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ON-LINE MONITORING OF NUTRIENT REDUCTION PROCESSES USING MULTIPLE WAVELENGTH REAGENTLESS ULTRAVIOLET ABSORBANCE PROCESS ANALYZERS

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Abstract

The nitrification reaction that takes place at wastewater treatment plants (WWTPs) is a two-step process carried out by two separate microorganisms. Dissolved oxygen level in the water has served as the primary indicator that the microorganisms have sufficient oxygen to function and that the reaction is proceeding properly.

An on-line, real-time, reagentless multiple wavelength ultraviolet (UV) absorbance monitoring device has been developed, partly funded by NASA research, that is capable of accurately differentiating between and measuring nitrate-nitrogen (NO_3-N) and nitrite-nitrogen (NO_2-N) in water and wastewater streams. This product line, the ChemScan™ on-line process analyzer, is able to instantaneously measure the amount of NO_2-N produced during the first step of the nitrification process. The analyzer can also monitor the second-step conversion of NO_2-N to NO_3-N. The addition of a sample conditioning module to the main analyzer can provide ammonia-nitrogen and reactive/orthophosphate analysis. Used alone or in combination with dissolved oxygen, pH, ORP or alkalinity measurements, the process analyzer can be used to provide automated control of the nitrification/denitrification reaction and/or biological phosphorus removal processes in WWTPs, resulting in greater efficiency and cost savings.

I. Introduction

During the past 10 years, many research reports, technical papers and publications have discussed the ammonia, nitrite and nitrate reaction kinetics in some detail (Brennen and Argaman, 1990; Hanaki, et al., 1990; Sedlak, 1989; Manual of Practice, 1983; U.S. EPA, 1993). However, few of the papers have placed great emphasis on the monitoring of nitrite concentration as part of the nitrification process control strategy. This may in part be due to the lack of instrumentation for the rapid, on-line measurement of nitrite. However, as this technology is now available, nitrite and nitrate concentration monitoring should be considered for inclusion in process control strategies. On-line ammonia nitrogen and phosphorus monitoring will also enhance process control.

This new ability to respond rapidly to changes in nutrient reduction processes at WWTPs using multiple analyte, multiple wavelength UV process analyzer measurements should result in operation and maintenance savings. The on-line process analyzer registers process chemistry changes instantaneously. It has up to 8 channels of 4-20 mA output and RS-232 signal capability. Through the use of automated blower and pump controls, the system can be engineered to adjust oxygen delivery, equalization basin flow, return activated sludge (RAS) flow and sludge wasting rates. This real-time, reagentless control strategy has the potential to reduce the frequency of manual laboratory process control testing and can reduce WWTP power costs by optimizing oxygen delivery, mixing and pumping during the entire 24-hour diurnal cycle. Automated adjustments could be made to the RAS rate or the mean cell residence time (MCRT) by changing the waste activated sludge (WAS) rate. In cases where flow equalization is used, this device could also be connected to the equalization tank controls.

The need for on-line nutrient monitoring is great because these biological processes are effected by biological mechanisms rather than simple chemical reactions. Therefore the rate of the various stages of the reaction can

be affected by the non-optimization of any one of several parameters, rapidly putting the process into non-compliance. Some of the factors that affect nitrification and denitrification are listed in Table I.

Table I
Factors Affecting Nitrification
- Temperature - pH and alkalinity relationships - MCRT/WAS rate, RAS rate - Bulk dissolved oxygen concentration - Heterotroph/nitrifier competition - Nitrosomonas to Nitrobacter ratio - Substrate composition variation, electron donor substrate concentration - Half saturation coefficient in relationship to mass transport limitations and diffusional resistances - Multi-substrate limiting kinetics that may affect the relative oxygen uptake rate under transient conditions and varying floc sizes - Toxic substance concentrations, including free ammonia, or free nitrous acid or nitrite buildup.

