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The fate of crop nutrients during digestion of swine manure in psychrophilic anaerobic sequencing batch reactors

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Abstract

The objectives of the study were to measure the levels of manure nutrients retained in psychrophilic anaerobic sequencing batch reactors (PASBRs) digesting swine manure, and to determine the distribution of nutrients in the sludge and supernatant zones of settled bioreactor effluent. Anaerobic digestion reduced the total solids (TS) concentration and the soluble chemical oxygen demand (SCOD) of manure by 71.4% and 79.9%, respectively. The nitrogen, potassium, and sodium fed with the manure to the PASBRs were recovered in the effluent. The bioreactors retained on average 25.5% of the P, 8.7% of the Ca, 41.5% of the Cu, 18.4% of the Zn, and 67.7% of the S fed to the PASBRs. The natural settling of bioreactor effluent allowed further nutrient separation. The supernatant fraction, which represented 71.4% of effluent volume, contained 61.8% of the total N, 67.1% of the NH_4–N, and 73.3% of the Na. The settled sludge fraction, which represented 28.6% of the volume, contained 57.6% of the solids, 62.3% of the P, 71.6% of the Ca, 89.6% of the Mg, 76.1% of the Al, 90.0% of the Cu, 74.2% of the Zn, and 52.2% of the S. The N/P ratio was increased from 3.9 in the raw manure to 5.2 in the bioreactor effluent and 9.2 in the supernatant fraction of the settled effluent. The PASBR technology will then substantially decrease the manure management costs of swine operations producing excess phosphorus, by reducing the volume of manure to export outside the farm. The separation of nutrients will also allow land spreading strategies that increase the agronomic value of manure by matching more closely the crop nutrient requirements.

Keywords: Anaerobic digestion; Fertilizer recovery; Manure nutrients; Nutrient mineralization; Nutrient settling

1. Introduction

Current manure management practices are often detrimental to the environment and potentially hazardous to human and animal health. Manure treatment using the psychrophilic anaerobic sequencing batch reactor (PASBR) developed by Agriculture and Agri-Food Canada (AAFC) and Bio-Terre Systems Inc. deodorizes manure slurry, reduces the organic pollution load, and allows the utilisation of manure energy in the form of methane gas. Thus, the PASBR technology represents an interesting solution that addresses several environmental problems and allows farmers to adopt sustainable and environmentally sound agricultural practices that integrate animal manure into the overall production system. However, in order to assess the cost of the biotechnology and the potential agronomic value of the effluent, it is important to evaluate the fate of crop nutrients during anaerobic treatment. It is also important to determine if the biotechnology can provide a solution for production units that have nutrient surpluses.

The anaerobic digestion (AD) process is carried out by a consortium of microorganisms that transforms biodegradable organic compounds into carbon dioxide and methane. The biochemical process results in physico-chemical changes in manure composition. Gas production as well as reduction in COD and solids content are relatively well documented (Massé et al., 1997). However, there are fewer reports on the impact of AD on manure fertilizer value, and

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most studies dealt mainly with the fate of nitrogen and phosphorus. Total manure P and N are generally conserved during AD, but the mineralized fraction of the two nutrients is increased (Field et al., 1984; Larsen, 1986; Messner and Amberger, 1987; Plaixats et al., 1988). Increased NH_4–N concentration in digested manure could favour nitrogen volatilization following land application. However, studies generally reported similar NH_4–N volatilization losses following AD and raw manure applications (Chantigny et al., 2004; Rubaek et al., 1996). Results were attributed to the lower viscosity of AD manure, which increased infiltration below soil surface. On dry, hydrophobic, bare soil, volatilization was even higher with raw than AD manure (Rubaek et al., 1996).

