Denitrification and Phosphate Removal in Experiments Using Al Stearate

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Abstract

Hydrophobic Al monostearate was tested as a low-solubility denitrification substrate for anaerobic bacteria and a source of aluminum for phosphate precipitation. Flow-through laboratory columns at 25 ± 2^aC were used with O_2-saturated solutions containing 1x, 2x, 8x, and 16x concentrations of 2.26 mg/L NO_3-N and 3.26 mg/L PO_4-P. Denitrification was exponential, approximating first-order reaction kinetics with the rate constant being a function of the initial nitrate concentration. The half life in minutes can be approximated by 5.29 (mg/L NO_3-$N^o)^{1/2}$ where NO_3-N^o was the initial input nitrate concentration. The reaction times were significantly shorter than those required using Ca distearate as a carbon source and much shorter than those using cellulose (white pine shavings) as a carbon source. Al stearate has potential for use in a flow-through container for denitrification of oxidized effluent from home sewage systems.

Aqueous phosphate removal with Al stearate depended upon dissolution of the Al stearate followed by precipitation of Al phosphate. Only 5% to 10% of the phosphate was removed from the solution. The amounts removed were similar to those obtained using bauxite grains as an aluminum source, following saturation of sorption sites with phosphate on bauxite. Aqueous phosphate removal with Ca stearate was insignificant, as the released calcium was apparently precipitated as calcite rather than as hydroxyapatite.

Introduction

Household septic tanks are designed to release effluent containing nitrogen as ammonium and phosphorous as polyphosphates which in an aerobic drain field is followed by the conversion of polyphosphates to orthophosphate and the oxidation of the ammonium to nitrate (Wilhelm et al. 1994). Orthophosphate is often attenuated by soil sorption, and the nitrate can be removed by denitrification through interaction with anaerobic soil bacteria in oxygen-depleted soil zones (Lampert and Sommer 1997). Removal of these nutrients is important in preventing eutrophication from excessive plant growth in streams and lakes (Connell and Miller 1984; Manahan 1994).

Robertson and Cherry (1995) and Robertson et al. (2000) used denitrification in a subsurface infiltration barrier in the field to remove nitrate from effluent draining the aerobic drain fields of septic tanks. The method has also been used to remove nitrate from landfill leachate (Robertson and Anderson 1999). Their denitrification system was an anaerobic layer established in saturated sediment with cellulose (in sawdust) as a carbon source at the base of the aerobic drain field. Reaction times were estimated to be on the order of a week to a month during which the nitrate removal approached 100%. This method provides for long reaction times in a low maintenance environment and is suited for septic tanks with aerobic drain fields.

A problem arises in areas where an aerobic drain field is impractical, e.g., with high water tables or in

urban areas where yard space is limited. A high water table can result in the effluent rising from a drainage field and flowing on the ground surface. In such cases, e.g., St. Tammany Parish in south Louisiana, the effluent from a rural septic tank system is often piped directly to street ditches. Small towns and cities in Third World countries often lack municipal sewage facilities, e.g., in San Ramon, Costa Rica, where the senior author has taught for several years, and usually have individual home septic systems. In this situation in urban areas, the septic tank effluent is fed directly to the street curb. Septic tanks with oxidizing systems can be used to release the nutrients in the effluent as nitrate and orthophosphate, e.g., St. Tammany Parish, Louisiana, eliminating the need for an aerobic drain field. However, the problem remains in removing the nitrate and orthophosphate.

This study tested the use of low-solubility organo-metal compounds to remove these nutrients as part of a low maintenance, long-term, container system which could be placed in-line between the septic tank outflow of oxidized effluent and the drainage outflow. Several organo-metallic compounds were tested that both serve as a labile organic carbon source for heterotrophic bacteria denitrification and also release cations for possible precipitation of phosphates at near neutral pH values.

We also tested cellulose (in white pine shavings) as a carbon source for an in-line system. The problem is that an in-line system is expected to have short reaction times, on the order of an hour. The bacteria were not able to metabolize the cellulose rapidly enough in this time frame to complete most of the nitrate removal. The primary goal of this study was to identify a low-solubility solid carbon substrate that denitrifying bacteria could utilize more rapidly than cellulose.
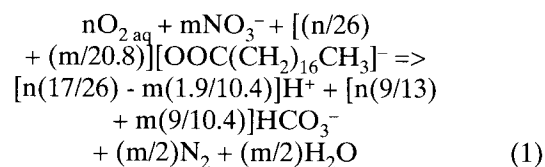
The organo-metallic compounds used in this study were Ca and Al stearates which are reported as low solubility solids by Lide (1991–1992). A low solubility is important in a long-term container system with low maintenance. The compound must not continue to dissolve significantly faster than used by the bacteria in removing nitrate or the container system will require frequent replacement. Other low-soluble organo-metallic compounds (Lide 1991–1992) tested included Al acetylacetonate and Al oxalate; however, the bacteria did not readily metabolize these organic compounds for denitrification.