With the current technology, when WWTP nitrification efficiency rate is reduced, the operations staff may take a long time to recognize the need for process changes and respond with manual adjustments such as an increase in the oxygen supply, the hydraulic retention time, the RAS rate or the sludge age (by decreasing the wasted sludge rate). In many cases, operator response time is slow because laboratory tests for process control purposes take time and are manpower intensive. Although field test kits are less costly and time consuming than laboratory tests, they still require sample collection and manpower to conduct the tests. Many times operators rely on sensory perception alone to operate the plants, only to find out several days later that the plant has been in non-compliance with effluent permit requirements. Real-time unit process chemistry analysis would lead to safer, more cost-effective plant operation. Loud (1986) predicted that computerization in WWTP's and rapid process data generation and analysis could be used to initiate operational or maintenance adjustments which would result in improved effluent quality, reduced plant upsets and increased process and energy efficiency or creation of more available staff time for other projects. Bundgaard, et al. (1993), studied five biological nutrient removal (BNR) plants that were using on-line measurements and concluded that on-line control strategies reduced consumption of both energy and chemicals and improved the effluent quality at the plants. Randall (1992) indicated that there are many environmental, economic, and operational benefits to designing or retrofitting a wastewater treatment plant for biological nutrient removal. On-line monitoring and automated control will provide operators with new tools that will allow data retrieval otherwise unattainable and will promote a greater understanding of their processes.

II. Nitrite Measurement as the Key Control Parameter

Although dissolved oxygen has historically been the main process control parameter, the effects of other limiting factors that may show up earlier can now be monitored. In some cases, an increasing level of nitrite may be used as a first indication of a low dissolved oxygen concentration, which may become inhibitory to the Nitrobacter organisms. However, with emphasis placed on increased plant efficiency and BNR (where a low D.O. zone is essential for some processes), there are more parameters than dissolved oxygen that become important. As

effluent permits become more restrictive, a greater understanding of the biological mechanisms and process conditions to achieve lower effluent values becomes necessary, specifically in terms of nutrient reduction. Automation in some European wastewater treatment works has been more fully accepted and in somewhat wider use for a longer period of time, particularly in France, Germany and the Netherlands. In the United States, the trend toward more stringent effluent quality has fueled the use and acceptance of automation in some facilities. The nature and design of the biological processes involved will determine the degree of monitoring and automated process control necessary.

Nitrogen Conversion Kinetics

The nitrification of ammonia is a two-step process. The first step, conversion of ammonium to nitrite, is mediated by Nitrosomonas. The second step, conversion of nitrite to nitrate, is mediated by Nitrobacter microorganisms. Oxygen is essential to both steps. The equations below demonstrate this two-step conversion of ammonia to nitrate (U.S. EPA, 1993):

$$NH_4^+ + 3/2\ O_2 \longrightarrow 2H^+ + H_2O + NO_2^-$$

$$NO_2^- + 1/2\ O_2 \longrightarrow NO_3^+$$
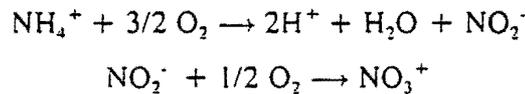
(The ammonium ion NH_4^+ is in a pH dependent equilibrium with ammonia and hydrogen ion: $NH_4^+ \rightleftharpoons NH_3^- + H^+$ [Sawyer and McCarty, 1978].)