Carbon loss as CH_4 and CO_2 during AD decreased the C/N ratio from 17.0 in raw swine manure to 10.5 in bioreactor effluent (Chaussod et al., 1986). The remaining carbon was found to be less available for biological degradation (Messner and Amberger, 1987). A lower C/N ratio in AD than raw manure reduced N immobilization by soil microflora and thus increased N mineralization and availability to the crops following application (Dahlberg et al., 1988; Demuynck et al., 1985; Juste et al., 1981; Messner and Amberger, 1987). Loria and Sawyer (2005), on the other hand, reported similar inorganic N production and net mineralization from raw and digested slurry applied to ryegrass, despite much lower COD after digestion. Rubaek et al. (1996) measured higher N uptake with AD than raw manure in the first ryegrass cut, but the reverse was observed for the second cut. They suggested that the nitrogen in AD manure was more readily available shortly after application, while the N immobilized on raw manure plots became available for the second cut. Rubaek et al. (1996) also observed higher denitrification losses on plots receiving raw than digested slurry. Other studies reported similar yield from AD and raw manure fertilized crops (Dahlberg et al., 1988; Kay and Mitchell, 1997).

Field et al. (1984) separated phosphorus, potassium, calcium, and magnesium into extractable and total fractions. The extractable fraction is used by soil testing laboratories as an indicator of nutrient availability to crop. Nearly 100% of total phosphorus, potassium, calcium and magnesium were recovered in anaerobically digested swine manure, indicating nutrient conservation during AD. However, the extractable P, Ca, and Mg fractions decreased in the AD effluent, possibly because of sorption on small particle surfaces (Field et al., 1984). The elements could also have complexed and precipitated as, for example, calcium phosphate and struvite.

The studies discussed previously were all conducted in completely mixed bioreactors. The PASBR, on the other hand, consists of a tank where the following five consecutive steps take place: (1) fill; (2) react; (3) settle; (4) draw; and (5) idle. During the settling period, no mixing is provided and quiescent conditions prevail to allow solid liquid separation. The nutrient fraction associated with suspended solids can thus be partly retained within the bioreactor sludge bed. Excess nutrient can also be further separated from the effluent through secondary sedimentation of the AD manure in storage structures. The objectives of this study were to quantify the amount of swine manure nutrients and micro-nutrients retained in PASBRs and to determine the distribution of manure nutrients in the sludge and supernatant zones of settled bioreactor effluent. This information will be useful to assess the agronomic, environmental and economic benefits of the anaerobic biotechnology.

2. Methods

The manure slurry was collected on a commercial growing–finishing swine operation. Feed samples for all bioreactors were prepared and stored in a freezer to ensure that all bioreactors received the same substrate during the whole experimental period. Feed samples were thawed and brought to room temperature prior to feeding the bioreactors.

The four 41-L laboratory scale PASBRs were located in a temperature-controlled room maintained at 17 °C. The treatment cycle lasted 4 weeks (2-week feeding and 2-week reaction periods). The organic loading rate was 4 g COD per L of sludge in the bioreactor at the beginning of each cycle per day of feeding. During the react period, the bioreactors were mixed 5 min every morning by recirculating the biogas. The mixed-liquor was allowed to settle for 72 h without mixing at the end of the react period. Bioreactor effluent was then removed from the bioreactors, measured and placed in storage structures where it was allowed to settle naturally for 3 weeks after cycle 1 and 6 weeks after cycle 2.