Organo-Ca and Al compounds were tested because of the possibility of phosphate removal by precipitating variscite [$AlPO_4 \cdot 2(H_2O)$] and hydroxyapatite [$Ca_5(PO_4)_3(OH)$] (Lindsay and Vlek 1977). Phosphate sorption onto aluminum hydroxides and precipitation of $AlPO_4$ hydrates have been documented in soils and in laboratory experiments (Hsu 1977; Lindsay and Vlek 1977). Laboratory experiments by Yamazaki (1999) demonstrated significantly lowered phosphate concentrations by sorption and possible minor precipitation on bauxite within hour-long reaction times. His experiments demonstrated insignificant lowering of phosphate concentrations occurred after the sorption sites were filled. For long-term results, the phosphate has to be removed by precipitation reactions.

The potential precipitation of variscite following aluminum stearate dissolution is reduced by the possibility of aluminum hydroxide precipitating first at near neutral pH values (Langmuir 1997). Likewise, the potential precipitation of hydroxyapatite following calcium distearate dissolution is reduced by the possibility of calcite precipitation due to the introduction of bicarbonate in the dissolution process. Other possible phosphate precipitates include iron phosphates, e.g., strengite ($FePO_4$) and vivianite ($Fe_3(PO_4)_2 \cdot 8H_2O$), which commonly precipitate as soil minerals (Lindsay and Vlek 1977). Yamazaki (1999) used metallic iron to precipitate phosphate during denitrification in flow-through column experiments, but found the effluent caused iron-staining problems.

Nitrate Removal: Denitrification Reactions

Denitrification experiments in this study used cellulose and stearate as a substrate for bacteria metabolism. Denitrification occurs following removal of the dissolved oxygen. Nitrate is reduced through nitrite to N_2 gas. Some of the nitrogen may be reduced to ammonium (Lampert and Sommer 1997); however, only trace amounts of ammonium (< 0.2 mg/L N) were detected in the denitrification experiments of this study. The intermediate reduction product nitrite occasionally showed up in the cellulose experiments but not in the stearate experiments. The carbon source in the stearate experiments was supplied as Al mono-stearate, $Al(OH)_2[OOC(CH_2)_{16}CH_3)]$, and Ca di-stearate, $Ca(OOC(CH_2)_{16}CH_3)_2$. These stearate compounds are described, respectively, by Lide (1991–1992) as being insoluble and slightly soluble (40 mg/L). The overall reaction to remove n moles of dissolved molecular oxygen followed by reducing m moles of nitrate to nitrogen gas is

$$nO_{2\,aq} + mNO_3^- + [(n/26)$$
$$+ (m/20.8)][OOC(CH_2)_{16}CH_3]^- =>$$
$$[n(17/26) - m(1.9/10.4)]H^+ + [n(9/13)$$
$$+ m(9/10.4)]HCO_3^-$$
$$+ (m/2)N_2 + (m/2)H_2O \qquad (1)$$

Equation 2 shows a similar reaction for the reduction of n moles of molecular oxygen and m moles of NO_3 using cellulose (CH_2O) as a carbon source. Carbon actually has a weight fraction of about 0.49 in wood tissue, so the formula is only an approximation (Haygreen and Bowyer 1996).