Benefield and Randall (1980) illustrate the nitrogen conversion reaction in a polluted stream in Figure 1. This figure can also be used to represent nitrate/nitrite levels within a wastewater treatment plant. The organic nitrogen (@ approx. 32 mg/L) would include proportionately about 20-22 mg/L of NH_3-N, given the general composition of domestic sewage. Upon initial nitrification, the NO_2-N concentration could peak at up to 5 mg/L or more. As NO_2-N oxidation to NO_3-N takes place, the NO_2-N concentration would be reduced to levels below 1.0 mg/L while the NO_3-N concentration would increase. In addition, as shown in formulas for nitrification reactions and equilibria (see equations below) by Anthonisen, et al. (1976), free ammonia and nitrous acid are also present during the reaction.
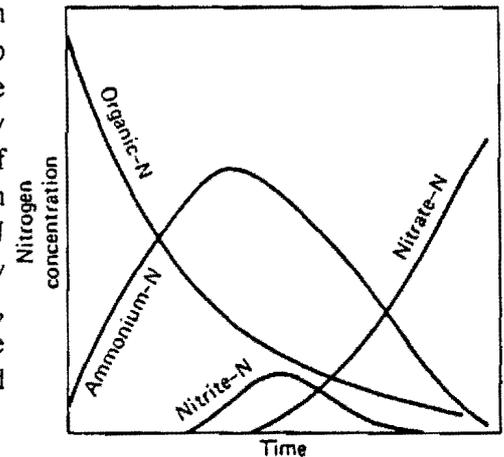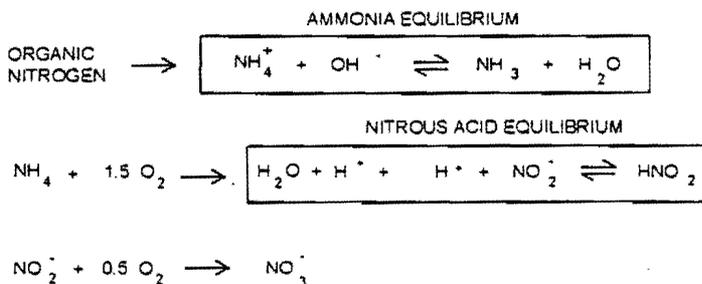

Figure 1. Nitrogen Conversion in a Polluted Stream. (After Sawyer and McCarty, 1967.)

AMMONIA EQUILIBRIUM

$$\text{ORGANIC NITROGEN} \longrightarrow \boxed{NH_4^+ + OH^- \rightleftharpoons NH_3 + H_2O}$$

NITROUS ACID EQUILIBRIUM

$$NH_4 + 1.5\ O_2 \longrightarrow \boxed{H_2O + H^+ + H^+ + NO_2^- \rightleftharpoons HNO_2}$$

$$NO_2^- + 0.5\ O_2 \longrightarrow NO_3^-$$

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In a plant that usually nitrifies fully (< 1.0 ppm ammonia-nitrogen [NH_3-N] remaining in the effluent), one of the first signs of a decrease in nitrification rate is usually a decrease in the NO_3-N concentration and an increase in the NH_3-N and NO_2-N concentrations. In shock organic load situations, a concentration-corresponding decrease in dissolved oxygen is usually noted as well. A reported 65-75% of the oxygen required for nitrification is used in the initial reaction from NH_3-N to NO_2-N.

Biological Phosphorus Reduction

Sen and Randall (1992), discuss a general activated sludge model for biological nitrogen and excess phosphorus removal. The primary model discussed is the IAWPRC model for nitrogen removal extended for the growth of Bio-P or Poly-P organisms. They point to research and observations that suggest there is considerable discrepancy in denitrification rates observed in full-scale nitrification/denitrification biologically enhanced phosphorus removal (NDBEPR) systems in the U.S. They also indicate that there is additional research needed to determine factors that influence denitrification rates for Poly-P and non-Poly-P organisms in NDBEPR systems. On-line real-time nutrient monitoring within the treatment tank may be able to assist in verification, modification or supplementation of model predictions. Of course, of interest to the operator is plant performance moment by moment, especially during unusual or process upset conditions.