The volume of biogas produced by the PASBRs was measured daily with wet cup gas meters. Biogas samples were collected on days 3, 8, 10, 15, 17, 22 and 24 of each treatment cycle. Biogas composition (CH_4 and CO_2) was determined with a HachCarle 400 AGC gas chromatograph. Bioreactor sludge was sampled at the beginning and at the end of each treatment cycle (day 0 and 28). Bioreactor effluent samples were collected at the end of treatment cycles (day 28). The supernatant and sludge fractions of settled effluent were measured and sampled at the end of the sedimentation period. Samples were analysed for total and volatile solids (TS and VS), total and soluble chemical oxygen demand (TCOD and SCOD), volatile fatty acids (VFAs), alkalinity, pH, total Kjeldahl nitrogen (TKN), NH_4–N, phosphorus, potassium, calcium, magnesium, aluminium, iron, copper, manganese, zinc, sodium and sulphur. The TS content was determined by drying a 10-ml sub-sample for 24 h at 105 °C. Dried solids were incinerated for 3 h at 550 °C for volatile solids measurement. The SCOD and TCOD were determined by the closed reflux colorimetric method (APHA, 1992). The SCOD was measured on the supernatant of a centrifuged sample. VFAs (acetic, propionic, butyric, isobutyric, isovaleric, valeric and caproic acids) were analysed using a gas chromatograph

(Perkin–Elmer Corporation; Norwalk, CT) equipped with a high-resolution megabore column connected to a flame ionization detector. Alkalinity was measured by titration to a pH of 4.38. The TKN and NH_4–N were determined according to the macro-Kjeldahl method (APHA, 1992) with a Tecator 1030 Kjeltec auto-analyzer (Tecator AB, Höganäs, Sweden). Phosphorus, potassium, calcium, magnesium, aluminium, iron, copper, manganese, zinc, sodium and sulphur concentrations were analysed by the inductively coupled plasma (ICP) method at the Research and Development Institute for the Agri-Environment Laboratory (Québec City, QC).

The significance of nutrient retention within the bioreactors was assessed with a repeated measure analysis over the two cycles. Difference between nutrient mass fed to the PASBRs and withdrawn with the effluent was tested for departure from zero using a Student t-test. For each element, differences were considered significant when $P < 0.05$ for both cycles. Separation efficiency following sedimentation of the effluent was measured using the reduced separation efficiency index (E'_t) as explained in Moller et al. (2000):

$$E'_t = \frac{E_t - R_f}{1 - R_f} \quad (1)$$

where, R_f is the ratio of settled sludge volume to total volume (sludge and supernatant), and E_t is the simple separation index or

$$E_t = R_f \frac{M_c}{S_c} \quad (2)$$

where M_c is nutrient concentration in the sludge fraction, and S_c is nutrient concentration in the effluent before sedimentation. The simple separation index will yield a value greater than 0 even if no separation occurred, that is, if nutrient concentration is similar in the sludge and the supernatant fractions. The reduced separation index, on the other hand, considers both nutrient transferred and the volume of the solid fraction. It will yield a value of 0 when no separation has occurred and 1 when separation is complete. The reduced separation index was tested for departure from 0 with a repeated measure analysis over the two cycles using a Student t-test. There was no cycle effect on (E'_t) for all tested parameters, even though the sedimentation period was different for the two cycles.

3. Results and discussion

The average physico-chemical characteristics of the swine manure slurry fed to the bioreactors in both cycles are given in Table 1. The raw manure was highly concentrated with a solids content of 99.5 g/l and a COD of 131.3 g/l. Total N, P and K represented 6.7%, 1.7%, and 2.7% of TS, respectively. These values are within the ranges reported in the literature (Campbell et al., 1997; Ford and Fleming, 2000; Overcash et al., 1984). However, nutrients were highly concentrated due to the elevated TS content of the manure fed to the bioreactors.

Table 1
Characteristics of the raw swine manure fed to the PASBRs

Parameter	Raw swine manure	
	Average (8 samples)	Standard deviation
Total solids (g/l)	99.5	2.1
Volatile solids (g/l)	81.6	3.1
COD total (g/l)	131.3	14.8
COD soluble (g/l)	74.7	2.2
TKN (mg/l)	6698	61
NH_4–N (mg/l)	3875	33
Phosphorus (mg/l)	1729	18
Potassium (mg/l)	2670	18

Average COD and solids reduction during the two digestion cycles in the four PASBRs is provided in Table 2. Influent TCOD and SCOD were reduced by 68.1% and 79.9%, respectively, by conversion of organic compounds into methane and the sedimentation of particles at the bottom of the PASBRs during the settling period. Average methane production was equivalent to 224.3 g COD (Table 3), suggesting that 59% of the TCOD removed was transformed into methane while 41% was removed by entrapment within the PASBR biomass during the settling period. The COD reduction during anaerobic digestion substantially decreased the C/N ratio of the digested manure with respect to the raw manure. A decreased C/N ratio will reduce N immobilization and increase N availability shortly after land application.

