

TREATMENT OF HIGH-AMMONIA ANIMAL WASTEWATER WITH NITRIFYING PELLETS

by

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Summary: Nitrification of ammonia (NH_4^+) is a critical component for improved systems of animal wastewater treatment. Biological removal of N is regarded as the most economically feasible N removal process, but the application to animal systems is difficult because of the inhibition of nitrifying bacteria by high-ammonia concentration. One of the most effective nitrification processes uses nitrifying microorganisms encapsulated in polymer resins. We adapted this technology for high-ammonia wastewaters by using acclimated microorganisms. High-ammonia nitrifying bacteria (HANB) were successfully immobilized in 3- to 5-mm polyvinyl alcohol (PVA) polymer pellets. Swine wastewater containing 350 to 2600 mg N/L was treated in aerated, suspended bioreactors using batch and continuous flow treatment. In batch treatment, ammonia removal rates of 38 to 41 mg-N/L-reactor/h (915 to 990 mg-N/L-reactor/day) were obtained with 97 to 100% nitrification efficiency. In continuous flow, efficiencies >80% were obtained with NH_4^+ loading rates < 1000 mg N/L-reactor/d and HRT of 24 to 48 h. All of the NH_4^+ -N removed was entirely recovered in oxidized N forms. Comparison between a single reactor vs. two or three reactors in series showed that there is no advantage to using the plug-flow regime. Our results indicated that the process pH is important for optimization of nitrification in high-ammonia wastewaters and that improper selection may result in 50% decrease in performance. Optimum nitrification performance (100% efficiency) was obtained when the pH was controlled at 8.5. Our results also indicated that immobilized HANB pellets were not inhibited by high NH_4^+ concentration in the range of 350 to 2600 mg N/L and that both NH_4^+ and NO_2^- oxidizers entrapped in these pellets operated simultaneously providing fast and complete nitrification. The results of this study indicate that the HANB pellets are a useful technology for fast and efficient nitrification treatment of high-ammonia animal wastewaters.

Keywords: Animal wastewater, Swine lagoons, Nitrification treatment, Nitrifying pellets, Ammonia Emissions

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INTRODUCTION

Animal waste treatment is a significant agricultural and environmental challenge that needs additional options as a result of expanded, confined animal production. Interest has greatly increased in finding alternative methods of nutrient management for confined animal production that are functional and affordable. For nitrogen, this need ranges from new technologies that reduce ammonia emissions from existing operations to the design of new systems without lagoons such as the use of liquid/solids separation and aerobic treatment of the liquid effluent. Although biological removal of N through the process of nitrification and denitrification is regarded as the most efficient and economically feasible method available for removal of N from municipal wastewaters (Tchobanoglous and Burton, 1991), the application to animal systems is difficult because of the inhibition of nitrifying bacteria by high-ammonia concentration (150 to 1800 mg $\text{NH}_4\text{-N/L}$) typical of these effluents.

Another problem is that nitrifying bacteria have a slow growth rate compared to heterotrophic microorganisms, and with effluents containing high BOD concentration, the nitrifiers tend to be overgrown or washed out of reactors (Figueroa and Silverstein, 1992). Thus, recycling of surplus activated sludge in an aerobic reactor or long hydraulic retention time (HRT) is required to retain slow growing autotrophic nitrifiers. Unfortunately, in the absence of enriched nitrifying populations, aerobic treatment of high-ammonia animal wastewater can potentially add to problems by stripping ammonia into the atmosphere (Burton, 1992; Vanotti and Hunt, 1998). The efficiency of the nitrification process can be improved by increasing the nitrifiers' retention time independent from the wastewater retention time (Wijffels et al., 1993). In most cases, this is done by immobilization via spontaneous attachment of cells to the surface of inert support materials. Applications of attached growth for treatment of high-ammonia swine wastewater (1000 to 1800 mg N/L) have been developed by Ciaccolini et al. (1984) and St.-Arnaud et al. (1991) who reported higher nitrification rates when compared to systems where microorganisms were in suspension. However, higher nitrification rates are feasible because of advances in biotechnology that uses immobilization entrapment of cells in polymer gels.