| Table II
Factors Affecting Biological Phosphorus Reduction |
|---|
| BOD: P Ratio (i.e., Readily Biodegradable COD; VFA: Acetate)
- Stress on/Stress off sequencing.
- Quality and Quantity of Bio-P organisms.
- Temperature pH, D.O., NO_2-N, NO_3-N.
- Oxidative State of Raw Wastewater (i.e., ORP).
- Presence of PC's or fermenter.
- Side Streams, inplant or internal recycle
- Sludge depth in clarifier.
- Operator Dedication to Bio-P Reduction.
- Percent of Enriched Phosphorus in Biomass.
- MCRT, SRT, HRT.
- Suspended Solids in the effluent.
- Ability to remove phosphorus enriched sludge from main treatment flow routinely. |
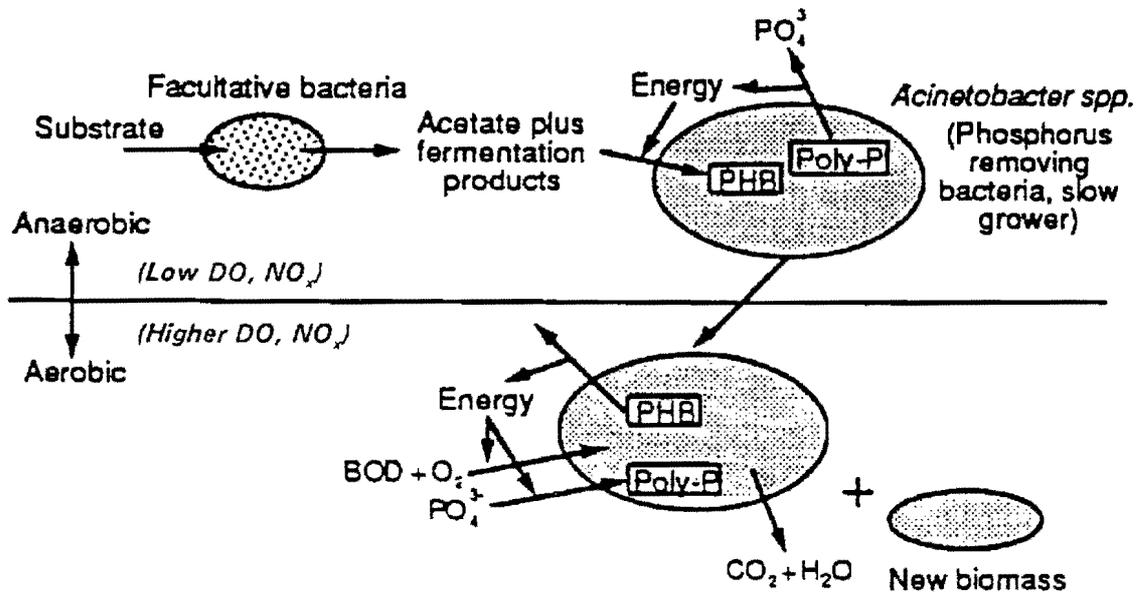
Figure 2. Biological Phosphorus Uptake Mechanism

Figure 2 shows the typical biological phosphorus uptake mechanism. Figure 3 shows the relative ortho-phosphorus concentrations as treatment occurs in a biological phosphorus reduction plant. Note that both figures have been supplemented with low DO/higher DO to indicate that this phenomenon has been documented to also take place in the presence of oxygen (both free and combined forms).
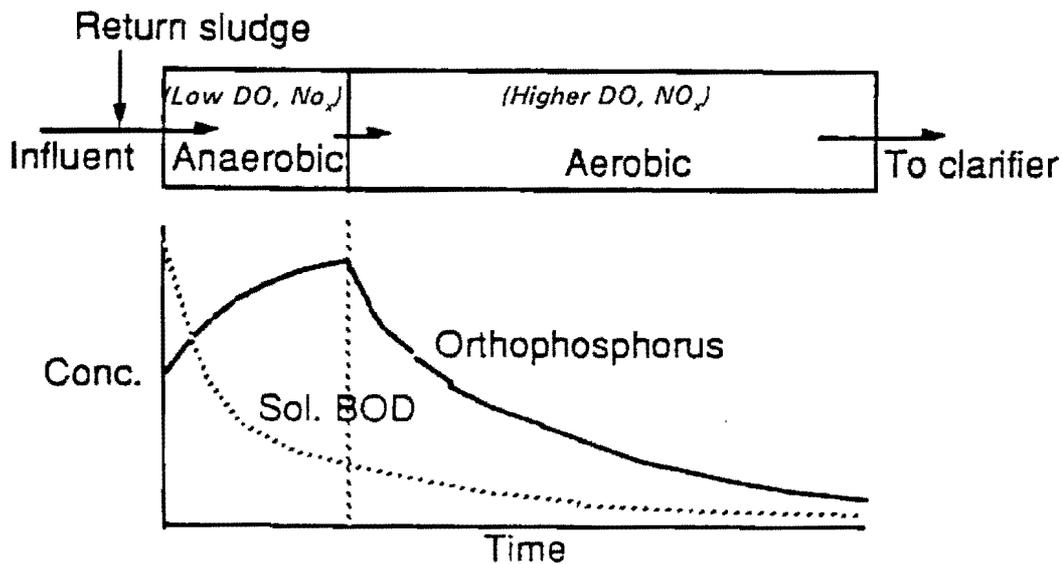