The anaerobic process reduced the total solids of manure by more than 71% (Table 2) as a result of particle hydrolysis and sedimentation within the PASBRs. The reduction in TS concentration, from 99.5 g/l in raw manure to 28.7 g/l in AD effluent, should substantially decrease viscosity. Kumar et al. (1972) reported a 14-fold decrease in apparent viscosity as manure solids content was reduced

Table 2
COD and solids mass reduction during anaerobic digestion of swine manure in PASBRs – average of two cycles and four PASBRs

Parameter	Feed (g)	Effluent (g)	Reduction (%)[a]
COD total	557.1 ± 68.1	173.8 ± 20.3	68.1 ± 7.1
COD soluble	316.7 ± 12.7	63.5 ± 2.9	79.9 ± 1.5
Total solids	422.1 ± 11.8	120.7 ± 6.9	71.4 ± 1.9
Total volatile solids	346.1 ± 16.5	78.5 ± 3.4	77.3 ± 1.3

[a] Transformed into methane or remained in bioreactor (%).

Table 3
Average biogas composition and total methane production from PASBRs digesting swine manure – average of two cycles and four PASBRs

Cumulative biogas production (L)	121.4 ± 2.1
Methane content of biogas (%)	69.2 ± 4.2
Cumulative methane production (L)	83.4 ± 1.4
Cumulative methane production (g COD CH_4)[a]	224.3 ± 3.7

[a] Based on 0.37 g COD/l CH_4 at 17 °C.

Table 4
Nutrient and micro-element mass balance in PASBRs digesting swine manure – average of two cycles and four PASBRs

Nutrient	Feed (g)	Effluent[a] (g)	Remaining in bioreactor (%)
N total	28.40 ± 0.54	28.35 ± 1.28	0.1 ± 5.8
NH_4–N	16.43 ± 0.09	22.76 ± 1.5*	
P	7.33 ± 0.06	5.46 ± 0.55*	25.5 ± 7.5
K	11.32 ± 0.17	11.52 ± 0.98	−1.9 ± 9.5
Ca	3.09 ± 0.01	2.82 ± 0.31	8.7 ± 9.8
Mg	2.24 ± 0.58	1.72 ± 0.38	18.7 ± 29.5
Fe	0.39 ± 0.06	0.32 ± 0.04	15.0 ± 20.0
Cu	0.15 ± 0.04	0.09 ± 0.01*	41.5 ± 14.8
Mn	0.06 ± 0.01	0.05 ± 0.01	21.0 ± 21.9
Zn	0.24 ± 0.04	0.19 ± 0.01	18.4 ± 17.7
Na	4.13 ± 0.43	3.88 ± 1.06	3.8 ± 33.0
S	1.14 ± 0.23	0.46 ± 0.06*	67.7 ± 22.9

[a] The * symbol indicates that element mass fed to the bioreactors was significantly different from the mass withdrawn with the effluent at the $P = 0.05$ level of significance in both cycles.

from 86 to 41 g/l. A decrease in viscosity should increase the infiltration rate of manure below soil surface and reduce emissions of ammonia, thereby increasing nitrogen uptake by the crops.