$$nO_{2\,aq} + mNO_3^- + (n + 1.25m)CH_2O =>$$
$$(n + 1.25m)HCO_3^-$$
$$+ (n + m/4) H^+ + (m/2)N_2 + (m/2)H_2O \qquad (2)$$
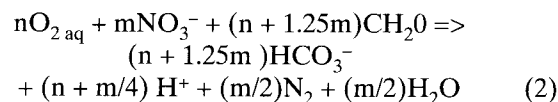
Assuming the initial oxygen concentration is known, the mg of stearate and cellulose needed to denitrify solutions can be calculated from Equations 1 and 2 as a function of aqueous nitrate concentration. O_2 saturation is about 10 mg/L dissolved O_2 at room temperatures in fresh water (Drever 1997); however, unpublished measurements on oxidized septic tank effluent by the senior
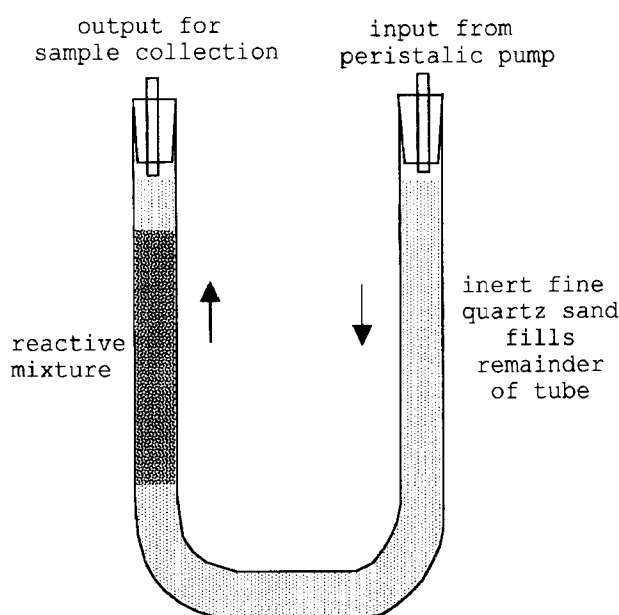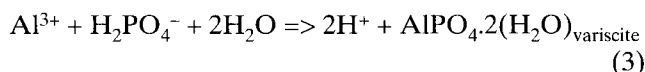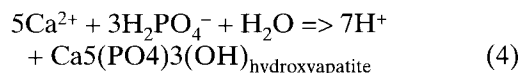
Figure 1. Schematic of column setup.

Table 1			
Experimental Conditions			
Experiment Type	**Components and Pores**	**Weight**	**Volume[a]**
Al Stearate	Al monostearate powder	3.00 g	2.7 mL
	fine quartz sand	12.00 g	4.5 mL
	anaerobic canal soil	0.15 g	0.1 ml
	estimated pore space[b]		5.9 ml
Ca Stearate	Ca distearate powder	2.65 g	1.6 mL
	fine quartz sand	12.35 g	4.7 mL
	anaerobic canal soil	0.15 g	0.1 mL
	estimated pore space[b]		4.2 mL
Cellulose	dried pine shavings	1.99 g	4.0 mL
	fine quartz sand	1.44 g	0.5 mL
	anaerobic canal soil	0.30 g	0.2 mL
	estimated pore space[b]		4.7 ml
Bauxite	fine bauxite sand	3.00 g	1.3 mL
	fine quartz sand	2.95 g	1.1 mL
	estimated pore space[b]		1.9 mL

[a]Densities used in computing volumes were 1.5, 1.1, 1.7, 0.5, 2.4, 2.65 g/mL, respectively, for soil, Al monostearate, Ca distearate, white pine shavings, bauxite, and quartz.
[b]Pore volumes were based on the measured volume minus the volumes of the solid components.

author were always less than 5 mg/L. Once the O_2 is removed, weight ratios of 0.220 of stearate to nitrate and 0.605 of CH_2O (our cellulose approximation) to nitrate are required to complete denitrification.

Phosphate Precipitation Reactions

Phosphate removal by precipitation was tested using bauxite and Al stearate as Al sources and Ca stearate as a Ca source. At pH values just below neutral, phosphate should be predominantly in the $H_2PO_4^-$ species and as HPO_4^{2-} at pH values just above neutral (Stumm and Morgan 1981). In both cases, precipitation of variscite and hydroxyapatite should make the solution more acidic. The decrease in pH is greater if phosphate is in the form of $H_2PO_4^-$. Example reactions are

$$Al^{3+} + H_2PO_4^- + 2H_2O => 2H^+ + AlPO_4 \cdot 2(H_2O)_{variscite}$$
(3)

and

$$5Ca^{2+} + 3H_2PO_4^- + H_2O => 7H^+$$
$$+ Ca5(PO4)3(OH)_{hydroxyapatite}$$
(4)

Experimental Procedure

The experimental system is schematically shown in Figure 1, and the experimental conditions are listed in Table 1. Porous reactive components were mixed with fine quartz sand and placed within the arms of 50 mL, 28 cm-long U-shaped glass tubes which were used as columns. Fine quartz sand was used to pack the tubes on both sides of each reactive mixture. The soil which served as a source for anaerobic bacteria in the experiments was a dark clay taken below the bottom at different sites along the eastern edge of the West Side University of New Orleans Canal. New soil samples were taken for use in each new experiment.