NITRIFYING PELLET TECHNOLOGY

The use of microorganisms immobilized in polymer gels is a common technique in drug manufacturing and food processing where conditions are modified to enhance the activity of specific microorganisms performing a desirable chemical process. The application for municipal wastewater treatment has been recently developed and tested in Japan (Takeshima et al., 1993), and there are currently several full-scale municipal wastewater treatment plants using this technology. Through the immobilization process, the nitrifying microorganisms are provided with a very suitable environment to perform at maximum effectiveness. The nitrifiers are entrapped in 3- to 5-mm pellets made of polymers that are permeable to NH_3 , oxygen, and carbon dioxide needed by these microorganisms. Tanaka et al. (1991) reported nitrification rates three times higher than those of the conventional activated sludge process. Typical materials are polyethylene glycol (PEG) and polyvinyl alcohol (PVA); these pellets are

functional for more than 10 years. We previously reported that this technology can be adapted for fast and efficient removal of NH_4^+ contained in anaerobic lagoons by using acclimated microorganisms (Vanotti and Hunt, 1998). Nitrifying bacteria were cultivated in a medium containing 300 mg NH_4^+ N/L, immobilized in PVA polymer pellets, and then used for nitrification of swine lagoon wastewater containing ~ 230 mg NH_4^+ N/L. Nitrification efficiencies of more than 90% were successfully obtained, even at both short HRT of 12 h and an NH_4^+ loading rate of 420 mg N/L-reactor/day. One limitation in using batch treatment was that NO_2^- oxidizing bacteria were inhibited by initially high NH_4^+ N concentration, and this inhibition was not relieved until NH_4^+ N concentration was < 70 mg/L. Thus, in batch mode, the total treatment time needed for complete NH_4^+ oxidation to NO_3^- was almost doubled the time necessary for simple elimination of ammonia.

NITRIFYING PELLETS FOR HIGH-AMMONIA WASTEWATER

Our approach was to use immobilized pellet technology with nitrifying bacteria acclimated to high-ammonia wastewater (900 ppm). The bacteria were obtained from a pilot reactor used for nitrification treatment of swine lagoons (Vanotti and Hunt, 1998). The reactor used PEG pellets that were produced in Japan by the Hitachi Plant Engineering Company and contained 2% activated municipal sludge. We first conducted a field acclimation procedure to adapt the nitrifiers contained in the PEG pellets for swine wastewater, which contains 10 to 20 times more ammonia-N than the municipal wastewater treatment plant where the bacteria were extracted. The nitrifying pellets were successfully acclimated to lagoon effluent during a 3-month period in which the ammonia loading rate was increased by decreasing the hydraulic residence time. In this work, nitrification activity of the PEG pellets increased from 21 to 433 g N/m³ tank/d (Vanotti and Hunt, 1998). We used these microorganisms to prepare a culture of free nitrifying cells that were not inhibited to high-ammonia wastewater. For this work, which is presented in this paper, 10 g of PEG pellets from the pilot plant were ground and used as a seed in a fill-and-draw culture of microorganisms tolerant to high-ammonia strength wastewater. The ammonia concentration of the inorganic salts medium (pH 8.5) used for the culture of the free cells was increased gradually from 300 to 900 mg N/L over a 20-d period to overcome inhibitory effects caused by high levels of free ammonia in the medium. After 45 d of incubation at 35°C, the high-ammonia nitrifying bacteria (HANB) cells were harvested by sedimentation and entrapped in PVA pellets using the polymerization method of Hashimoto et al., 1986. The HANB pellets were subsequently tested with various ammonia strength wastewater containing up to 2500 mg NH_4 -N/L

LIQUID SWINE MANURE

The wastewater used was a lagoon effluent from a swine operation near Kennansville, in Duplin Co., North Carolina. This was a single-stage anaerobic lagoon of 4100-m³ volume providing treatment and storage of flushed swine manure from 2600 pigs. The lagoon liquid contained 350 NH_4 -N/L, 400-mg Total Kjeldahl N/L, and 0-mg/L NO_2^- and NO_3^- . Other characteristics were 470-mg total suspended solids/L, 1500-mg total solids/L, 824-mg COD/L, 1920-mg alkalinity/L, and a pH of 7.9. The concentration of ammonia in this wastewater was

increased to levels of 900, 1600, and 2500 mg N/L by mixing various amounts of $(\text{NH}_4)_2\text{SO}_4$ reagent with the liquid waste and used to test the performance of HANB pellets under batch and continuous flow treatment modes.