The nutrient and micro-nutrient mass balance in the PASBRs is presented in Table 4. Total N was not retained in any significant quantity in the bioreactors, but total NH_4–N was significantly higher in the effluent than in the feed. The increase in the NH_4–N/TKN ratio, from 57% in raw manure to 80% in effluent, should increase the short term fertilizer value of manure, because NH_4–N is more readily available to crops than organic nitrogen. Additionally, the increase in NH_4–N concentration is generally offset by the decrease in viscosity such that volatilization becomes similar for raw and digested manure, even though the latter contains a higher NH_4 concentration (Chantigny et al., 2004; Rubaek et al., 1996).

In both cycles, 25.5% of the phosphorus was retained in the bioreactors, thereby increasing the N/P ratio from 3.9 in the raw manure to 5.2 in the treated effluent. The increase in the N/P ratio would make the effluent from PASBRs treating swine manure more appropriate for crop fertilisation by reducing the deficit in N when the allowed land application rate is based on the phosphorus requirement. In both cycles, there was no statistically significant K, Fe, Mg, and Na retention in the PASBRs. The bioreactors retained on average 8.7% of the Ca, 21.0% of the Mn, and 18.4% of the Zn. However, retention was statistically significant in one of the two cycles only. Copper (41.5%) and sulphur (67.7%) retention was significant in both cycles.

The physico-chemical characteristics of the supernatant and sludge fractions of settled effluent are presented in Table 5. The sludge fraction represented 28.6% of effluent volume and contained 57.6% of the solids, 41.6% of the TCOD, 34.5% of the total N, 62.3% of the P, 71.6% of the Ca, 89.6% of the Mg, 76.1% of the Al, 90.0% of the Cu, 74.2% of the Zn, and 52.2% of the S. The reduced separation index (E'_t), which is an indicator of the degree of nutrient separation in the sludge fraction, was significantly greater than 0 for all parameters except NH_4–N, potassium, and sodium (Table 4). The E'_t reached 0.52 for phosphorus, compared to a E'_t of 0.82 for phosphorus removal from AD swine manure with a decanting centrifuge (Moller et al., 2000), a more costly technology than natural sedimentation. The E'_t was over 0.60 for all other elements except sulphur, which mostly remained in the PASBRs.

The supernatant fraction, which represented 71.4% of total effluent volume, contained 61.8% of total nitrogen and only 33.5% of the phosphorus. Further P retention

Table 5
COD, solids, and elemental mass balance of settled bioreactor effluent – average of two PASBR cycles and four settling tanks

Parameter	Decanted bioreactor effluent				Reduced separation efficiency index[a]
	Mass (g)		Fraction of total effluent (%)		
	Supernatant	Settled sludge	Supernatant	Settled sludge	
TCOD	71.32 ± 5.14	71.38 ± 5.35	41.6 ± 6.0	41.6 ± 6.1	0.31 ± 0.04**
SCOD	49.67 ± 5.94	20.33 ± 2.19	78.6 ± 12.7	32.1 ± 4.3	0.01 ± 0.04
Total solids	50.59 ± 2.29	69.60 ± 8.31	42.0 ± 2.7	57.6 ± 5.0	0.42 ± 0.03**
VS	29.50 ± 1.01	47.79 ± 5.48	37.7 ± 2.3	60.9 ± 6.1	0.47 ± 0.04**
TKN	17.54 ± 1.43	9.79 ± 0.86	61.8 ± 3.3	34.5 ± 2.3	0.11 ± 0.01**
NH_4–N	15.26 ± 1.47	7.17 ± 0.39	67.1 ± 5.5	31.5 ± 1.0	0.05 ± 0.04
P	1.82 ± 0.08	3.42 ± 0.63	33.5 ± 2.5	62.3 ± 7.0	0.52 ± 0.04**
K	8.03 ± 0.20	3.24 ± 0.10	70.1 ± 5.6	28.3 ± 2.8	0.01 ± 0.01
Ca	0.58 ± 0.08	2.02 ± 0.36	20.6 ± 3.3	71.6 ± 8.4	0.70 ± 0.04**
Mg	0.10 ± 0.02	1.54 ± 0.40	6.1 ± 1.5	89.6 ± 9.7	0.91 ± 0.02**
Al	0.03 ± 0.00	0.07 ± 0.01	24.0 ± 11.2	76.1 ± 10.9	0.62 ± 0.06**
Fe	0.06 ± 0.01	0.25 ± 0.04	19.9 ± 2.3	78.4 ± 5.7	0.72 ± 0.03**
Cu	0.03 ± 0.00	0.08 ± 0.01	32.8 ± 3.7	90.0 ± 17.4	0.63 ± 0.05**
Mn	0.01 ± 0.00	0.04 ± 0.01	11.5 ± 2.0	87.6 ± 7.2	0.84 ± 0.03**
Zn	0.05 ± 0.00	0.14 ± 0.02	25.8 ± 1.8	74.2 ± 7.1	0.65 ± 0.03**
Na	2.60 ± 0.61	0.65 ± 0.11	73.3 ± 31.5	19.0 ± 9.8	−0.11 ± 0.07
S	0.22 ± 0.03	0.28 ± 0.03	42.7 ± 12.0	52.2 ± 13.9	0.38 ± 0.04**

