



# Technical and operational feasibility of psychrophilic anaerobic digestion biotechnology for processing ammonia-rich waste

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## HIGHLIGHTS

- Long-term anaerobic digestion (AD) process at high-ammonia (>5 gN/L) is limited.
- PADSBR technology was validated to treat N-rich waste with  $8.2 \pm 0.3$  gNH<sub>3</sub>-N/L.
- Excess ammonia (8.2 gN/L) did not affect the digestion process with no inhibition.
- VFA, an indicator for process stability, did not accumulate in PADSBR.
- Biomass acclimation in PADSBR ensured a high-stabilization of the AD process.

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## ABSTRACT

Ammonia nitrogen plays a critical role in the performance and stability of anaerobic digestion (AD) of ammonia rich wastes like animal manure. Nevertheless, inhibition due to high ammonia remains an acute limitation in AD process. A successful long-term operation of AD process at high ammonia (>5 gN/L) is limited. This study focused on validating technical feasibility of psychrophilic AD in sequencing batch reactor (PADSBR) to treat swine manure spiked with NH<sub>4</sub>Cl up to  $8.2 \pm 0.3$  gN/L, as a representative of N-rich waste. CODt, CODs, VS removals of  $86 \pm 3$ ,  $82 \pm 2$  and  $73 \pm 3\%$  were attained at an OLR of 3 gCOD/L.d, respectively. High-ammonia had no effect on methane yields ( $0.23 \pm 0.04$  L CH<sub>4</sub>/gTCOD<sub>fed</sub>) and comparable to that of control reactors, which fed with raw swine manure alone (5.5 gN/L). Longer solids/hydraulic retention times in PADSBRs enhanced biomass acclimation even at high-ammonia. Thus VFA, an indicator for process stability, did not accumulate in PADSBR. Further investigation is essential to establish the maximum concentrations of TKN and free ammonia that the PADSBR can sustain.

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## 1. Introduction

Hog production is a vital element of Canada's agricultural economy. The agricultural sector in Québec, one of the largest provinces in Canada, generated 7.5% of total greenhouse gas emissions (GHG) in the year 2006, that is, 6.36 Mt of carbon dioxide equivalents [1]; whereas emissions created by pig production, due, among other things, to the spreading of swine manure as fertilizer, contributed to about 15% of total farm emissions, which indicates less than 1% of total GHG emissions in Québec [2]. Although the fact that the pig farming industry is not a leading source of GHG emissions, an association is occasionally made between these emissions including ammonia and odours, that is the primary cause for the swine sector

considers it essential to promote the good farming practices as a means of reducing these emissions.

Replacing fossil fuels with renewable energy, for instance biomethanization, is an effective manure management options to reduce the total GHG emissions for the agricultural sector [3]. Despite these benefits, digestion of swine manure as a sole substrate has previously been shown to be unsuccessful, mainly due to the high content of ammonia in this waste. Ammonia is regularly reported as the primary cause of digester failure because of its direct inhibition of microbial activity [4–6]. Total ammonia concentration (TAN) greater than 4 gN/L was shown to be inhibitory during digestion of livestock manure [4,7,8]. TAN comprises of free (un-ionized) ammonia (NH<sub>3</sub>) [FAN] and ionized ammonium nitrogen (NH<sub>4</sub><sup>+</sup>), in which FAN has been suggested as the cause of inhibition in high ammonia loaded process since it is freely membrane-permeable [7,9]. FAN concentration primarily depends on few important parameters such as TAN, temperature, pH and ionic strength of the digesting material. An increase in digester

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operating temperature commonly increases the metabolic rate of the microorganisms and studies have suggested that increase in temperature will lead to an increase in the fraction of FAN [7,8,10]. Thus, psychrophilic AD process has a tendency to inhibit less and found to be more stable than thermophilic/mesophilic temperatures especially for high-ammonia loaded digesters [11].

Several studies have concentrated on the prevention of various process imbalances, predominantly via development of different process control strategies, automation and augmentation of process monitoring [11]. Few other studies have attempted to come up with practical solutions to avoid inhibition and harvest stable biogas production such as: (i) dilution of reactor content [9,12]; (ii) addition of materials – such as bentonite, glauconite and phosphorite with ion exchange capacity [13,14]; (iii) Struvite precipitation [15] and use of carbon fiber textiles [16]; (iv) adjustment of the feedstock C/N ratio and pH [11,12]; and (v) lowering temperature from thermophilic (55 °C) to more moderate conditions [40–50 °C] [17]. However, some of these techniques either had a significant negative effect on methane production or economically not feasible; and according to our best understanding none of these control techniques have been successfully implemented on the farm scale. Adaptation of microbes especially the methanogens to high ammonia concentration could accelerate the ammonia tolerance [8,18]. Several high-rate anaerobic processes are developed in the recent past and to date, practically all full-scale AD applications are restricted to mesophilic temperatures, which typically require heating for efficient treatment. Dague and co-workers (U.S. Patent No. 5, 185,079) have developed the high-rate anaerobic sequencing batch reactor (ASBR) for the treatment of swine manure (total chemical oxygen demand (COD), 71.5 g/L; and total kjeldahl nitrogen (TKN), 4.5 g/L) at psychrophilic (25 °C) temperature. However, to avoid ammonia toxicity in the reactors, they have diluted the raw swine manure by a factor of four. For swine waste, Zhang et al. [19] reported the successful operation of ASBR, with biogas production rate of 0.9–1.8 L/L/d, at the maximum ammonia concentration of 2.5 g/L in the pH range of 6.8–7.4. Zeeman et al. [20] and Wellinger and Sutter [21] have stated that start-up of anaerobic reactors to be operated at low temperatures needs inoculation with bacteria already acclimated to psychrophilic temperatures. Besides, the long-term successful operation of the AD process at higher ammonia concentrations (i.e. >5 gN/L) has not yet been reported. In addition, such research for the low temperature AD process is limited.

