Dairy Manure Treatment, Digestion and Nutrient Recovery as a Phosphate Fertilizer

ASIF QURESHI [a], KWANG VICTOR LO [a], DONALD S. MAVINIC [a], PING HUANG LIAO [a], FREDERICH KOCH [a] & HARLAN KELLY [b]

[a] Department of Civil Engineering, University of British Columbia, Vancouver, Canada

[b] Dayton and Knight Ltd., North Vancouver, Canada
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Asif Qureshi,[1] Kwang Victor Lo,[1] Donald S. Mavinic,[1]
Ping Huang Liao,[1] Frederich Koch,[1] and Harlan Kelly[2]

[1]Department of Civil Engineering, University of British Columbia, Vancouver, Canada
[2]Dayton and Knight Ltd., North Vancouver, Canada

A combined approach of biological treatment, solids digestion and nutrient recovery was tested on dairy manure. A sequencing batch reactor (SBR) was operated in three modes, in order to optimize nutrient (nitrogen and phosphorus) removals. The highest average removal efficiencies of 91% for NH_4-N, 59% for PO_4-P and 80% for total chemical oxygen demand (COD) were achieved. Staining experiments suggested the coexistence of glycogen and phosphorus accumulating organisms. Anaerobic digestion of wasted bio-solids was able to produce a PO_4-P concentration of 70 mgL^{-1} in the supernatant. A pilot-scale experiment, designed to recover phosphorus in the supernatant as struvite (magnesium ammonium phosphate), was able to remove 82% of soluble PO_4-P.

Key Words: Dairy manure treatment; Sequencing batch reactor; Anaerobic digestion; Phosphorus recovery; Struvite.

INTRODUCTION

Research in animal manure management is very important. It affords us an opportunity to avoid the harmful impacts that animal manure can have on the environment, as well as advantageously use the nutrients present in them. Currently, land spreading is the most common means to manage animal manure. However, it has led to issues of groundwater and surface water contamination, and of odor emissions.[1–6] The amount of available land is also

limited and society may eventually face an acute shortage of suitable land for ultimate disposal.

As an alternative or aid to land application, manure treatment can help to reduce the environmental loading and recover the nutrients released from the biomass in a concentrated form. Such an approach has already been tested and established for treating municipal wastewaters, where centrates of digested sludges were processed for recovery of phosphorus as struvite (magnesium ammonium phosphate).[7–10] To the knowledge of the authors of this paper, no one has taken such a combined approach for manure treatment. Other studies have, in general, addressed either treatment alone, on swine manure,[11–14] and on dairy manure,[15,16] or digestion alone,[17,18] or phosphorus recovery directly from digested manure.[19–21]

The studies on treatment have primarily used sequencing batch reactors (SBR) for treatment, due to their many advantages; namely reduced space requirements and the ability to be designed specifically for individual sources. Also, most of the published studies on SBR treatment of agricultural manure are on swine wastewater and a paucity of literature is available with regard to dairy manure. With a view to develop a comprehensive approach of SBR treatment of dairy manure with optimized nutrient removal, solids digestion and phosphorus recovery, the current study was carried out and preliminary results are reported herein. This on-going study is part of a much larger research program in liquid waste management and nutrient recovery, being undertaken at the University of British Columbia (UBC), Vancouver, British Columbia, Canada.

MATERIALS AND METHODS

Fresh dairy manure was collected from a dairy farm in Agassiz, British Columbia, Canada and stored at 4°C, until used. The feed for SBR was prepared by diluting the manure ten times with tap water and then filtering it through a 2 mm sieve. The SBR was operated in different anoxic/aerobic modes (Table 1), with a view to achieving enhanced biological phosphorus removal. The anoxic feed and settling durations were kept constant at 30 minutes and

Table 1: Description of various modes of operation (presented chronologically).

Mode	Anoxic length	Aerobic length	Total cycle length	Corresponding hydraulic retention times	Duration studied
A	3 hours	7.5 hours	12 hours	2.5 days	129 days
B	5 hours	9.5 hours	16 hours	3.3 days	40 days
C	5 hours	7.5 hours	14 hours	2.9 days	60 days

85 minutes, respectively. Solids retention time (SRT) was kept at 20 days, and the mixed liquor suspended solids (MLSS) concentration was 8122 ± 1051 mg/L during the study. The mixed liquor volatile suspended solids (MLVSS) concentration averaged 6439 ± 956 mgL^{-1} (MLVSS/MLSS = 0.79 ± 0.04). All the modes were tested over a period at least double the SRT.

SBR influent and effluent were analyzed for chemical oxygen demand (COD), total organic carbon (TOC), ortho-phosphates (PO_4-P), ammonia nitrogen (NH_4-N), nitrite and nitrate nitrogen (NO_x-N), total phosphorus (TP) and total Kjeldahl nitrogen (TKN). Each day, 500 mL of wasted solids from the SBR were fed to a 12-liter anaerobic digester operated at room temperature (18–22°C) and 40 days SRT. The contents were suspended by mixing at about 50 rpm. Initially, it was observed visually that the contents did not settle well in 3–4 hours of settling; subsequently, a different philosophy was adopted in which sludge was added everyday but decanting (and wasting from digester) was undertaken every three days, with a settling duration of 12 to 15 hours. The supernatant was analyzed for PO_4-P and NH_4-N.