The porosity of the reactive mixture was needed to estimate the hydraulic retention time which is the time required to flow through the reactive mixture a fluid volume equal to one pore volume in the mixture. This is a maximum reaction time because of possible short-circuiting the flowpath within the reactive mixture. The masses and densities of the reaction components, together with the measured volume occupied in the column, were used to estimate the porosity.

One reactive mixture was used per column in the Ca and Al stearate experiments. The stearate was a fine hydrophobic powder: aluminum stearate monobasic (USP grade, 15.4% by wt Al_2O_3, Pfaltz & Bauer #A16510) and calcium distearate (USP grade, 98% pure, Pfaltz & Bauer #C01875). The stearate material was mixed with fine quartz sand and 1 wt % of anaerobic soil (to provide anaerobic bacteria) and placed in the upstream arm of the U-shaped tube. The densities of the aluminum monostearate and the calcium di-stearate were approximated by 1 g/cm³, the density of aluminum tri-stearate (Lide 1991–1992). The amounts of Ca and Al stearate used in the experiments were adjusted so that equal moles of stearate were present (Table 1), resulting in smaller reaction volumes in the column for Ca stearate.

Cellulose and bauxite experiments were also run to contrast their potential for nutrient removal with those obtained from the stearate experiments. These experiments were run concurrently (in adjacent arms in the U-shaped tubes). A mixture of white pine shavings (cellulose), 1 wt % anaerobic soil, and fine quartz sand was placed in the downstream arm and a fine sand-size mixture of bauxite and quartz sand was placed in the upstream arm with fine quartz sand separating the two reaction regions. The cellulose was assumed to have a density of 0.5 g/cm³ (Haygreen and Bowyer 1996). The bauxite
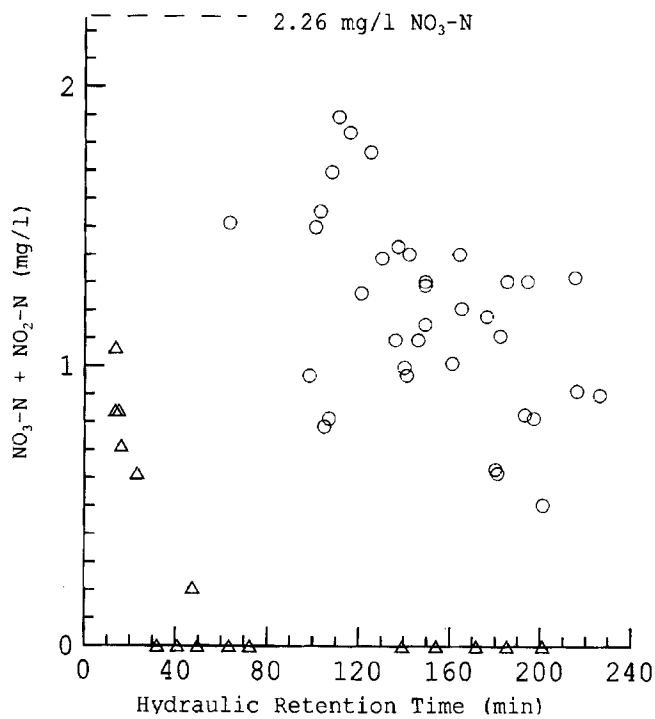
Figure 2. Denitrification of 2.26 mg/L NO_3-N solutions. The experiments utilized cellulose (pine shavings), O, and Al stearate, △, as a carbon source.

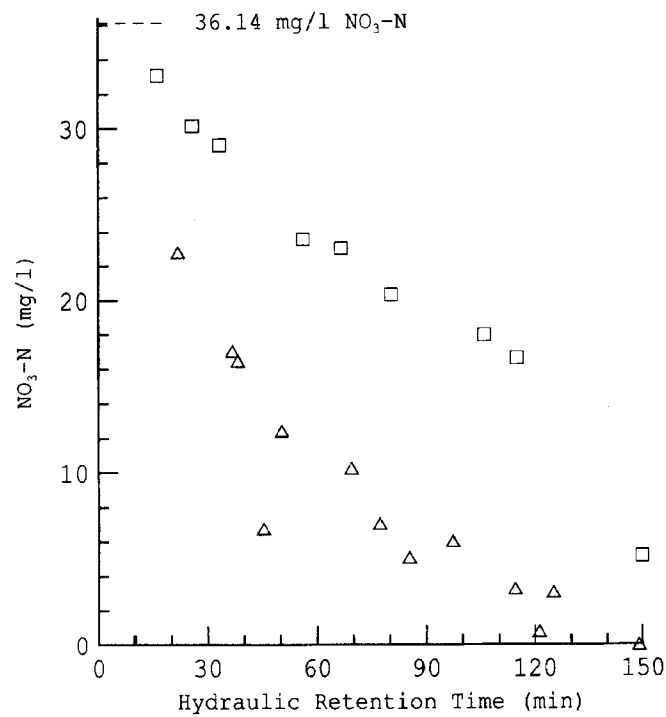**Figure 3.** Denitrification of 36.14 mg/L NO_3-N solutions, utilizing Al stearate, △, and Ca distearate, □, as carbon sources. NO_2 was below detection.