REACTOR CONFIGURATION

A diagram of the experimental apparatus used in the nitrification experiments is shown in Fig. 1. The basic reactor consisted of a 1.2 L aeration tank and was modeled after the Pegasus process (Takeshima et al., 1993). Fine bubble aeration was supplied with a porous diffuser from the bottom of the tank at a flow rate of 0.4 L min^{-1} . This ensured full fluidization of pellets and provided high oxygen transfer. The lowest DO concentration was 6.7 mg L^{-1} (average = 7.7 mg L^{-1}). Pellets were added at 15% (w/v) pellet to reactor volume ratio. A 1-mm screen was placed before the outlet port to separate the pellets and free cells, and retain the pellets inside the reactor. Temperature was controlled using a heat regulator and a circulated water bath that accommodated up to three reactors. All the experiments were conducted at a wastewater temperature of 30°C . A variable flow peristaltic pump was used in the continuous flow experiments to feed the lagoon wastewater to the nitrification tank. The $\text{NH}_4\text{-N}$ loading rate was adjusted by varying the flow rates. The process pH was regulated using pH controllers and pumps that injected 1 N NaOH and kept the pH within a narrow band of 0.2 units. Modifications to the configuration shown in Fig. 1 included two and three reactors in series where the effluent of one reactor was gravity fed into a subsequent reactor and batch experiments using the same 1.2-L reactors without an influent line.

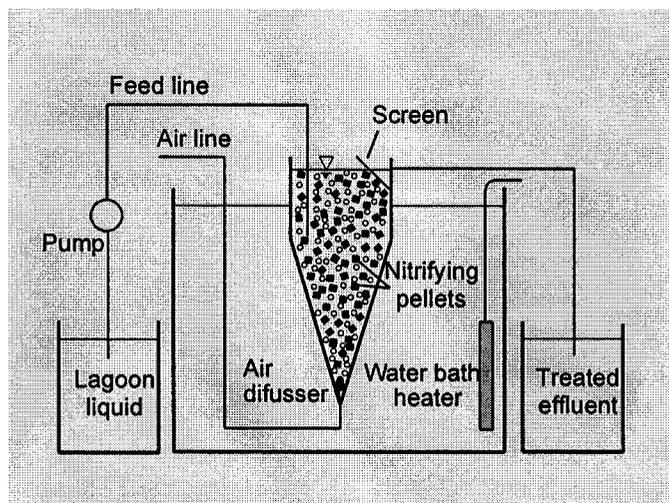


Figure 1. Reactor configuration.

BATCH TREATMENT EXPERIMENTS

The HANB pellets showed excellent capability for fast and efficient removal of NH_4^+N from high-ammonia wastewater. Data in Fig. 2 show nitrification performance in a batch run for swine wastewater containing $344 \text{ mg NH}_4^+\text{-N}$. Ammonia was nitrified readily, decreasing

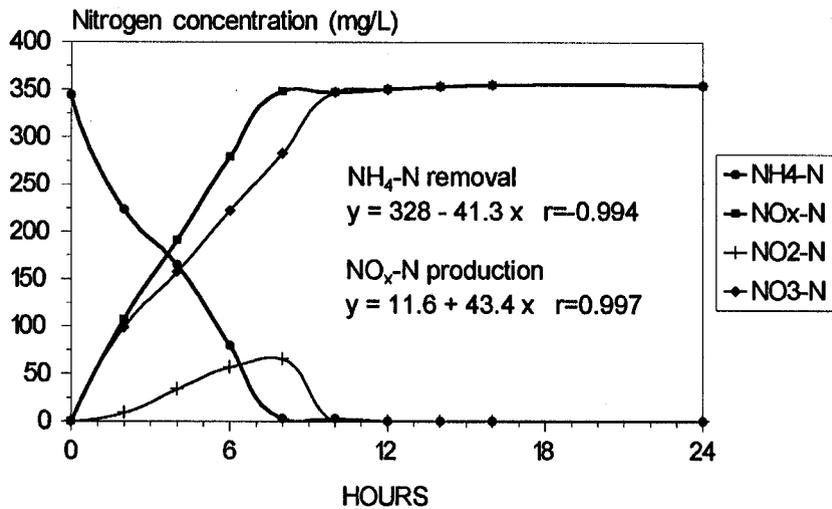


Figure 2. Nitrification of swine lagoon wastewater with nitrifying pellets in batch treatment. Process pH was controlled at 8.2-8.5 with alkali supplements. NO_x^- is NO_2^- plus NO_3^- . The nitrifiers were isolated from a nitrification tank treating lagoon wastewater. The enrichment culture was prepared in a medium containing 900 ppm $\text{NH}_4\text{-N}$; the high-ammonia nitrifying bacteria (HANB) cells were then entrapped in PVA polymer pellets.