Figure 3. Biological Phosphorus Removal

Throughout the years, researchers have attempted to explain the biological phosphorus release and uptake mechanisms with various biochemical pathway and kinetic models (Comeau, et al, 1985; Gerber, et al, 1986; Tracy and Flammino, 1987; Wentzel et al, 1986, 1990; Park, 1991).

While there are differences between models, most models recognize that stimulation of enhanced biological phosphorus reduction (EBPR) processes require a low dissolved oxygen/higher dissolved oxygen sequencing which creates a stress condition for the microorganisms, and that short chain volatile fatty acids (VFA) such as acetate, play a key role. Wilderer and Dettmer (1987) have conducted experiments with sequencing batch reactors. They concluded that once phosphate accumulating bacteria are selected with low DO conditions, they exhibit polyphosphate uptake and release mechanisms that are independent but inter-related processes. They suggest that once present, the phosphate removal bacteria will function whether or not anaerobic (<0.3 ppm DO) conditions are present, provided there is a food source (VFA) to keep the orthophosphorus release mechanism operable.

A readily biodegradable COD to P ratio of 10:1 was mentioned by Murphy (1993) and Abramson (1994) suggested a short chain VFA:P ratio of 5:1 in the bioreactor as adquate food source for biological phosphorus reduction to take place.

Tracy and Flammino (1987) and Gerber and his associates (1986) and other researchers have proposed that the key feature linking organic substrate metabolism and polyphosphate storage is the polyphosphate kinase enzyme. Wentzel et al (1987) added acetate to phosphorus uptake bacteria under aerobic conditions. They found that phosphorus was released as acetate addition occurred, and that acetate was taken up and stored as Poly-B Hydroxybuterate (PHB) by the bacgteria in the aerobic test tank. Park (1991) seemed to suggest that the stimulus to trigger the phosphorus release mechanism is the presence of VFA's which stimulate the polyphosphate bonds in the phosphate accumulating bacteria to break, usually by enzyme reactions. Wentzel et al (1987) then noted phosphorus concentration in the aerobic liquid increased until the acetate was all taken up, after which the phosphorus concentration declined to substantially lower levels than had been reported in the liquid prior to the acetate addition.

Gerber, de Villiers, Mostert and Diet (1987) studied phosphorus release and uptake following exposure to acetate under both aerobic and anoxic conditions. They discovered that the phosphorus concentration in the liquid is determined and probably dominated by acetate-induced release no matter at which point of the release cycle the liquid becomes aerobic or anoxic. The release then continues until the acetate is dissipated, at which time phosphate uptake commences in the manner historically described with the aerobic stage of treatment. In their experiments, these researchers found that the aerobic environment supported a much higher phosphorus uptake rate than the anoxic environment (low D.O., but with nitrate present). They concluded further that microorganisms with excess phosphate accumulation capability under aerobic conditions, had the capacity to release and take up phosphate simultaneously if an organic substrate such as acetate is present. Which reaction predominates depends on the VFA content present in the liquid, the amount of VFA's converted to PHB in the cells, the reserve of both PHB and polyphosphate granules in the cell structure, and the relative number of electron acceptors in the bulk liquid. The polyphosphate accumulators liberate phosphate by the acetate initiation of the release reaction every time substrates with a high VFA polyphosphate kinase enzyme are present. At the same time the microorganisms are ready to initiate the phosphorus uptake cycle whenever suitable electron acceptors such as oxygen or nitrate are available. In summary, in aerobic or anoxic mixtures of microbiological growth containing acetate or other suitable VFA material, both the phosphorus release and uptake reactions occur concurrently.

