. Tech.*, **43**(7), 97–102.

Olsson, L. and Nielsen, J. (1997). On-line and *in situ* monitoring of biomass in submerged cultivations. *TIBTECH*, **15**, 517–522.

Patry, G.G. and Takács, I. (1995). Modelling, simulation and control of large-scale wastewater treatment plants: an integrated approach. *Med. Fac. Landbouww. Univ. Gent*, **60**, 2335–2343.

Pauss, A. and Guiot, S.R. (1993). Hydrogen monitoring in anaerobic sludge bed reactors at various hydraulic regimes and loading rates. *Water Environ. Res.*, **65**, 276–280.

Pauss, A., Guiot, S.R., Dochain, D. and Perrier, M. (1993). Hydrogen as a key-parameter for the monitoring and control of methane producing processes. In: *Proceedings FAB '93 Workshop on Modelling, Monitoring and Control of Wastewater Treatment Processes*. September 30, Gent, Belgium.

Peck, M.W. and Chynoweth, D.P. (1992). On-line fluorescence-monitoring of the methanogenic fermentation. *Biotech. Bioeng.*, **39**, 1151–1160.

Pedersen, K.M., Kümmel, M. and Soeberg, H. (1990). A real time measurement system for an activated sludge wastewater treatment plant. *Instrumentation, Control and Automation of Water and Wastewater Treatment and Transport Systems (Advances in Water Pollution Control 10)*. Ed. Briggs R., Pergamon Press, London. 171–178.

Petersen, B., Gernaey, K., Henze, M. and Vanrolleghem, P.A. (2002a) Evaluation of an ASM1 model calibration procedure on a municipal-industrial wastewater treatment plant. *J. Hydroinformatics*, **4**, 15–38.

Petersen, B., Gernaey, K. and Vanrolleghem, P.A. (2002b). Anoxic activated sludge monitoring with combined nitrate and titrimetric measurements. *Wat. Sci. Tech.*, **45**(4–5), 181–190.

Pind, P.F., Angelidaki, I. and Ahring, B.K. (2002). A novel in-situ sampling and VFA sensor technique for anaerobic systems. *Wat. Sci. Tech.*, **45**(10), 261–268.

Powell, G.E. and Archer, D.B. (1989). On-line titration method for monitoring buffer capacity and total volatile fatty acid levels in anaerobic digesters. *Biotech. Bioeng.*, **33**, 570–577.

Preininger, C., Klimant, I. and Wolfbels, O.S. (1994). Optical fiber sensor for biological oxygen demand. *Anal. Chem.*, **66**, 1841–1846.

Qian, Z. and Tan, T.C. (1998). Response characteristics for a dead-cell BOD sensor. *Wat. Res.*, **32**, 801–807.

Ramadori, R., Rozzi, A. and Tandoi, V. (1980). An automated system for monitoring the kinetics of biological oxidation of ammonia. *Wat. Res.*, **14**, 1555–1557.

Rasmussen, M.R. and Larsen, T. (1997). On-line measurements of settling characteristics in activated sludge. *Wat. Sci. Tech.*, **36**(4), 307–312.

Rawson, D.M., Willmer, A.J. and Turner, A.P.F. (1989). Whole-cell biosensors for environmental monitoring. *Biosensors*, **4**, 299–311.

Reid, J.M.C. and Nason, R.B. (1993). Automatic monitoring of activated sludge settling characteristics and plant control applications. *J. Inst. Water Environ. Man.*, **7**, 636–645.

Revsbech, N.P., Nielsen, L.P., Christensen, P.B. and Sørensen, J. (1988). Combined oxygen and nitrous oxide microsensor for denitrification studies. *Appl. Environ. Microbiol.*, **54**, 2245–2249.

Riedel, K., Renneberg, R., Kühn, M. and Scheller, E. (1988). A faster estimation of biochemical oxygen demand using microbial sensors. *Appl. Microbiol. Biotech.*, **28**, 316–318.