For the nutrient recovery experiments, a struvite crystallizer (developed by researchers at UBC in 1999) was used. The crystallizer (a patented design) shown in Figure 1, acts as a fluidized bed reactor, where precipitation is accomplished by adding magnesium and caustic to the up-flowing supernatant
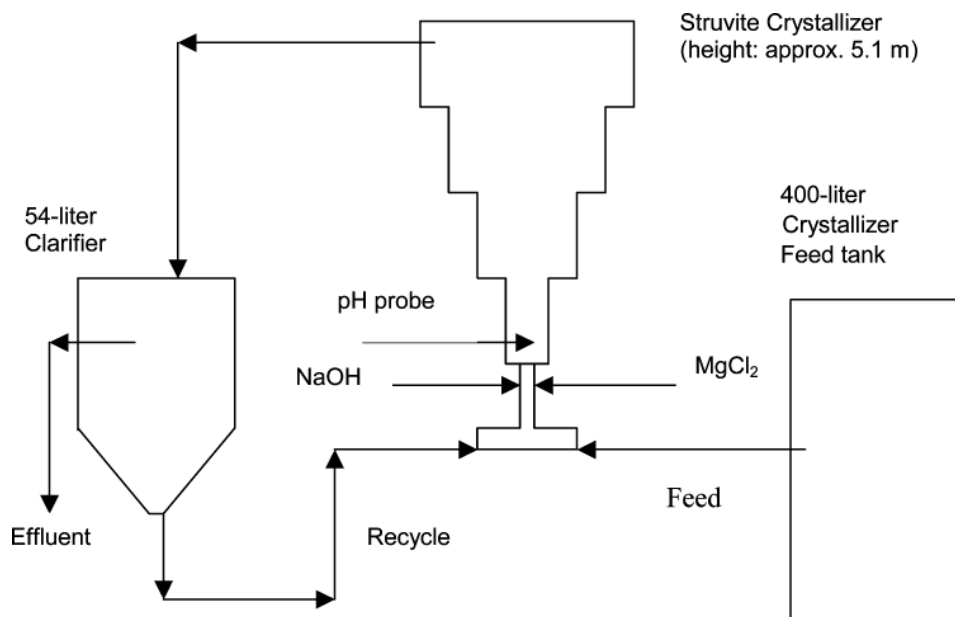

Figure 1: . Schematics of the P-release reactor used to recover phosphorus from wasted solids.
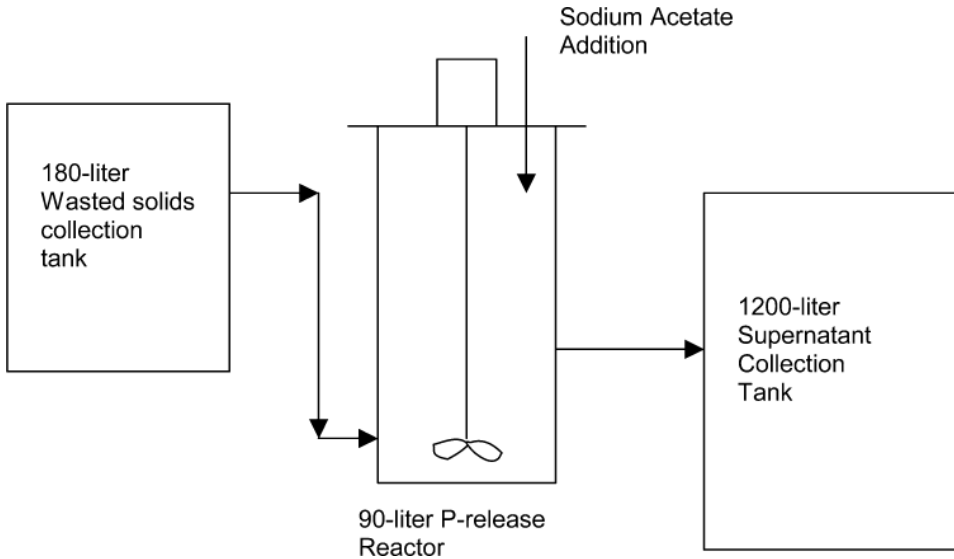
Figure 2: . Basic flow diagram of the University of British Columbia (UBC) pilot-scale crystallizer.

(ammonia is also added if required). Since the volume of supernatant obtained by lab scale digestion was not enough to run the pilot scale crystallizer, biosolids wasted from a pilot scale SBR treating diluted dairy manure were used. Every two days, 75 liter of these biosolids were subjected to the addition of 1000 mgL^{-1} of sodium acetate, in a 90-liter capacity P-release reactor (Fig. 2), and mixed intermittently for 5 minutes, every 1 hour 15 minutes. Every 2 days, after a settling period of 15–18 hours 40–45 liter of supernatant was then withdrawn and collected. The remaining liquid was discharged to the drain. After about 400 liter of supernatant was collected, 200 liter of it was subjected to a "struvite recovery" run in the crystallizer. The theoretical and operational principles of running the crystallizer have been reported elsewhere.[22–24] The performance of the crystallizer was evaluated from the influent and effluent PO$_4$-P values, only.