Neu (1992, 1993a, b) found that in the range of 0.3-1.1 mg/l DO, phosphorus uptake predominated even though nitrification/denitrification was occurring in a combined fixed film/suspended growth RBC/BNR system. Adequate VFA material was present to stimulate the phosphorus release/uptake mechanism even with NO_3-N present at times.

Automated On-Line Monitoring

There are many variables that will determine the appropriate process control strategies using on-line monitoring and automated process control. Each plant will have its own unique set of requirements. Tracy and Flammino (1987) stated that optimum overall nutrient reduction will be achieved when the biological system is operated at the highest rate that will permit near complete ammonia reduction. In a low DO environment when free and combined oxygen (such as NO_x) occur, the nitrification/denitrification reaction and simultaneous phosphorus release and uptake can occur. Of course, staged configurations may be more efficient for overall nutrient reduction. However, as more attention is given to on-line monitoring in nutrient removal plants, it may be seen that instantaneous nutrient balances can be estimated within the process tank itself, and certain chemical forms of nutrients will become important in optimizing treatment, especially when engineers and operators are being asked to produce cleaner water without increased expenditure. For example, Comeau and his associates (1987) found that nitrate, but not nitrite, could replace oxygen for phosphate uptake and PHA (PHB and PHV) consumption. This could mean that partial nitrification/denitrification in the low DO zone where phosphorus release is stimulated is acceptable and even desirable to improve process efficiency. In other words, nitrite-nitrogen will not adversely affect the biological phosphorus removal process, whereas nitrate-nitrogen could affect this process.

In summary, in the first stage and subsequent stages of a nutrient removal plant, then, it may be important to monitor ammonia-N, nitrite-N and nitrate-N, and/orthophosphate. Wentzel et al (1986) differenciates between Acinetobacter species that are: unable to reduce nitrate, able to reduce nitrate to nitrite, and those species that are able to reduce nitrate to nitrogen gas. This work clearly provides evidence that could justify the need to monitor ammonia-N, nitrate-N, nitrite-N and orthophosphorus within the treatment tankage of a nutrient removal plant. Filtration systems for solids separation have been and are being developed to provide soluble component sample water for on-line process analyzers.

Control Parameters

Historically, automation of process controls in the U.S. wastewater treatment industry has been slow in acceptance by design engineers and use by WWTP operators. Until approximately 10 years ago, technologies were basically not available for on-line automation in WWTPs. Manual operation by physical manipulation of motor/blowers and pump controls was the usual level of control provided to the operator.

The majority of the WWTPs in the U.S. were built prior to 1985, when automated process control was virtually non-existent. So, at a practical level, corrective action to maintain or to increase nitrification or BNR efficiency usually involves operator intervention to manually adjust mixing oxygen supply, RAS and/or WAS rate or to change reaction time by manual adjustment of equalization tank flow.

Because dissolved oxygen is an important parameter to monitor during the biological treatment process, and given the state of technology until recently, the mode of operation was usually to provide a sufficiently high dissolved oxygen level to assure adequate biological oxygen demand (BOD) reduction.

However, some researchers have indicated that nitrite build-up is independent of oxygen concentration. Stenstrom and Song (1991) indicated that an optimal dissolved oxygen concentration to achieve nitrification while reducing aeration costs has not been established. These researchers attempted to explain the effects of oxygen transport on nitrification because to achieve efficient nitrification, a clearer quantification of the effects of dissolved oxygen concentration and the identification of other interdependent factors and their effects were needed. Stenstrom and Song developed a model, compared it with the Monod model and other models and described the reaction kinetics.

Among many conclusions reached by Stenstrom and Song, one is that under steady-state (laboratory) conditions the apparent limiting dissolved oxygen concentration for nitrification in the activated sludge process ranges from 0.5 mg/L to 2.5 mg/L, depending on MCRT (i.e., sludge age) and the degree of mass-transport resistance. They "recorded nitrite accumulation in a high dissolved oxygen (6-8 mg/L) steady-state (laboratory) reactor and concluded in part that the rate of NH_3-N oxidation by Nitrosomonas is typically the rate-limiting step under steady-state conditions, but the rate of NO_2-N oxidation under transient conditions appears to be correlated with transient increases in the rate of NO_2-N production rather than with low dissolved oxygen concentrations."