Rozzi, A. (1991). Alkalinity considerations with respect to anaerobic digesters. In: *Proceedings 5th Forum Applied Biotechnology. Med. Fac. Landbouww. Rijksuniv. Gent*, **56**, 1499–1514.

Rozzi, A., Ficara, E., Cellamare, C.M. and Bortone, G. (1999). Characterization of textile wastewater and other industrial wastewaters by respirometric and titration biosensors. *Wat. Sci. Tech.*, **40**(1), 161–168.

Rozzi, A., Massone, A. and Amtonelli, M. (1997a). A VFA measuring biosensor based on nitrate reduction. *Wat. Sci. Tech.*, **36**(6–7), 183–189.

Rozzi, A., Remigi, E. and Buckley, C. (2001). Methanogenic activity measurements by the MAIA biosensor: Instructions guide. *Wat. Sci. Tech.*, **44**(4), 287–294.

Rozzi, A., Tomei, M.C., Di Pinto, A.C. and Limoni, N. (1997b). Monitoring toxicity in anaerobic digesters by the Rantox biosensor: theoretical background. *Biotech. Bioeng.*, **55**, 33–40.

Ruzicka, J. and Hansen, E.H. (1975). Flow injection analyses. I. A new concept of fast continuous flow analysis. *Anal. Chim. Acta.*, **78**, 145–157.

Sasaki, K., Yamamoto, Y., Tsumura, K., Hatsumata, S. and Tatewaki, M. (1993). Simultaneous removal of nitrogen and phosphorus in intermittently aerated 2-tank activated sludge process using DO and ORP-bending-point control. *Wat. Sci. Tech.*, **28**(11–12), 513–521.

Sekine, T., Tsugura, H., Urushibara, S., Furuya, N., Fujimoto, E. and Matsui, S. (1989). Evaluation of settleability of activated sludge using a sludge settling analyzer. *Wat. Res.*, **23**, 361–367.

Sikow, M. and Pursiainen, J. (1995). Use and maintenance of on-line measurements at Suomenoja wastewater plant. In: *Preprints of IAWQ Specialized Conference on Sensors in Wastewater Technology*, October 25–27, Copenhagen, Denmark.

Sin, G., Malisse, K., Kotte, K. and Vanrolleghem, P.A. (2003). An integrated sensor for the monitoring of aerobic and anoxic activated sludge activities in biological nitrogen removal plants. *Wat. Sci. Tech.* (this issue).

Sonnleitner, B., Locher, G. and Fiechter, A. (1992). Biomass determination. *J. Biotech.*, **25**, 5–22.

Spanjers, H., Olsson, G. and Klapwijk, A. (1993). Determining influent short-term biochemical oxygen demand by combined respirometry and estimation. *Wat. Sci. Tech.*, **28**(11–12), 401–414.

Spanjers, H. and Vanrolleghem, P.A. (1995). Respirometry as a tool for rapid characterization of wastewater and activated sludge. *Wat. Sci. Tech.*, **31**(2), 105–114.

Spanjers, H., Vanrolleghem, P.A., Olsson, G. and Dold, P.L. (1998). *Respirometry in Control of the Activated Sludge Process: Principles*. IAWQ Scientific and Technical Report No. 7., London, UK.

Spierings, L. (1998). On-line measurement of viable biomass. *Advanced Instrumentation, Data Interpretation and Control of Biotechnological Processes*. Eds. J. Van Impe, P. Vanrolleghem, and D.M. Iserentant. Kluwer Academic Publishers, Dordrecht, The Netherlands, 41–66.

Stephenson, J.P., Luxon, E.G., Monaghan, B.A. and Gillespie, R.G. (1981). Evaluation of instruments for continuous activated sludge monitoring. *Wat. Sci. Tech.*, **13**(10–11), 721–728.