Analytical Methods

COD, TSS, TP and TKN were analyzed by methods prescribed in Standard Methods.[25] PO$_4$-P, NO$_x$-N, NH$_4$-N were analyzed using an automated ion analyzer (Lachat QuickChem series 8000, Zellweger Analytics, Inc.), after filtration through 0.45 μm membrane filters. All soluble parameters in this paper refer to values obtained after filtration through this 0.45 μm filter. TOC analyses were done using a Total Carbon Analyzer (Type TOC-500, Shimadzu Corporation). All analyses, other than COD (unless otherwise stated) and

suspended solids, were conducted on soluble samples. Staining experiments to identify bacteria were conducted according to Jenkins et al.[26]

RESULTS AND DISCUSSIONS

SBR Treatment

A comparison of the SBR performance, in the three modes, is shown in Tables 2, 3 and 4. The increase in anoxic duration allowed for more organic particulates to become soluble in modes B and C, compared to mode A. This solubilisation may explain the differences in soluble COD (s-COD) removals in the three modes. For example, between modes A and C, the only difference is a greater anoxic length in mode C, while the same length of aerobic time reduced more s-COD in mode A ($61.8 \pm 16.4\%$) than in mode C ($42.9 \pm 11.0\%$). Mode B had a longer aeration period and allowed for more s-COD removal than the other two modes. A high portion of total COD (t-COD) was in insoluble form.

NH_4-N values in mode A indicated incomplete nitrification. To achieve additional nitrogen removal, mode B was subjected to a longer aeration time. Consequently, NH_4-N removal efficiencies averaged 91.3% and effluent NH_4-N <2 mgL^{-1} (efficiency $\sim$99%) was observed on numerous occasions. It was observed that higher nitrogen removal did not resulted in higher effluent NO_x-N concentration, the values being even lower than mode A (which had incomplete nitrification). This may have been due to the occurrence of simultaneous nitrification and denitrification in the SBR, a known characteristic of this system.

Mode B also resulted in the highest amount of PO_4-P removal, with an average removal efficiency of $58.9 \pm 14.3\%$, compared to $48.9 \pm 23.1\%$ in mode A and $45.1 \pm 19.1\%$ in mode C. It was recognized that, since absolute PO_4-P removals were quite high (22.4 ± 14.6 mgL^{-1}) and that the bacteria uptake phosphorus by mass and not percentage, enhanced phosphorus uptake

Table 2: Summary of results during mode A.*

Parameter	Influent (mg/L)	Effluent (mg/L)	Removal per cycle (mg/L)	Removal efficiency (%)
t-COD	—	—	—	—
s-COD	2384 ± 503	840 ± 291	1537 ± 529	61.8 ± 16.4
TOC	718 ± 404	298 ± 129	433 ± 336	54.2 ± 19.8
NH4-N	129 ± 47.5	46.6 ± 45.2	82.2 ± 49.0	65.3 ± 25.8
PO4-P	37 ± 13.8	16.6 ± 4.5	20.1 ± 14.5	48.9 ± 23.1
NOx-N	1.1 ± 1.5	8.2 ± 10.4	—	—
TP	38.1 ± 19.8	17.3 ± 6.7	20.8 ± 17.4	45.5 ± 27.4
TKN	145 ± 60	60 ± 50	83 ± 51	59.2 ± 26.1

*± values represent one standard deviation.

Table 3: Summary of results during mode B.*

Parameter	Influent (mg/L)	Effluent (mg/L)	Removal per cycle (mg/L)	Removal efficiency (%)
t-COD	6334 ± 2658	1180 ± 480	5154 ± 2476	80.2 ± 5.8
s-COD	2124 ± 472	668 ± 117	1457 ± 437	67.8 ± 6.0
TOC	511 ± 103	210 ± 59	300 ± 110	57.5 ± 14.0
NH4-N	103 ± 23.4	9.0 ± 8.6	93.7 ± 2.1	91.3 ± 7.6
PO4-P	35.4 ± 14.3	13.0 ± 2.9	22.4 ± 14.6	58.9 ± 14.3
NOx-N	1.1 ± 0.4	3.8 ± 2.4	—	—
TP	51.1 ± 23.0	19.8 ± 7.0	31.3 ± 19.1	58.4 ± 14.3
TKN	155 ± 35	42 ± 26.5	113 ± 46.5	70.5 ± 23.5

*± values represent one standard deviation.

may have occasionally taken place, even though the removal efficiencies were not in the order or 90% or more. In the last full week of mode B, the mixed liquor from the SBR was tested for percent phosphorus in the sludge (samples taken at the end of aerobic phase). The % P per MLVSS was found to be 2.60 ± 0.56%, a value marginally higher than the normally assumed value in non-enhanced biological phosphorus removal (non-EBPR) sludges but lower than the normally reported% P values in municipal EBPR sludges (3–5%).