As temperature is considered as a evident factor, which affects the threshold of ammonia inhibition, the purpose of this work was to investigate the technical feasibility of psychrophilic anaerobic digestion in sequencing batch reactor (PADSBR) to treat swine manure with excess ammonia nitrogen. We induced the ammonia inhibition by spiking with ammonia chloride (NH<sub>4</sub>Cl) to laboratory-scale PADSBRs to simulate the sharp increase in TAN levels to 8.2 ± 0.3 gN/L that may occurs in actual centralized biogas plants when proteinaceous co-substrates are fed to the reactors. This research is crucial to prevent ammonia inhibition primarily to get rid of bioreactors failure or avoid reduced performance in terms of energy recovery and biogas plant economic efficiency. Further, the proposed low-temperature AD technology is expected to promote the good farming practices in cold weather countries, for instance North America.

## 2. Methods and materials

### 2.1. Feedstock and inoculum sources

The fresh raw manure slurry was collected from a manure transfer tank on a commercial swine operation located in

Sherbrooke, Quebec province of Canada. The manure was screened to remove particles larger than 3.5 mm to avoid the operational problems especially plugging of the influent line with the small scale digesters. The manure was then mixed to prepare homogenize feed samples and stored in a cold room at 4 °C to prevent biological activity. NH<sub>4</sub>Cl was chosen as the source of ammonia instead of any other ammonium salt was due to the fact that chloride was reported less inhibitory than ammonium [8]. The inoculum was sourced from the farm-scale bioreactor of Pelloquin's swine farm located in Ste-Edwidge de Clifton (Quebec), which was already acclimatized to the treatment of swine manure slurry. Average manure and inoculum characteristics during the experimental period are given in Table 1.

### 2.2. Experimental set-up and operating principles of PADSBR process

The anaerobic fermentation of swine manure was performed using six PADSBRs, such that four identical (replicates) digesters were used to study the effect of excess ammonia concentrations on the AD process using swine manure spiked with NH<sub>4</sub>Cl; whereas another two (replicates) PADSBRs were kept as control, which fed with pig manure only. PADSBRs were installed at a controlled-temperature room, adjusted at a temperature of 24.5 ± 0.5 °C. The sludge volume in the all the reactors were maintained at 20-L (effective volume, 24 L) and the OLRs were based on the amount of COD<sub>fed</sub> (gTCOD<sub>fed</sub>) per L of sludge. All the reactors were operated for more than 1 year (375 days) and the operating conditions are presented in Table 2.

A typical operation cycle length consists of 4 weeks which included the fill, react, settle and draw phases. The fill step involves the addition of swine manure to the PADSBR system. The feeding was carried out on day 0 and 7 of each cycle and the feed volume was determined on the basis of desired OLR used in this study. During the react phase, the soluble organics and some of the suspended organic particulates were transformed into biogas by the anaerobic microorganisms. At the end of every 4 week cycle (i.e. end of react phase), the settling of biomass was completed and the supernatant (treated) wastewater was drawn out from the PADSBRs leaving 20-L sludge volume before feeding with fresh manure. The volume decanted is normally equal to the volume fed during the fill step. The high food to microorganism (F/M) ratio occurred immediately after feeding step resulted in high-rate of substrate utilization and hence, high-rate of waste conversion to biogas. Whereas, towards the end of react phase, the F/M ratio was at its lowest level with low biogas production, provided ideal conditions for biomass settling and thus enhanced longer solids (biomass) retention time. Mixing was done by recirculating the biogas using a dual-head air pump twice a week for about 5 min just before taking mixed liquor samples for analysis purpose only. Otherwise, no additional external mixing was employed primarily to simulate more suitable operational conditions on a commercial

**Table 1**  
Properties of swine manure and inoculum.

Parameter	Swine manure	Inoculum
Total COD (g/L)	146.71 ± 24.5	18.08 ± 2.9
Soluble COD (g/L)	42.22 ± 6.0	5.87 ± 0.1
Total solids (%)	10.5 ± 2.0	1.9 ± 0.2
Volatile solids (%)	8.7 ± 2.2	1.0 ± 0.1
Fixed solids (%)	1.9 ± 0.3	0.9 ± 0.1
Total VFA (g/L)	22.1 ± 4.3	0.14 ± 0.01
TKN (g/L)	8.4 ± 0.6	5.1 ± 0.4
NH <sub>3</sub> -N (g/L)	6.3 ± 0.4	4.1 ± 0.2
pH	6.91 ± 0.2	7.80 ± 0.1
Alkalinity (gCaCO <sub>3</sub> /L)	22.0 ± 2.4	18.1 ± 0.7
Phosphorous (g/L)	1.9 ± 0.1	0.58 ± 0.4

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