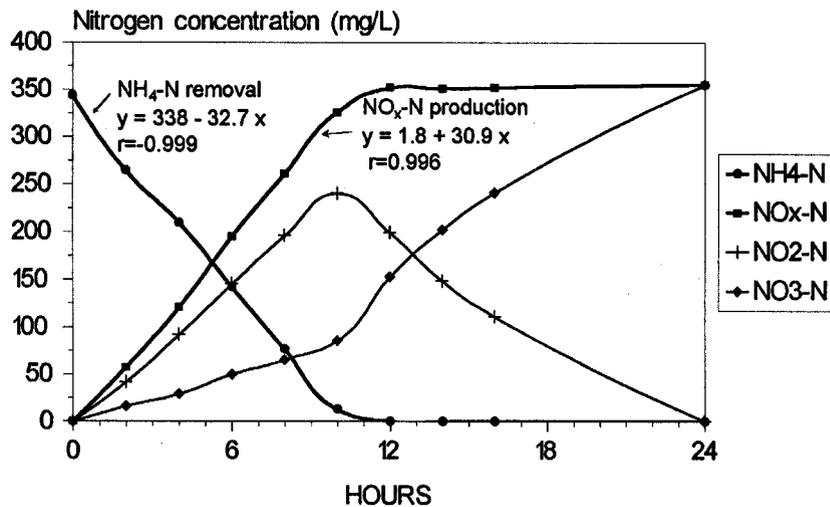


Figure 3. Nitrification of swine lagoon wastewater with encapsulated nitrifiers where NO_2^- -oxidizing bacteria (*Nitrobacter* sp) are inhibited to high-ammonia wastewater. Process pH was controlled at 8.2-8.5 with alkali supplements. NO_x^- is NO_2^- plus NO_3^- . The nitrifiers were isolated from an overland flow sludge. The bacteria culture was prepared in a medium containing 300 ppm $\text{NH}_4\text{-N}$ and subsequently entrapped in PVA polymer pellets.

linearly at a rate of 41.3 mg NH₄⁺-N/L-reactor/d. The NH₄⁺ was completely removed in only 8 h of aeration treatment. It was transformed into oxidized N forms without losses of NH₃ by volatilization; full conversion of NO₂⁻ to NO₃⁻ was completed in 10 h. The batch experiments also included separate runs (Fig. 3) using pellets with nitrifying cells that were cultivated in a growth medium containing 300 mg NH₄⁺ N/L. Results indicate that NO₂⁻ oxidation using these pellets was severely inhibited when ammonia was present in the system (0 to 10 h), resulting in a large accumulation of nitrite. Thus, total time needed for full conversion of NH₄⁺ to NO₃⁻ was about double the time needed for NH₄⁺ disappearance. In contrast, high NO₂⁻ concentration was not detected using the HANB pellets (Fig. 2). This indicates that NO₂⁻ oxidizing bacteria in the newly developed pellets were not inhibited by high-ammonia levels and operated simultaneously with NH₄⁺ oxidizers.