The above statement makes it clear that there could be parameters other than dissolved oxygen to adjust and control to optimize nitrification efficiency. The NO_2-N accumulation phenomenon appears to be a significant parameter to monitor for more rapid adjustment of process controls to optimize treatment.

The organism responsible for nitrite oxidation to nitrate, Nitrobacter, has been reported by some researchers (Alleman, 1984; Randall and Buth, 1984; U.S. EPA, 1984; Anthonisen, et al., 1976; Suthersan and Ganczarczyk, 1986; Pantea-Kiser, et al., 1990; Lazarova, et al., 1994) to be more sensitive (i.e., its metabolic processes are more easily affected negatively) than Nitrosomonas (ammonia -> nitrite) to changes in oxygen concentration, pH and toxics, even though Nitrobacter exhibits a more rapid metabolic rate under non-limiting conditions. That being the case, nitrite usually gets converted into nitrate quickly because of Nitrobacter's more rapid metabolic rate. However, slight changes in dissolved oxygen or pH (or other parameters) may become limiting for Nitrobacter before they become limiting for Nitrosomonas. If Nitrobacter has insufficient oxygen or is inhibited in some other way, the nitrite to nitrate kinetic conversion rate may decrease and nitrate concentration will decrease. Then, nitrite production continues but the nitrite is not converted to nitrate. Thus, the nitrite may build-up in some cases. Figures 4 and 5 show Anthonisen, et al. (1976), documentation of typical nitrogen transformations during non-inhibited and inhibited nitrification batch experiments. Alleman (1984) lists at least seven conditions under which an elevated nitrite concentration might be realized in a nitrification system, including reduced temperatures, limiting O_2 or CO_2 presence, elevated pH, free ammonia presence, elevated solids wastage, acute process loadings and cryptic nitrate reduction. Alleman also provided laboratory results of bench-scale nitrification studies to demonstrate that a nitrite or nitrate product could be regulated by manipulating certain of these conditions.

If the process is incorrectly monitored, nitrite (and possibly free NH_3, free nitrous acid or other substances) may build up to toxic concentrations or dissolved oxygen
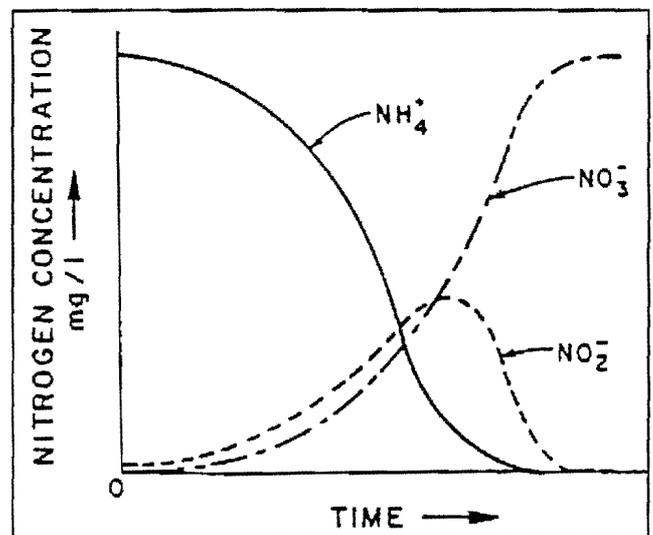

Figure 4. Schematic of nitrogen transformations during noninhibited nitrification, batch experiment.

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could drop to inhibition levels, all of which could create ammonia bleed-through. Some researchers (Anthonisen, et al., 1976; Suthersan and Ganczarczyk, 1986) estimate that nitrite, free ammonia, free nitrous acid, and other materials are up to 100 times more inhibitory to the metabolism of the microbes than the equivalent concentration of nitrate. Therefore, on-line, real-time NO_2-N observation is a key control parameter. ChemScan™ is the only technology available for real-time detection of NO_2-N and NO_3-N in wastewater.