Exponential Denitrification Curves

$$mg/l\ NO_3\text{-}N = (mg/l\ NO_3\text{-}N°)\exp[-kt_{min}]$$

$$k = 0.131/(mg/l\ NO_3\text{-}N°)^{1/2}$$
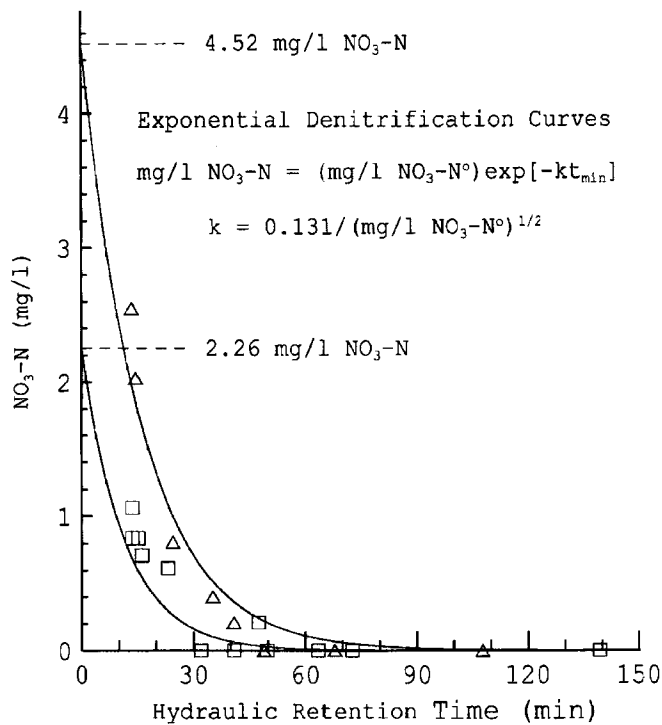
Figure 4. Denitrification of NO_3 solutions of 2.26 mg/L NO_3-N, □, and 5.52 NO_3-N mg/L, △. NO_2 was below detection.

Exponential Denitrification Curves

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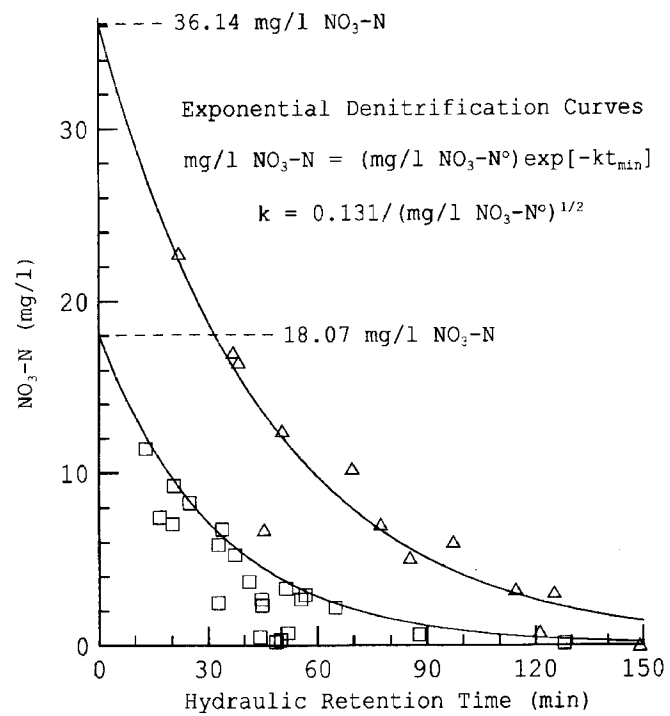
Figure 5. Denitrification of NO_3 solutions of 18.07 mg/L NO_3-N, □, and 36.14 NO_3-N mg/L, △. NO_2 was below detection.

sand had a grain size range from 0.009 to 0.035 cm. Assuming a cubic shape, the surface area of the bauxite ranged from 278 cm²/g to 71 cm²/g. X-ray diffraction runs of the bauxite indicated gibbsite to be the only crystalline mineral present; however, the brown surface color indicated trace amounts of ferric oxides. The density of the bauxite was taken to be that of pure gibbsite, 2.4 g/cm³. Bauxite usually also contains diaspore and boehmite

(Hsu 1977). If these aluminum hydroxides were present in the sample, they must have been poor crystalline phases.