For mode C, the aerobic length was reduced, in an attempt to reduce the amount of NO_x-N going to the anoxic stage of the next cycle. That is, nitrification efficiency was compromised in an attempt to achieve higher phosphorus removal. However, it was observed that absolute PO_4-P removal actually decreased to 16.6 ± 9.6 mgL^{-1}. The % P per MLVSS in sludge was only 1.55 ± 0.17% for the last full week in mode C. This indicated that some part of the phosphorus uptake sequence in mode B was lost in mode C. Although the sampling for % P was not extensive, the results do suggest

Table 4: Summary of results during mode C.*

Parameter	Influent (mg/L)	Effluent (mg/L)	Removal per cycle (mg/L)	Removal efficiency (%)
t-COD	12575 ± 8206	3276 ± 742	9300 ± 7983	67.3 ± 14.0
s-COD	2236 ± 457	1281 ± 313	972 ± 358	42.9 ± 11.0
TOC	427 ± 105	275 ± 76	155 ± 106	33.4 ± 21.6
NH_4-N	123 ± 25.8	93.3 ± 24.1	29.7 ± 22.9	23.3 ± 17.1
PO_4-P	34.3 ± 7.9	17.6 ± 4.1	16.6 ± 9.6	45.1 ± 19.1
NOx-N	1.1 ± 0.8	1.3 ± 2.3	—	—
TP	44.1 ± 9.7	24.0 ± 9.2	20.2 ± 9.1	45.4 ± 17.9
TKN	248 ± 87	171 ± 59.6	75.0 ± 55.0	28.1 ± 17.9

*± values represent one standard deviation.

that EBPR is possible in dairy wastewater treatment, under appropriate cycle times.

A comparison of the track-study profiles (Figs. 3, 4 and 5) also shows that the P release in the anoxic stage of the cycle was about 15 mgL^{-1}, in mode B, after considering the effect of influent mixing, whereas it was only about 3.3 mgL^{-1} in mode A, and was absent in mode C. The uptake in the aerobic cycle of mode B was incomplete, suggesting only a partial dominance of phosphorus-accumulating organisms, which is consistent with the % P observations. Simultaneous observations of NH_4-N and NO_x-N profiles in Figures 3 and 4 show evidence of occurrence of simultaneous nitrification and denitrification.

Photo micrographs obtained after poly-β-hydroxyalkanoate (PHA) staining (Fig. 6) indicated the uptake of volatile fatty acids (VFA) as PHA granules. These micrographs were made on day 20 of mode C. The observation of coccoid-shaped bacteria staining positively on cell walls (Fig. 7), as well as bacteria staining positively in intracellular spaces after staining with Neisser's Reagent (Fig. 8), indicates the coexistence of glycogen-accumulating organisms and phosphorus-accumulating organisms in the mixed liquor. The relative
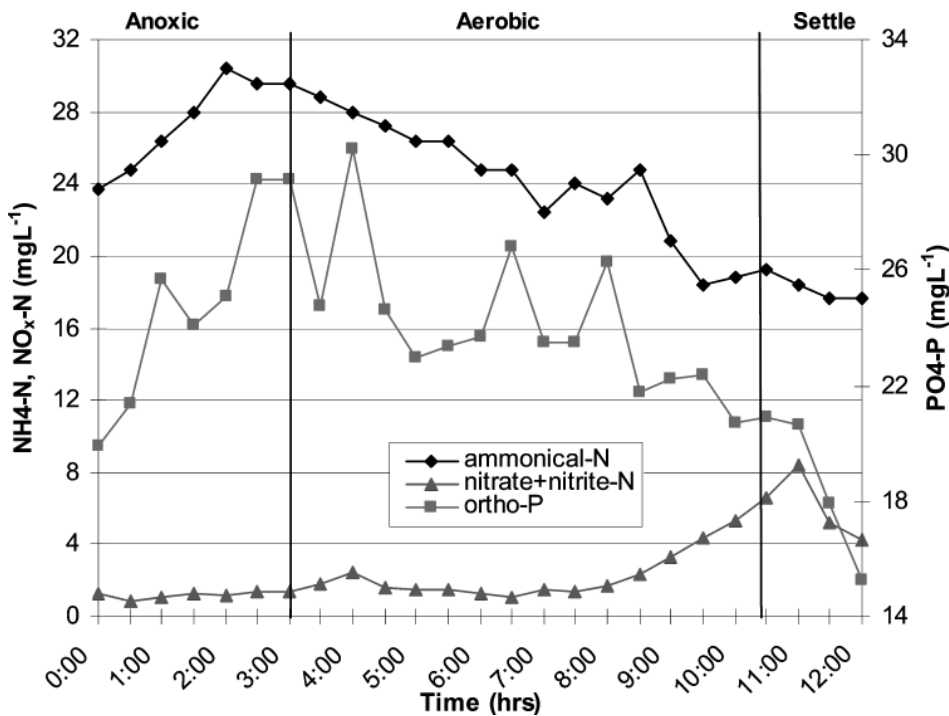