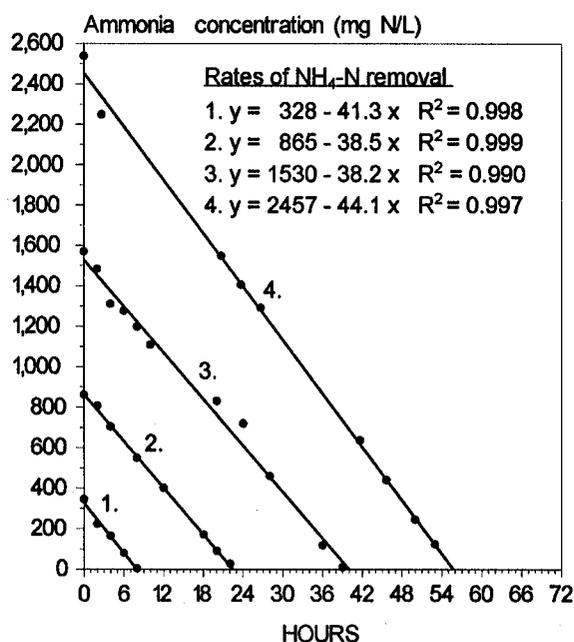


Figure 4. Ammonia removal from swine wastewater via biological treatment with polymer encapsulated nitrifiers. Process pH was controlled at 8.2-8.5 with alkali supplements. Each line is a separate batch experiment with different initial NH₄-N concentration; data for exp. 1 are also shown in Fig. 1.

Data in Fig. 4 show rates of ammonia removal for HANB pellets in different batch runs using varying NH₄⁺ strength wastewater. Ammonia was readily oxidized in all cases, decreasing linearly with time. Similar NH₄⁺ removal rates (38 to 41 mg-N/L-reactor/d) were obtained for initial NH₄⁺ N concentration in the range of 300 to 1600 mg/L. Corresponding nitrification efficiencies were >97% (Table 1). Ammonia removal rate was higher (44 mg-N/L-reactor/d) with the 2540-mg N/L wastewater. However, 15% of the N was unaccounted for (85% efficiency) indicating that the increased NH₄⁺ removal rate was due to some losses by volatilization and not due to increased biological activity. Nitrite oxidizers in the HANB pellets were not inhibited by NH₄⁺ concentration up to the maximum level of 2600 mg N/L that was

tested (data not shown). Both NH_4^+ and NO_2^- oxidation operated simultaneously similar to performance at ~ 350 mg N/L shown in Fig. 2.

Table 1. Batch treatment of high-ammonia animal wastewater with nitrifying pellets

Initial $\text{NH}_4\text{-N}$ concentration†	Ammonia removal rate‡	Ammonia disappearance §	Final nitrate + nitrite concentration	Nitrification efficiency ¶
mg N/L	mg N/L reactor/h	hours	mg N/L	%
344	41.3	8	348	100
860	38.5	22	855	99
1570	38.2	40	1525	97
2540	44.1	56	2152	85

† Initial NO_3^- and NO_2^- concentration = 0

‡ Ammonia removal rates calculated from slope of lines in Figure 4

§ Conc. $\text{NH}_4^+ = 0$

¶ Nitrification efficiency = [(final $\text{NO}_3\text{-N} + \text{NO}_2\text{-N}$ conc.)/initial $\text{NH}_4\text{-N}$ conc.] $\times 100$

CONTINUOUS FLOW NITRIFICATION EXPERIMENTS

Treatment Using a Single-Stage Reactor

Ammonia removal potential of HANB pellets was also evaluated under continuous flow. In an initial experiment, we evaluated the nitrification performance of a single-stage reactor. The evaluation consisted of consecutive runs with varying NH_4^+ loading rates (Table 2) using two wastewater treatments (~ 900 and 1600 mg $\text{NH}_4^+\text{N/L}$). Loading rates were adjusted by changing the influent flow rate so that nitrification efficiency could be compared at similar NH_4^+ loading value across influent strength types. The process pH was controlled at 7.5. Nitrification efficiencies of more than 90% were obtained at NH_4^+ loading rates from 623 to 782 mg-N/L-reactor/d. In all cases, NH_4^+ N was entirely recovered in NO_3^- and NO_2^- forms. This is shown in Table 2 by comparing values of NO_3^- plus NO_2^- production vs. NH_4^+ removal rates. In general, NH_4^+ loading rates of ~ 1000 mg-N/L-reactor/d maximized both NH_4^+ removal and NO_x^- production and provided nitrification efficiencies $> 80\%$. Higher NH_4^+ loading decreased performance. Thus, loading rates of 1000 mg-N/L-reactor/d are considered the maximum recommended N loading for a single-stage suspended reactor using nitrifying pellets with these acclimated bacteria.

Table 2. Continuous flow treatment of swine wastewater with nitrifying pellets using a single-stage suspended reactor and a process pH of 7.5.