Steyer, J.P., Bouvier, J.C., Conte, T., Gras, P., Harmand, J. and Delgenes, J.P. (2001) On-line measurements of COD, TOC, VFA, total and partial alkalinity in anaerobic digestion processes using infra-red spectrometry. *Wat. Sci. Tech.*, **45**(10), 133–138.

Strand, S.E. and Carlson, D.A. (1984). Rapid BOD measurement for municipal wastewater samples using a biofilm electrode. *J. Water Pollut. Control Fed.*, **56**, 464–467.

Surmacz-Gorska, J., Gernaey, K., Demuynck, C., Vanrolleghem, P.A. and Verstraete, W. (1996). Nitrification monitoring in activated sludge by oxygen uptake rate (OUR) measurements. *Wat. Res.*, **30**, 1228–1236.

Teichgräber, B. (1993). Control strategies for a highly loaded biological ammonia elimination process. *Wat. Sci. Tech.*, **28**(11–12), 531–538.

Thomas, O., Theraulaz, F., Cerda, V., Constant, D. and Quevauviller, P. (1997). Wastewater quality monitoring. *Trs. Anal. Chem.*, **16**, 419–424.

Thomsen, H.A. and Kisbye, K. (1996). N and P on-line meters: requirements, maintenance and stability. *Wat. Sci. Tech.*, **33**(1), 147–157.

Thomsen, H.A. and Nielsen, M.K. (1992). Practical experience with on-line measurements of NH_4^+, NO_3^-, PO_4^{3-}, redox, MLSS and SS in advanced activated sludge plants. In: *Proceedings HYDROTOP92, The City and The Water.* Vol. 2, Marseille, France, 378–388.

Vaidyanathan, S., Macaloney, G., Vaughan, J., McNeil, B. and Harvey, L.M. (1999). Monitoring of submerged bioprocesses. *Crit. Rev. Biotech.*, **19**, 277–316.

Vandenhove, H. (1998). Microclaorimetric characterization of bacterial inocula. In: *Advanced Instrumentation, Data Interpretation, and Control of Biotechnological Processes*. Eds. Van Impe, J.F.M., Vanrolleghem, P.A. and Iserentant, D.M., Kluwer Acedamic Publishers, Dordrecht, The Netherlands, 121–158.

Van Der Pol, J.J., De Gooijer, C.D., Biselli, M., Wandrey, C. and Tramper, J. (1996). Automation of selective assays for on-line bioprocess monitoring by flow-injection analysis. *Trs. Biotech.*, **14**, 471–477.

Van Kleeff, B.H.A., Kuenen, J.G. and Heijnen, J.J. (1993). Continuous measurement of microbial heat production in laboratory fermentors. *Biotech. Bioeng.*, **41**, 541–549.

Vanrolleghem, P.A. and Coen, F. (1995). Optimal dosing of in-sensor-experiments for on-line modelling of nitrogen removal processes. *Wat. Sci. Tech.*, **31**(2), 149–160.

Vanrolleghem, P.A., Kong, Z., Rombouts, G. and Verstraete, W. (1994). An on-line respirographic biosensor for the characterization of load and toxicity of wastewaters. *J. Chem. Tech. Biotech.*, **59**, 321–333.

Vanrolleghem, P.A., Van der Schueren, D., Krikilion, G., Grijspeerdt, K., Willems, P. and Verstraete, W. (1996). On-line quantification of settling properties with in-sensor-experiments in an automated settlometer. *Wat. Sci. Tech.*, **33**(1), 37–51.

Vanrolleghem, P.A. and Van Impe, J.F. (1992). On the use of structured compartment models for the activated sludge biodegradation process. In: *Proceedings Workshop Modelling, Monitoring and Control of the Activated Sludge Process. Med. Fac. Landbouww. Rijksuniv. Gent*, **57**, 2215–2228.