Figure 3: Mode A NH_4-N, NO_X-N and PO_4-P track study profiles (Mixed liquor concentrations expected after influent addition: NH_4-N $= 38.9$ mgL^{-1}, NO_x-N $= 1.68$ mgL^{-1} and PO_4-P $= 25.8$ mgL^{-1}; note the scales).
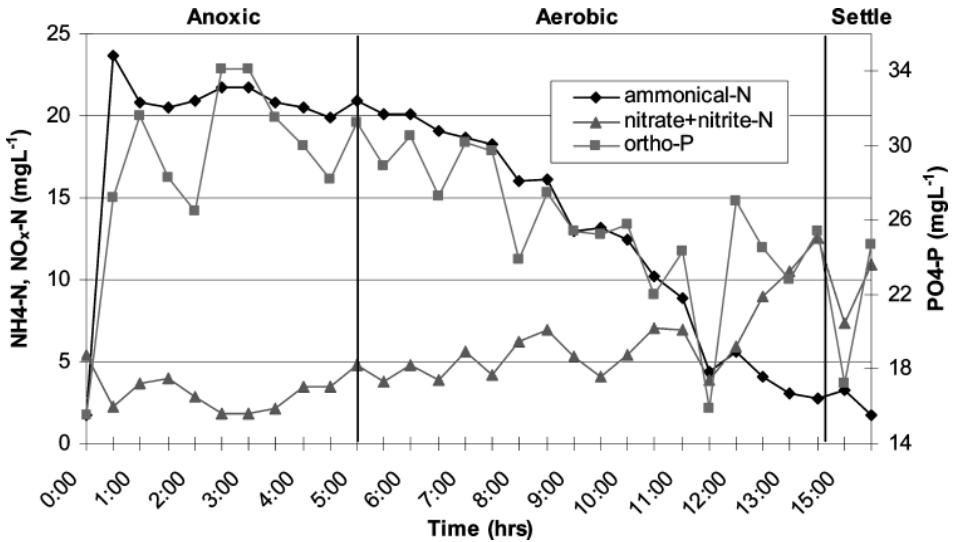
Figure 4: Mode B NH_4-N, NO_X-N and PO_4-P track study profiles (Mixed liquor concentrations expected after influent addition: NH_4-N = 21.2 mgL^{-1}, NO_x-N = 4.9 mgL^{-1} and PO_4-P = 19.1 mgL^{-1}; note the difference in scales).

activities of these two classes of organisms may have led to the different extents of PO_4-P removals in the three modes. Since the micrographs were obtained on day 20 of mode C, a steady state was not yet achieved for this mode, and the bacteria were in transition from mode B to C. This line of research is

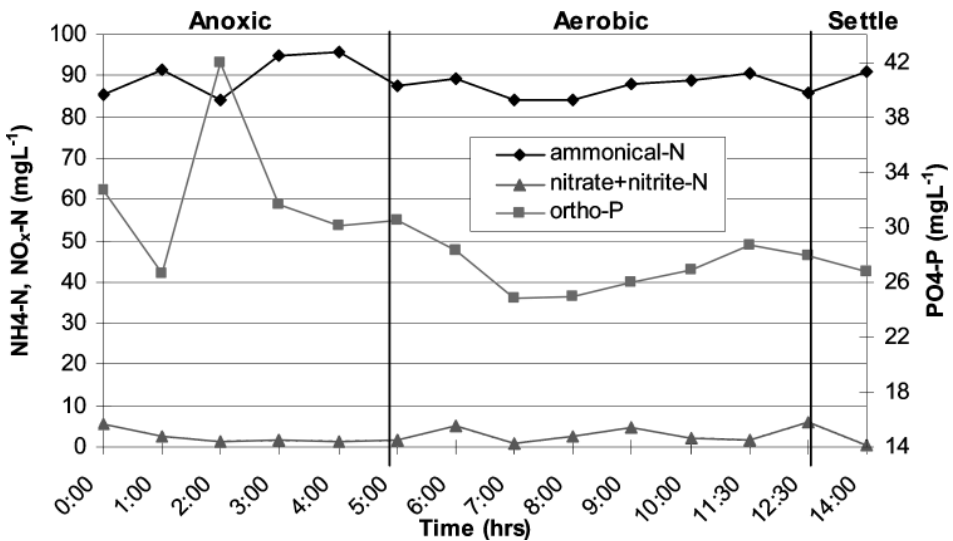**Figure 5:** Mode C NH_4-N, NO_X-N and PO_4-P track study profiles (Mixed liquor concentrations expected after influent addition: NH_4-N = 91.4 mgL^{-1}, NO_x-N = 4.9 mgL^{-1} and PO_4-P = 32.8 mgL^{-1}; note the difference in scales).
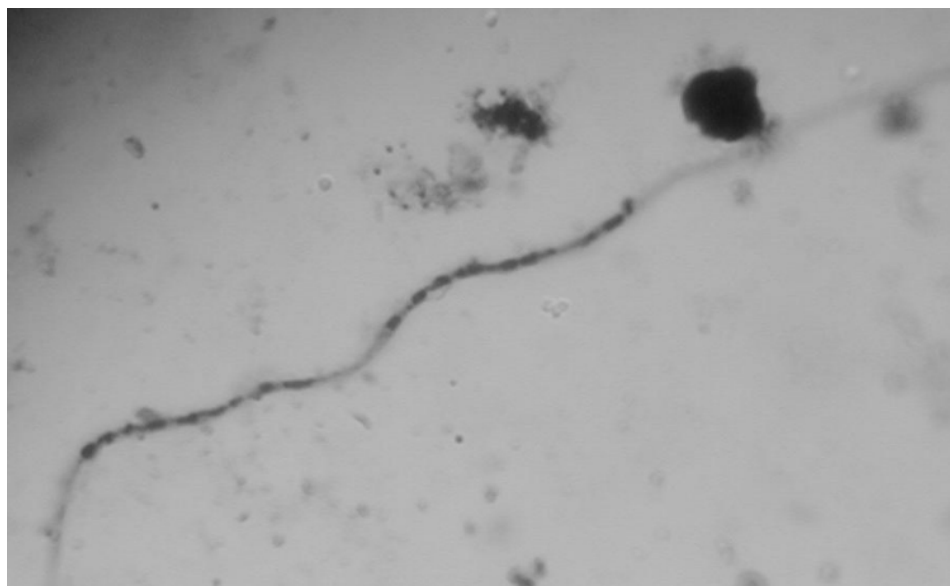
Figure 6: Results of poly-β-hydroxyalkanoate (PHA) staining for intracellular PHA granules.