An oxygen-saturated (O_2) solution made from distilled and deionized water and reagent sodium or potassium salts of NO_3 and PO_4 was pumped through each tube at 25 ± 2^aC by a peristaltic pump. Four solution compositions were used in the experiments: 1x, 2x, 8x, and 16x of 2.26
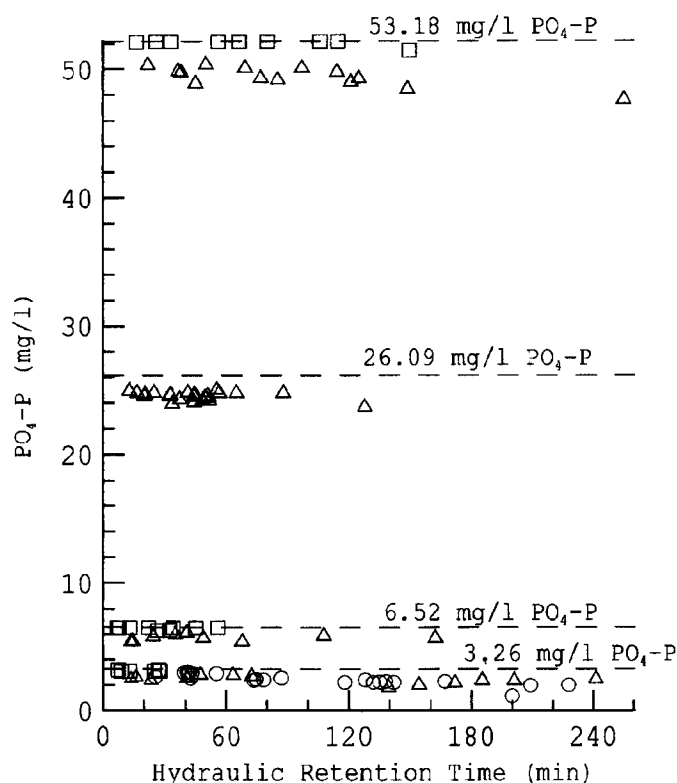
Figure 6. Phosphate removal after PO_4 sorption was complete utilizing bauxite, ○, Al stearate, △, and Ca stearate, □. The dashed lines show the four input concentrations.

mg/L NO_3-N and 3.26 mg/L PO_4-P. The NO_3^- concentrations covered the range found by the authors in an unpublished survey of oxidized septic tank effluent from homes in unincorporated St. Tammany Parish near the north shore of Lake Pontchartrain.

Each new column was conditioned by flushing the influent solution through it for two weeks prior to the beginning of sampling. The conditioning allowed the anaerobic bacteria to increase in numbers to produce maximum denitrification. Experiment durations were from a few hours to a few days. The in situ fluid in the column was flushed out at the new flow rate, prior to the beginning of a new experiment. During a column experiment, the effluent was collected continuously in flasks for weighing at the end of the experiment to calculate the flow rate. An average of 50 pore volumes were collected for each data point.

The columns were changed when the flow rates at a particular peristaltic pump setting either increased or decreased unexpectedly. The life of a column was about two months. The reason for an unexpected change in flow rates was probably due to either fractures opening in the flowpath or the plugging of pore holes with the hydrophobic stearate.

All chemical analyses were done by the senior author. A portion of the output samples were filtered and a DIONIX 100 liquid ion chromatograph was used to analyze for dissolved concentrations of major anions using EPA Method 300.0. Major cations were also analyzed

by liquid ion chromatography for selected samples. The estimated accuracy of the measurements, based on reproducibility with different sets of standards, was 0.1 mg/L. For the stearate samples, the pH was also measured on the unfiltered samples and the alkalinity was titrated for selected filtered samples. The pH of the samples generally varied from 6.6 to 7.4. Aluminum was measured on a DCP Emission Spectrograph for representative samples, and the values were below detection (<2 µg/L). The data are not adequate to determine if the solution reached chemical equilibrium with an aluminum phase. Organic carbon was not measured because of the lack of a carbon analyzer. The chemical data taken from all the experiments are available from the senior author upon request.