Vanrolleghem, P.A. and Verstraete, W. (1993). Simultaneous biokinetic characterization of heterotrophic and nitrifying populations of activated sludge with an on-line respirographic biosensor. *Wat. Sci. Tech.*, **28**(11–12), 377–387.

Van Vooren, L., Van De Steene, M., Ottoy, J.-P. and Vanrolleghem, P.A. (2001). Automatic buffer capacity model building for the purpose of water quality modeling. *Wat. Sci. Tech.*, **43**(7), 105–113.

Van Vooren, L. and Willems, P. (1993). Milieumetingen: Uitvoering & interpretatie. Analyse van organische stoffen: Groepsparameters. Milieumetingen: Uitvoering en Interpretatie. Vlomitech Course, TI-KVIV, Antwerpen, Belgium (in Dutch).

Van Vooren, L., Willems, P., Ottoy, J.-P., Vansteenkiste, G.C. and Verstraete, W. (1996). Automatic buffer capacity based sensor for effluent quality monitoring. *Wat. Sci. Tech.*, **33**(1), 81–87.

Von Stockar, U. and Marison, W. (1989). The use of calorimetry in biotechnology. *Adv. Biochem. Eng. Biotech.*, **40**, 93–136.

Wacheux, H., Da Silva, S. and Lesavre, J. (1993). Inventory and assessment of automatic nitrate analyzers for urban sewage works. *Wat. Sci. Tech.*, **28**(11–12), 489–498.

Wacheux, H., Million, J.-L., Guillo, C. and Alves, E. (1996). NH_4^+ automatic analysers for wastewater treatment plant: evaluation test at laboratory and field level. *Wat. Sci. Tech.*, **33**(1), 193–201.

Wareham, D.G., Hall, K.J. and Mavinic, D.S. (1993). Real-time control of wastewater treatment systems using ORP. *Wat. Sci. Tech.*, **28**(11–12), 273–282.

Wasiak, K., Heduit, A., Audic, J.M. and Lefèvre, F. (1993). Oxidation reduction potential (ORP)-control of the aeration in a wastewater treatment plant: Nitrogen removal efficiency. A case study: The Benfeld activated sludge plant. *Med. Fac. Landbouww. Univ. Gent*, **58**, 2107–2112.

Watanabe, S., Baba, K., Matuzaki, H., Horaoka, M. and Tsumura, K. (1990). Operation condition diagnosis system for activated sludge processes based on microorganism image information. *Instrumentation, Control and Automation of Water and Wastewater Treatment and Transport Systems (Advances in Water Pollution Control 10)*. Ed. Briggs, R., Pergamon Press, London, 683–690.

Watts, J.B., Evans, K. and Molloy, A. (1990). A wastewater/water process monitoring system allowing operational control – Developed by an operational manager. *Instrumentation, Control and Automation of Water and Wastewater Treatment and Transport Systems (Advances in Water Pollution Control 10)*. Ed. Briggs, R., Pergamon Press, London, 143–154.

White, M.J.D. (1976). Design and control of secondary settlement tanks. *J. Water Pollut. Control Fed.*, **48**, 459–467.

Yano, T., Okano, K., Masduki, A. and Nishizawa, Y. (1993). Measurement of cell density in cultures of aggregrate organisms by continuous-dilution-photometric-assay. *J. Ferm. Bioeng.*, **76**, 123–127.

Yuan, Z. and Bogaert, H. (2001). Titrimetric respirometer measuring the nitrifiable nitrogen in wastewater using in-sensor-experiment. *Wat. Res.*, **35**, 143–150.

Zips, A. and Faust, U. (1989). Determination of biomass by ultrasonic measurements. *Appl. Environ. Microbiol.*, **55**, 1801–1807.

Zumbusch, P.V., Meyer-Jens, T., Brunner, G. and Märkl, H. (1994). On-line monitoring of organic substances with high-pressure liquid chromatography (HPLC) during the anaerobic fermentation of wastewater. *Appl. Microbiol. Biotech.*, **42**, 140–146.