Influent NH ₄ ⁺ conc.	HRT †	NH ₄ ⁺ loading rate	NH ₄ ⁺ removal rate‡	NO ₃ ⁻ plus NO ₂ ⁻ production rate§	Nitrification efficiency¶
mg N/L	hours	-----mg N/L-reactor volume/day-----			%
933	72	311	311	312	100
930	48	465	465	476	100
935	36	623	620	638	100
939	24	939	755	785	84
936	16	1403	666	705	55
932	12	1864	547	657	37
1576	96	394	393	405	100
1530	72	510	507	553	100
1564	48	782	747	757	97
1577	32	1183	864	987	83
1588	24	1588	811	957	60
1570	16	2355	631	632	27

† Hydraulic residence time = (v/Q) × 24; v=reactor volume (1.2 L); Q = influent flow rate.

‡ NH₄⁺ removal rate = Q (ΔNH₄⁺)/v

§ NO_x-N production rate = Q (ΔNO_x⁻)/v; influent contained 0 mg L⁻¹ of NO₃⁻ and NO₂⁻.

¶ Nitrification efficiency = (NO_x⁻ production rate/NH₄⁺ loading rate) × 100.

Treatment Using Three Reactors in Series

In a second experiment, we evaluated nitrification performance using three reactors in series where the effluent of one reactor was gravity fed into a subsequent reactor. Ammonia strength of the wastewater was ~1600 mg N/L and process pH was also controlled at 7.5. Table 3 presents the results of this system. The nitrification performance using three reactors in series was comparable to those obtained with a single reactor. For example, nitrification efficiencies >90% and NO_x⁻ production rates of ~760 mg-N/L-reactor/d were obtained in both systems with HRT of 48 h and influent NH₄⁺ concentration of about 1600 mg N/L. These results indicate that multiple reactors do not improve nitrification efficiency of HANB pellets and that a single reactor is preferred because of simplicity.

Table 3. Continuous flow treatment of swine wastewater with nitrifying pellets using three reactors in series and a process pH of 7.5.

Influent NH ₄ ⁺ conc.	HRT †	NH ₄ ⁺ loading rate	NH ₄ ⁺ removal rate‡	NO ₃ ⁻ plus NO ₂ ⁻ production rate§	Nitrification efficiency¶
mg N/L	hours	-----mg N/L-reactor volume/day-----			%
1712	144	285	284	362	100
1751	72	584	566	645	100
1658	48	829	738	759	92
1606	32	1205	658	733	61
1608	24	1608	716	886	55
1609	16	2413	612	645	27

† Hydraulic residence time = (v/Q) × 24; v=total reactor volume (3.6 L); Q = influent flow rate.

‡ NH₄⁺ removal rate = Q (ΔNH₄⁺)/v

§ NO_x-N production rate = Q (ΔNO_x⁻)/v; influent contained 0 mg L⁻¹ of NO₃⁻ and NO₂⁻.

¶ Nitrification efficiency = (NO_x⁻ production rate/NH₄⁺ loading rate) × 100.

Effect of pH on Treatment Efficacy of Nitrifying Pellets

Control of pH is an important consideration for optimum nitrification of swine wastewater. During nitrification, there is a release of hydrogen ions, at a rate of 2 mol for each mole of NH₄⁺ oxidized, that decreases the pH to an extent related to the buffering capacity of the system. The alkalinity concentration in the wastewater used in these experiments (1920 mg CaCO₃/L) was sufficient for nitrification of 270 mg NH₄⁺ N/L, considering an alkalinity consumption ratio of 7.14 mg CaCO₃ per mg NH₄-N oxidized. Therefore, biological removal of NH₄⁺ above 270 mg/L needed alkalinity corrections. As in all other experiments, the process pH was regulated using pH controllers and pumps that injected NaOH solution and kept the pH within a 0.2-unit band. In this particular experiment, we evaluated the effect of process pH on nitrification efficiency using two reactors in series with a fixed HRT of 24 h. The process pH was regulated separately in each reactor with pH values between 6.5 and 8.5 (Table 4). Results showed that the optimum nitrification treatment was obtained when the process pH was maintained at 8.5 in both reactors. At this pH, the NH₄⁺ removal rate obtained was 816 mg N/L-reactor/d, and the nitrification efficiency was 100%. In addition, NO₂⁻ accumulation was low, with 90% of the N recovered in NO₃⁻ forms. Using lower pH set points resulted in lower efficiencies, whether this was done in the first or second reactor or in both. A pH set point of 6.5 in the first stage greatly reduced NH₄⁺ removal performance. This was likely due to the inhibitory effect that free nitrous acid have on NH₄⁺ oxidizing bacteria (Anthonisen et al., 1976). At the pH of 7.7, the NH₄⁺ removal rate with the two-reactors in series (693 mg N/L-reactor/d, Table 4) was similar to that obtained using a