Results: Nitrate Removal

A comparison of the use of cellulose (white pine shavings) and Al stearate in denitrification is shown in Figure 2 for input solutions that had 2.26 mg/L NO_3-N. During denitrification, some of the samples had nitrite present in the cellulose experiments, although none was found in the stearate experiments. In the Al stearate experiments, the nitrate concentrations drop below detection with hydraulic retention times longer than 50 minutes. In the cellulose experiments, even after three hours the nitrate plus nitrite concentrations ranged from 25% to 60% of the input nitrate concentration.

The use of Al stearate and Ca stearate in denitrification is compared in Figure 3 for input solutions that had 36.14 mg/L NO_3-N. After two hours of hydraulic retention time, nitrate had been reduced to less than 10% in the Al stearate experiments and it was still above 45% on the Ca distearate experiments. We had initially expected Ca distearate to be more effective than Al stearate, because it is more soluble and would provide more carbon in the water column for denitrification. However, similar relative disparities showed up in experiments run with both compounds utilizing 4.52 mg/L NO_3-N. The results with the 2.26 mg/L NO_3-N were inconclusive.

The data from the Al stearate experiments are plotted in Figures 4 and 5. The solid curves are computed using first-order kinetics in which the rate constant k is a function of the initial N concentration, mg/L NO_3-N°. The kinetic equation with time t in minutes is

$$mg/L \ NO_3\text{-}N = (mg/L \ NO_3\text{-}N°) \ exp(-kt) \qquad (5)$$

where

$$k = k° / (mg/L \ NO_3\text{-}N°)^{1/2} . \qquad (6)$$

k° was 0.131 $(mg/L)^{1/2}$/min). The half life in minutes, $t_{1/2}$ becomes

$$t_{1/2} = 5.29 \ (mg/L \ NO_3\text{-}N°)^{1/2} \qquad (7)$$

For mg/L NO_3-N° of 36.14 on Figure 5, the denitrification appears to be more linear and rapid with time than at the other input concentrations. Because bacterial

growth is known to follow first-order kinetics (Manahan 1994), denitrification is expected to also follow first-order kinetics. The kinetics will probably vary with a number of additional factors, including different species of bacteria and temperature, and additional experiments are needed to further quantify the reaction kinetics.

The important point in Figures 4 and 5 is that denitrification was (with the exception of one data point) more than 90% complete within 30, 50, 90, and 120 minutes of hydraulic retention, respectively, for 2.26, 4.52, 18.07, and 36.14 mg/L NO_3-$N°$. These are short reaction times and support the possibility of using aluminum stearate as a denitrification substrate for a container system to remove nitrate from the oxidized effluent of home septic tanks.

Results: Phosphate Removal

Minor phosphate removal, generally 5% to 10%, occurred in the bauxite and Al stearate experiments and insignificant removal occurred in the Ca stearate experiments. The aqueous phosphate concentrations are plotted in Figure 6 as a function of reaction time. The data shown in the figure are for samples taken after all surface sorption sites had been filled, as shown by constant phosphate concentrations in the effluent at a constant flow rate during the initial column conditioning. The phosphate sorption on the Al and Ca stearate compounds could not be measured directly because of the hydrophobic nature of stearate in water; however, from column results, the sorption appeared to be insignificant. Phosphate sorption on the bauxite was less than 10 meq/100 g as determined from monitoring the column effluent as a function of time for a constant flow rate in the experiments. The absence of phosphate removal in the Ca stearate experiments is presumed due to precipitation of calcite. The increase in alkalinity and the rise in pH to above 7 that occurred due to Ca stearate dissolution and denitrification should promote the rapid precipitation of calcite.

The phosphate removal shown in Figure 6 in the Al stearate and bauxite experiments is presumed due to precipitation of an aluminum phosphate phase. For the aluminum stearate experiments, the decrease in phosphate is less than 50% predicted from the amount of aluminum released from the destruction of aluminum stearate during denitrification. Apparently much of the released aluminum is being precipitated as an aluminum hydroxide phase, rather than an aluminum phosphate phase such as variscite. In the bauxite experiments, the release of aqueous aluminum by bauxite dissolution is limited by slow reaction kinetics at room temperature and the low solubility of aluminum hydroxide phases at near neutral pH (Drever 1997, their Figure 10.4). Both the Al stearate and bauxite experiments showed a small increase in phosphate removal with increasing hydraulic retention time (Figure 6), presumably due to increased phosphate precipitation with increasing reaction time.