III. Simultaneous Nitrate and Nitrite Ammonia and Orthophosphate Monitoring Strategy
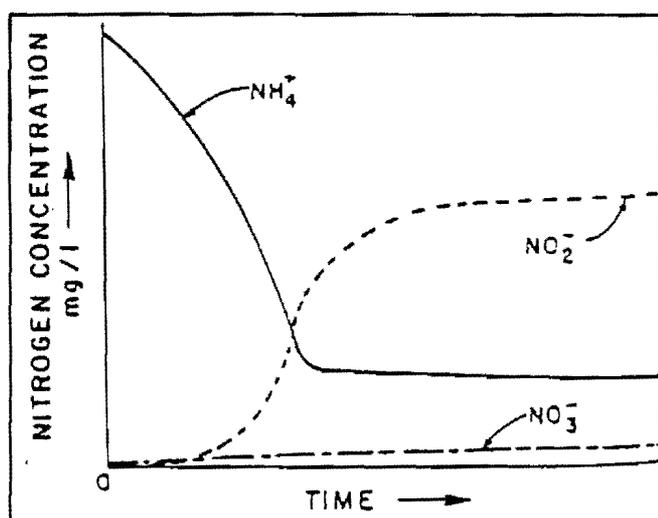

Figure 5. Schematic of nitrification with inhibition of nitrite oxidation, batch experiment.

A ChemScan™ on-line, real-time monitoring instrument can provide instantaneous NO_2-N and NO_3-N concentration readings simultaneously. A sample conditioning module will allow additional analyses such as ammonia and orthophosphate. In conjunction with dissolved oxygen concentration and pH data, an initial automated process control strategy can be developed. In a WWTP that is designed with total nutrient reduction capability, actual in-plant data patterns should be generated, logged and interpreted. These concentration relationships would provide set-point concentration ranges of operation for each parameter of importance. This process control strategy would include, but not be limited to, in-plant manual, or automated adjustments in process flow rate, mixing energy, air delivery, RAS rate or WAS rate. Critical parameter high and low set points (dissolved oxygen, NH_3-N, NO_2-N, NO_3-N, Ortho-P, ORP, Alk., pH) could also be used as signals to initiate further corrective action steps, should the process not come into line with normal or expected concentration ranges.

Chen and Lin (1993) have described denitrification in detail and show the NO_2-N and NO_3-N concentrations over time in several experiments. The new, on-line NO_2-N/NO_3-N monitoring system could be useful to monitor and optimize denitrification in a WWTP, as well as phosphorus release and uptake once basic parameter concentration range set points have been defined. Suthersan and Ganczarczyk (1986) suggest that denitrification could proceed directly from nitrite and be more economical because reduced oxygen and energy requirements would result. This would coincide directly with the fact that nitrite is less likely to affect the phosphorus release/uptake mechanism than nitrate.

This control strategy could also save energy dollars. The system could be automated so that during low-flow or low-organic load periods the process air delivery could be automatically trimmed, based on satisfactory NO_2-N, NO_3-N, and OP results. Chemical phosphorus removal could also be monitored with this process analyzer. The analyzer could monitor orthophosphorus prior to, during or after chemical precipitation. The two most common coagulants, iron and aluminum, could also be monitored with the same instrument. Feedback loops for control purposes could assist in control of coagulant feed. Process monitoring of NH_3-N would still need to be conducted and compliance testing would need to be performed according to the National Pollution Discharge Elimination System (NPDES) permit schedule.

IV. Summary

The advent of the ChemScan™ on-line process analyzer, a device sensitive enough to accurately differentiate NO_2-N from NO_3-N instantaneously and repeatedly in an on-line wastewater treatment environment, could provide a dimension of nitrification process control not previously available. Sample conditioning would also provide ammonia-nitrogen and orthophosphorus values which would produce a clearer visualization of actual biological reactions in the process tankage. This tool could reduce potential permit violations and improve effluent quality and process efficiency. It would improve plant personnel awareness of the treatment process and process control strategies. In summary, on-line process monitoring with a multiple analyte process analyzer would save energy and lower operation and maintenance costs due to overall energy, chemical and manpower cost reductions.

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