single-stage reactor at comparable HRT (24 h) and influent strength (Table 2). This supports our conclusion that there is no real advantage to using a plug-flow treatment mode vs. a single reactor. Our results also indicate that process pH is a much more important consideration for optimum nitrification of high-ammonia wastewater and that nitrification efficiencies can be halved by using lower than optimum pH values.

Table 4. Effect of pH on nitrification efficiency of swine wastewater using two reactors in series.†

Process pH		Influent NH ₄ ⁺ conc.‡	Effluent quality			NH ₄ ⁺ removal rate §	Nitrification efficiency ¶
First reactor	Second reactor		NH ₄ ⁺	NO ₃ ⁻ plus NO ₂ ⁻	NO ₂ ⁻		
-----mg N /L-----						mg N/L- reactor/d	%
6.5	6.5	837	406	435	234	431	52
7.5	7.5	830	137	706	361	693	85
8.5	8.5	855	39	856	91	816	100
6.5	8.5	825	445	411	222	380	50
8.5	6.5	843	249	665	76	594	79

† Influent flow rate (Q) = 2.4 L/day; Total HRT = 24 hours

‡ Lagoon influent contained 0 mg L⁻¹ of NO₃⁻ and NO₂⁻

§ NH₄⁺ removal rate = Q (ΔNH₄⁺)/v; v = volume of two reactors in series (2.4 L)

¶ Nitrification efficiency = [(effluent NO₃⁻ plus NO₂⁻) / influent NH₄⁺] × 100

CONCLUSIONS

The use of large populations of nitrifying bacteria entrapped in polymer resins offers the potential for NH₄⁺ removal rates that are much faster than those occurring in conventional waste treatment systems based on activated sludge. This technology has been successfully used for treating municipal wastewaters that have lower concentrations of nitrogen and organic carbon compared to liquid animal manure. However, there was a concern that the high NH₄⁺ strength typical of liquid animal manure may be harmful to the immobilized bacteria. Our approach was to use immobilized pellet technology with nitrifying bacteria acclimated to high-ammonia wastewater. The enrichment culture was prepared in a medium containing 900 ppm NH₄-N; the high-ammonia nitrifying bacteria (HANB) cells developed were successfully immobilized in polymer pellets using a PVA-freezing method.

Our results showed that immobilized HANB can rapidly remove NH₄⁺ from high-ammonia animal wastewater containing 350 to 2600 mg N/L. Ammonia removal rates from 38 to 41 mg-N/L-reactor/h (915 to 990 mg-N/L-reactor/day) and 97 to 100% nitrification efficiency were obtained in batch treatment of these wastes. In continuous flow, nitrification efficiencies > 80% were obtained with NH₄⁺ loading rates < 1000 mg-N/L-reactor/d and HRT of 24 to 48 h. All of the NH₄⁺ N removed was entirely recovered in oxidized N forms. A

comparison of a single reactor vs. two or three reactors in series showed that there is no improvement in nitrification performance of high-ammonia wastewater using the plug-flow regime. Our results indicate that process pH is a much more important consideration for optimization of the nitrification process of high-ammonia wastewaters and that improper selection may result in 50% decrease in performance. Optimum nitrification performance (100% efficiency) was obtained when the pH was controlled at 8.5. Our results also showed that immobilized HANB were not inhibited by high-ammonia strength wastewater in the range of 350 to 2600 mg N/L that was evaluated and that the NH_4^+ and NO_2^- oxidizing bacteria entrapped in these pellets operated simultaneously providing for fast and complete nitrification of the NH_4^+ contained in these wastes.

The results of this study indicate that the immobilized pellets are a useful alternative method for the nitrification treatment of high-ammonia animal wastewaters and that this technology can be adapted for fast and efficient removal of NH_4^+ removal by using acclimated microorganisms.

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