Potential Denitrification Applications

These experiments indicate that Al stearate has potential for use in denitrification of waste water in either a container system or within a porous barrier. The chemical is used as a thickening additive in industrial processes and technical grades in powder form that can be purchased in bulk from many chemical companies, e.g., Mallinckrodt. Complete denitrification of 1500 L of septic-tank effluent per day (Equation 1) with an average 20 mg/L NO_3-N and 5 mg/L O_2 would require about 70 kg of Al stearate over five years at a present-day (year 2000) bulk price of about $300. The price should be significantly reduced if a market developed for the process.

A provisional patent (U.S. Serial Number 60/205,158; May 18, 2000) was recently awarded to the senior author and the University of New Orleans for use of metal stearates in denitrification of waste water. The hydrophobic nature of the compound allows it to coat solid surfaces and form micelles which float upwards in water. A denitrification container is being designed for removing nitrate from the oxidized effluent of home septic tanks in which the outlet pipe is placed below the water level to prevent outlet fluid from removing floating Al stearate.

A schematic drawing of a denitrification cell is shown in Figure 7 using aluminum stearate, in either granules or in sheet form. Mallinckrodt has produced a granule form in the past and is researching the idea of producing it in sheets. Excess powder can clog the system, making sheets or granules the preferred form of the stearate. The sheet form would be the most convenient because new sheets could be inserted to replace those eaten by bacteria, and space could be made available for pumping out any accumulated sludge by temporarily pulling out some of the sheets. Field tests will be run on modifications of the system shown in Figure 7 with the results available in 2001.

Summary

Nitrate and phosphate removal from aqueous solutions with equal mg/L of nitrate and phosphate were tested in the laboratory column experiments. Al stearate was found to be an effective low-solubility carbon substrate for rapid denitrification by bacteria of nitrate in nitrate-rich solutions. Complete removal of nitrate occurred in less than an hour from input solutions containing 4.52 mg/L NO_3-N, in less than two hours from input solutions containing 18.07 mg/L NO_3-N and in two and one-half hours from input solutions containing 36.14 mg/L NO_3-N. The removal generally followed first-order kinetics in which the rate constant for each experiment was a function of the input concentration. The half life in minutes was 5.29 (mg/L NO_3-$N°$)$^{1/2}$ where mg/L NO_3-$N°$ was the initial input concentration. Ca stearate was less effective and cellulose (white pine shavings) was not nearly as effective as a carbon substrate for denitrification.

Insignificant phosphate removal occurred in experiments using Ca stearate. However, 5% to 10% of the aqueous phosphate was removed by using Al stearate in the denitrification process and by bauxite dissolution. The removal occurred within minutes and showed a small increase for reactions times of a few hours.
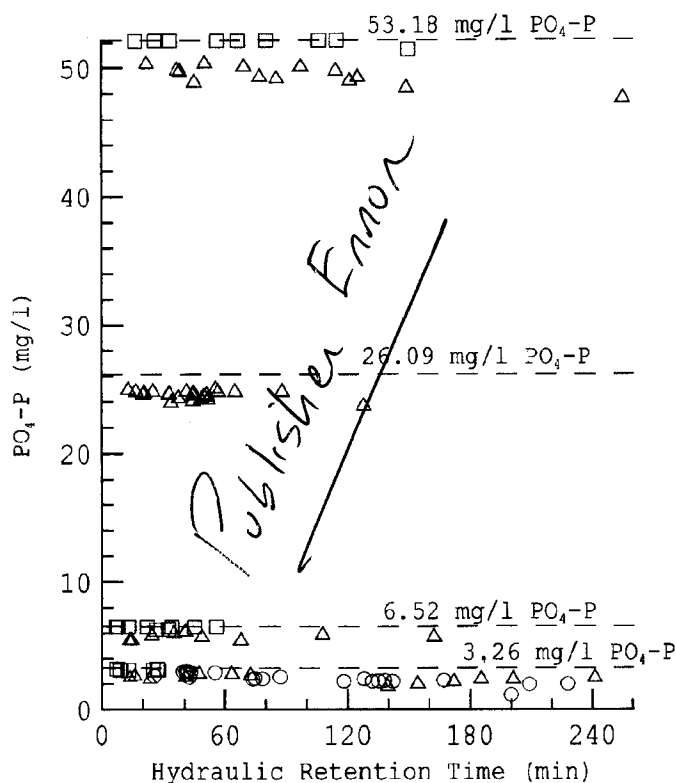
Figure 7. Schematic of waste water denitrification cell.

Acknowledgments

Editor's Note: The use of brand names in peer-reviewed papers is for identification purposes only and does not constitute endorsement by the authors, their employers, or the National Ground Water Association.

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Biographical Sketches

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