[a] The ** symbol indicates that the reduced separation efficiency index was different from 0 at the $P = 0.01$ level of significance.

in the settled sludge increased the N/P ratio from 3.9 in the raw manure to 9.2 in the liquid fraction of the settled bioreactor effluent. Land application of settled AD manure will therefore not be controlled by phosphorus and the high N/P ratio will correspond more adequately to crop requirements. Corn, for example, has a N:P uptake of 7.5 to 1 (Edwards and Daniel, 1992). The application of settled AD manure would be especially recommended on fields already saturated in P, as is often the case in regions with high animal farming density. Separation of nutrients, even on farms that have no P surpluses, will allow land spreading strategies that increase the agronomic value of the manure by matching more closely applied nutrients to plant and soil requirements and reduce the risk of surface and ground water contamination. On farm with phosphorus surpluses, the retention of phosphorus in bioreactors and its accumulation in the settled sludge of treated effluent will allow the excess P to be exported outside the farm at a substantially lower cost.

4. Conclusions

The objectives of this study were to determine the level of manure nutrient recovery during the low temperature anaerobic digestion of raw swine manure and to establish, after a natural settling period, the distribution of manure nutrients between the sludge and supernatant zones of settled bioreactor effluents. This information is required to better assess the agronomic, environmental and economic benefits of the biotechnology. The experiment was conducted in laboratory scale PASBRs.

The anaerobic process reduced TS and SCOD contents of manure by 71.4% and 79.9%, respectively. The bioreactors retained on average 25.5% of the P, 8.7% of the Ca, 41.5% of the Cu, 18.4% of the Zn, and 67.7% of the S fed with the raw manure during both cycles. Nutrient retention was statistically significant in both cycles for P, Cu, and S. The natural settling of bioreactor effluent resulted in further separation of phosphorus and other elements. The supernatant fraction, which represented 71.4% of total effluent volume, contained 61.8% of the TKN, 67.1% of the NH_4–N and 73.3% of the Na. The settled sludge fraction, which represented 28.6% of the effluent volume, contained 57.6% of the solids, 62.3% of the P, 71.6% of the Ca, 89.6% of the Mg, 76.1% of the Al, 90.0% of the Cu, 74.2% of the Zn, and 52.2% of the S. The N/P ratio was increased from 3.9 in the raw manure to 5.2 in the bioreactor effluent and 9.2 in the settled effluent. On commercial farms with phosphorus surpluses, phosphorus retention in the sludge fraction of settled effluent will substantially decrease manure management costs by reducing the volume of manure to be exported outside the farm. The separation of nutrients will also allow land spreading strategies that increase manure's agronomic value by matching more closely the crop nutrient requirements, and therefore it will reduce the risk of surface and ground water contamination.

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