part of on-going studies on dairy manure treatment, in the Environmental Engineering lab, UBC.

Anaerobic Digestion

The PO_4-P concentration in the digester supernatant increased gradually during the first 90 days of the study period, and then stabilized at around 70 mgL^{-1} (Fig. 9). The relatively low values in the initial phase were attributed to the startup phase of the digester. The higher values in the later stages indicate

Figure 7: Coccoid shape bacteria seen after Neisser staining.
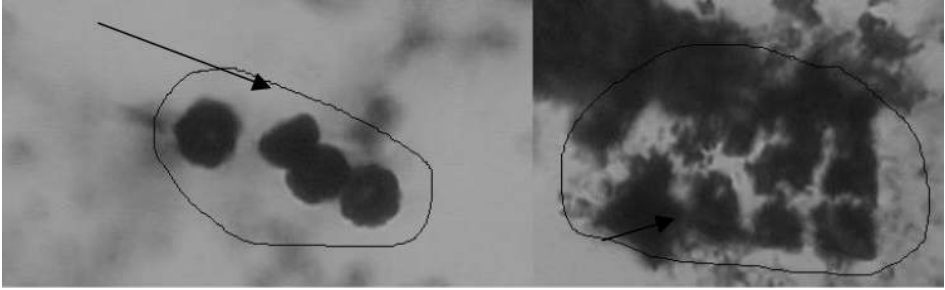
Figure 8: Bacteria showing positive Neisser staining.

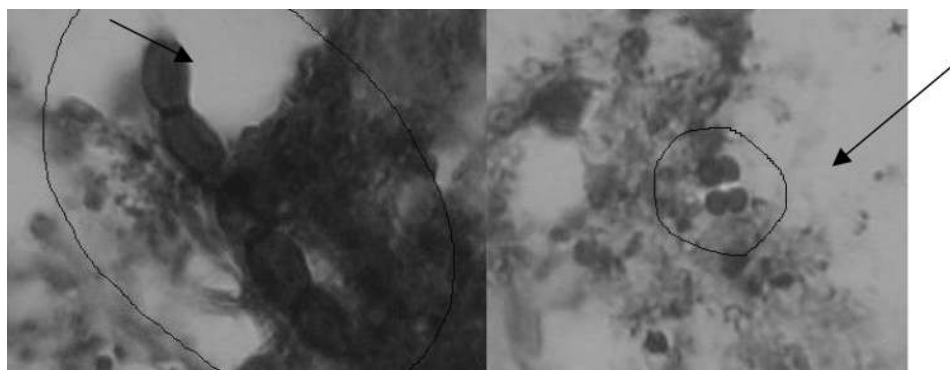proper functioning of the digestion system. NH_4-N values did not show any significant trend and varied consistently between 200 and 300 mgL^{-1}.

Since the supernatant PO_4-P values were on the higher side of the range previously tested by the struvite crystallizer at UBC,[22–24] it was assumed that recovery from this supernatant was also possible. Hence, a preliminary experimental run on struvite production was initiated, at a small, pilot-scale level.

Pilot Scale P-Recovery Experiment

About 200 liter of supernatant obtained from the P-release reactor (see materials and methods) was run in the pilot scale crystallizer (Fig. 1). The

Figure 9: Anaerobic digester supernatant PO_4-P and NH_4-N characteristics during the experiment.

Figure 10: Crystallizer effluent PO_4-P and NH_4-N characteristics during the experiment.

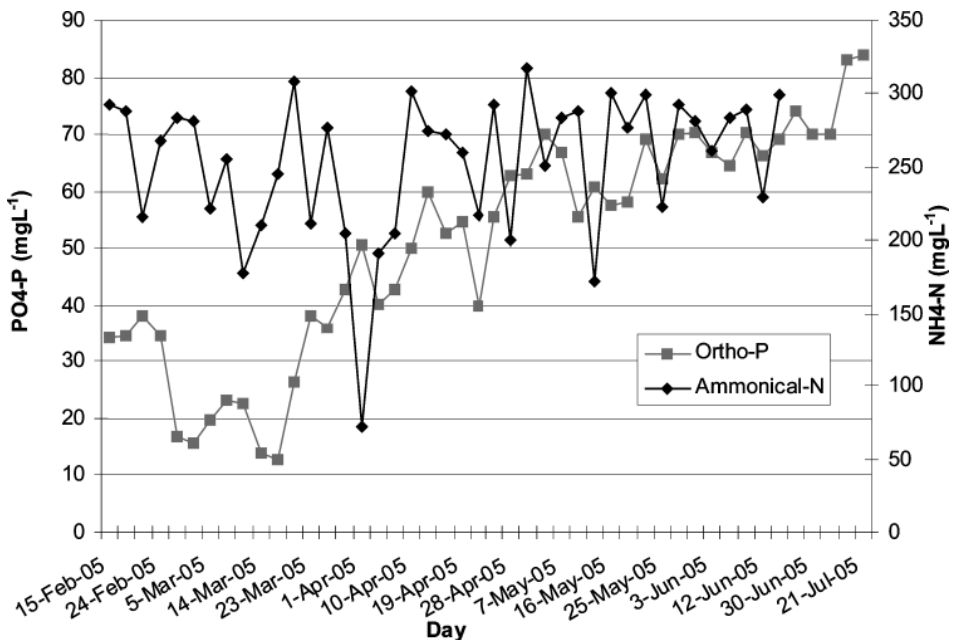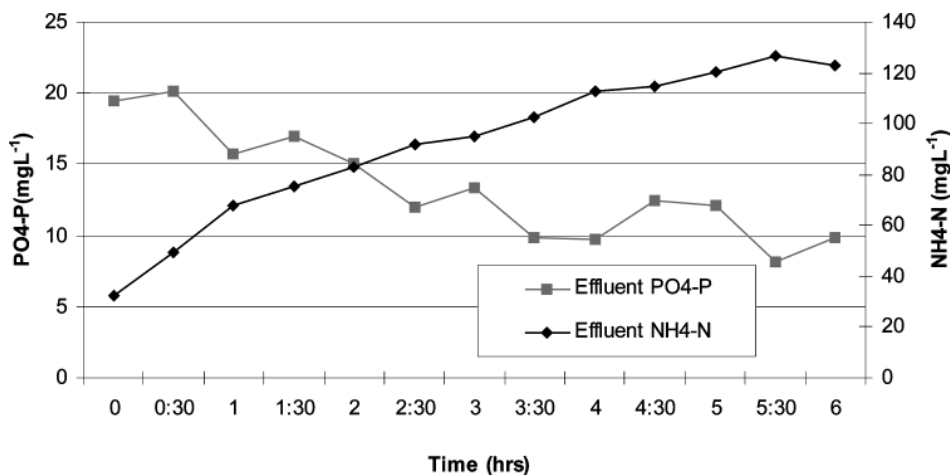pH of the supernatant was 8.04 prior to the experiment. This pH was maintained during the entire duration of experiment, using a pH controller. The crystallizer was initially filled with tap water and replaced gradually by the feed supernatant. The initial PO_4-P concentration for the experiment was 55 mgL^{-1}. The initial NH_4-N concentration was increased from 15 mgL^{-1} to 172 mgL^{-1} by the addition of the appropriate amount of ammonium chloride. The initial Magnesium concentration was 68 mgL^{-1}.

The PO_4-P concentration in the effluent started to decrease within the first half hour and stabilized to around 10 mgL^{-1}, at about the fourth hour of the experimental run (Fig. 10). Thus, a removal efficiency of 82% was achieved during the initial crystallization run. The analysis of crystals obtained after the experimental run revealed that they were in the form of fines <0.2 mm in size, on average. This size is consistently smaller than 1–3 mm size struvite crystals, normally produced in the UBC-based research program. Research is on going in this area with size optimization.

From the results obtained for the three modes, we see that different parameters, namely NH_4-N and PO_4-P were removed to different extents. It appears that there will be some particular cycle length at which the removal is optimum; and that this cycle length will be different for different types of wastewaters. Determination of an optimum cycle length using fixed-time strategies will be iterative and time consuming, since at least two SRTs will be required to give any conclusion with confidence. Also, in real-life situations, the loading to the SBR will vary and hence fixed-time strategy will not ensure optimum aeration; in other words, sometimes the mixed liquor may be under-aerated, and sometimes over-aerated. Over-aeration in an SBR has many negative effects: more nitrates and dissolved oxygen (DO) will be present

at the onset of the next anoxic/anaerobic stage, thereby hampering the activity of phosphorus-accumulating organisms. Moreover, over-aeration may lead to depletion of intra-cellular carbon reserves because of endogenous respiration by bacteria in the absence of a readily usable carbon source.

A better way to treat the wastewater of such high and variable strength will be to utilize real-time control, in which the DO and aeration lengths are controlled by electronic instrumentations and appropriate algorithms. Control of DO will ensure that the bacteria operated in SND mode; and controlling aeration length based on DO changes will ensure that the mixed liquor is not over aerated. Kim et al.[14] utilized ORP and pH real-time control for treatment of swine wastewaters, and achieved above 95% removals for carbon and nitrogen, though the TP removal efficiency was only 50%. Ra et al.[27] treated wastewater in a two-stage SBR using an ORP controlled logic and achieved >90% removals for both carbon, NH_4-N and PO_4-P. The control systems will detect the predefined set point and will control the air on/off logic accordingly. Logic can be added to keep DO always below a certain value, say 0.5 mgL^{-1}. The second part of this bigger study on dairy manure treatment studies the treatment of dairy manure using a pilot scale SBR running on a real-time control strategy and the results will be published shortly.

The supernatant from the lab-scale digester shows that for this partially treated (in terms of N and P) wastewater, the digester supernatant will contain both PO_4-P and NH_4-N, which are essential to struvite formation. Also, dairy manure is reported to be very strong in terms of metal (potassium, K; magnesium, Mg) concentration. Mg is required for formation of struvite, and K is required for formation of K-struvite ($MgKPO_4.6H_2O$). Both these forms are useful fertilizers and recovery of either or both will help in removing the nutrient load to the environment.

The struvite-recovery run gave some anomalous results for the NH_4-N profile. The NH_4-N value in the effluent increased with time, whereas the PO_4-P values remained approximately constant. It was expected that the NH_4-N profile would also remain fairly constant. For example, as in Figure 11 for a synthetic supernatant made from mixing ammonium chloride, potassium dihydrogen phosphate and magnesium chloride in tap water; initial conditions were: PO_4-P = 82 mgL^{-1}, NH_4-N = 210 mgL^{-1}, Mg = 181 mgL^{-1}. One possible explanation is the replacement of NH_3 from the crystal by K. Dairy wastewaters are quite high in potassium levels, and it is possible that potassium started to take the place of NH_3 in the crystals. We are now experimenting to confirm this finding by another run in crystallizer, as well as by jar tests. Such a phenomenon, if true, will open another vast area of research wherein K-struvite, along with struvite will provide an ideal mix of N-P-K fertilizers for the farmers.

In short, this particular research has raised questions on the treatment philosophies of high-strength wastewaters such as dairy manure, and on the
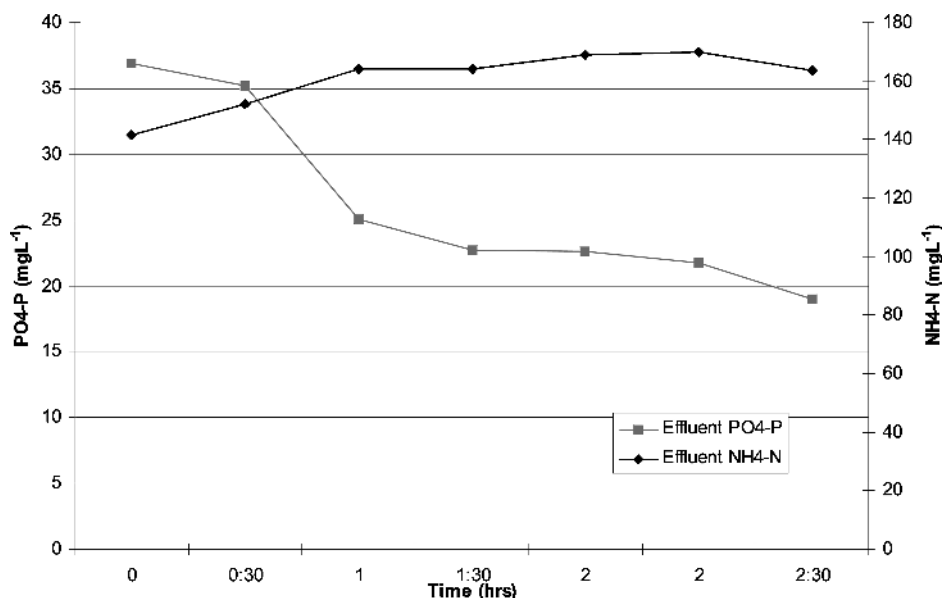
Figure 11: Crystallizer effluent PO_4-P and NH_4-N characteristics during the experiment for synthetic supernatant.

recovery potentials and possibilities. Future research areas were identified, which are likely to suggest viable solutions for a "perfect" treatment and recovery scheme.

CONCLUSIONS

Results of preliminary experimental studies showed that reasonably high t-COD, NH_4-N and PO_4-P removals are achievable, in SBR treatment of a dairy waste. Simultaneous nitrification and denitrification, and enhanced biological phosphorus removal were also possible, to different extents. There was also an indication of the coexistence of glycogen and phosphorus-accumulating organisms, in the missed liquor treatment system.

Subsequent anaerobic digestion of the solids enabled the recovery of the removed phosphorus in a sufficiently concentrated form that was suitable for recovery as struvite. The preliminary pilot-scale experiment indicated that up to 82% of PO_4-P could be removed from the dissolved phase, after processing for struvite recovery in the proprietary crystallizer, under development at the University of British Columbia.

ACKNOWLEDGMENTS

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