A thermophilic anaerobic digester with ultrafilter (TADU) for solids separation offers potential advantages of higher VS destruction, biomass retention, and pathogen removal. However, potential disadvantages include ultrafilter fouling, decreasing flux, and high VFA concentrations. In this study, a thermophilic anaerobic digester coupled to a sintered titanium, cross-flow ultrafilter was operated for over five months. Dairy manure was digested (HRT of 23 days). The filtrate VFA concentration was low (220 mg/L as HAc), average VS destruction was 49%, and a low average effluent fecal coliform concentration of $10^2$ MPN/100 mL was observed. The low coliform value may be beneficial if dewatered biosolids are used for livestock bedding since low pathogen counts help prevent mastitis. Ultrafilter fluxes of 40–80 L/m²-hr were maintained by cleaning using caustic (3.5% NaOH) followed by water and acid (3% phosphoric acid). Sand from livestock bedding was found to damage the pump and ultrafilter. If TADU were implemented at full scale, then replacing sand bedding with dewatered biosolids should be considered.

Keywords Anaerobic digestion; dairy manure; membrane bioreactor; thermophilic; ultrafiltration

Introduction

Anaerobic digestion of waste with high solids concentration, such as dairy manure, has been applied to reduce odors, produce biogas for energy, and produce stabilized biosolids. The most basic anaerobic technologies for dairy manure digestion include ambient temperature lagoons and covered ambient or mesophilic lagoons. More advanced processes now used include mesophilic complete mix digesters, and mesophilic plug flow digesters (Kramer, 2002; US Department of Agriculture, 2004). With these digesters, hydraulic and solids retention times are identical. The appropriateness of a given reactor configuration depends, to a large extent, on the solids concentration of the manure. High-solids manure (approximately 14% total solids) comes from mechanically scraped barns, whereas lower-solids manure comes from hydraulically flushed barns, or manure mixed with milking parlor wash water (<9% total solids). Plug flow digesters are typically recommended for manure with more than 11% total solids.

Other dairy manure digestion processes have been investigated or implemented, including thermophilic co-digestion of agricultural and solid wastes (Ahring, 1994), fill-and-draw thermophilic acid-gas phasing (Liao and Lo, 1985), anaerobic fixed-film reactors (Wilkie, 2000), temperature phased complete-mix digesters (Katers and Schultz, 2003), and thermophilic-mesophilic phased sequencing batch reactors (Dugba et al., 1997).

Ultrafilter separation and anaerobic reactors

Ultrafiltration, the use of permeable barriers to separate aqueous and solid phases, is being considered more widely in place of sedimentation for solids retention in suspended growth bioreactors. The process uses a material with pore openings in the range 0.005–0.1 μm to separate solid and aqueous phases (Reynolds and Richards, 1996). When
coupled to an anaerobic digester, essentially all the suspended solids can be retained in the digester. Therefore, the solids retention time (SRT) can be controlled separately from the hydraulic residence time (HRT). The benefits of ultrafilter-coupled anaerobic digesters can include relatively long SRT values that can lead to more complete volatile solids destruction, a high active biomass concentration for significant substrate removal at relatively short HRT values, good pathogen removal (since many pathogens are larger than 1 μm), and production of filtrate that contains very low or no suspended solids.

Membrane and ultrafilter technologies have been investigated in laboratory scale with anaerobic digestion for brewery and alcohol distillery wastewater (Nagano et al., 1992; Choo and Lee, 1996), piggery waste (Lee et al., 2001), municipal wastewater (Elmaleh and Abdelmoumni, 1998), and various industrial wastewaters (Vogel, 2004). No previous reports have described thermophilic anaerobic digestion with ultrafiltration (TADU) for dairy manure digestion to the authors’ knowledge.

The cost of using ultrafilters with anaerobic digesters is, in part, a function of ultrafilter flux rates, fouling, and cleaning requirements. The flux represents the flow of the aqueous phase that passes through a unit area of filter surface, and is a function of transmembrane pressure, water viscosity, and membrane resistance. Typically, membrane resistance increases rapidly during the first few days of operation, and then increases more slowly thereafter due to fouling caused by both inorganic precipitates, such as struvite, and microorganisms as they clog ultrafilter pores (Choo and Lee, 1996; Elmaleh and Abdelmoumni, 1998).

Cleaning can be conducted using caustic, acidic, or other solutions, and must typically address both biological and inorganic fouling. For example, Lee et al. (2001) report that an alkali solution (NaOH) effectively removed biological foulants, whereas a subsequent acid (HCl) solution was required to remove inorganic foulants. The flux recovered up to 24% of the original rate after cleaning with one-molar NaOH, and then recovered to 89% after a subsequent cleaning with one-molar HCl.

Ultrafiltration to retain solids and increased SRT along with thermophilic temperatures to increase digestion rate and pathogen inactivation may be advantageous when digesting dairy manure. However, potential disadvantages include filter clogging and damage to some ultrafilters caused by abrasive livestock bedding material (e.g., sand) and/or thermophilic temperatures. In this study, a thermophilic digester with ultrafilter (TADU) was employed to stabilize dairy manure and was maintained for over five months to determine operating characteristics, preliminary ultrafilter cleaning protocols, and flux rates.

Methods
Thermophilic anaerobic digester with ultrafilter
A pilot-scale TADU was operated at a dairy farm (Frost Farms, Waterford, WI, USA). The system consisted of an insulated polypropylene vessel (diameter = 0.78 m, height = 0.85 m) containing an active volume of 340 L. The digester temperature was maintained at 55 ± 2°C using a submerged heater and thermostat, and the content was continuously mixed using an impeller mixer. Liquid seals for the mixer shaft and waste/feed access were made from chlorinated polyvinyl chloride (CPVC) pipe suitable for temperatures as high as 80°C. Pipe was placed through the digester cover and submerged about 45 cm below the surface of digester content to help reduce biomass contact with air (see Figure 1).

The ultrafilter was a 1.9 cm diameter, 1.5 m long, sintered titanium pipe having a surface area and pore size of 0.09 m² and 0.2 μm, respectively (Arbortech, McHenry, IL, USA). The ultrafilter was annularly enclosed in a casing pipe to which filtrate flowed. During wasting operations, the ultrafilter-digester system was operated in a configuration in which digester content was pumped at a rate of 60 L/min for approximately four hours.
in a loop through the cross-flow ultrafilter, with retentate flowing back to the digester (see Figure 1). This gave a self-cleaning cross-flow velocity of 3.3 m/s through the ultrafilter.

The system was seeded with mesophilic biomass from an anaerobic contact reactor treating milk and cream waste (Kerry Ingredients, Jackson, WI, USA) and then fed prepared dairy manure at a rate of 15 L/day. To prepare digester feed, fresh cow manure was blended with an equal volume of well water, and the mixture was passed through a 6.35-mm screen. In this way, some undigested cow feed (e.g., grass, corn) was removed from the manure. The average volatile solids (VS), total chemical oxygen demand (TCOD), and soluble COD (SCOD) values for the prepared feed were 4.4, 5.3, and 2.1%, respectively. The average VS loading to the digester was 1.9 kg/m³-day.

Wasting and feeding were performed three times per week (Monday, Wednesday, and Friday). First, 17.5 L (i.e., 7.5 L/day) of digester contents as well as 17.5 L of either filtrate or, when the recycle pump was inoperable, digester supernatant were removed. Digester supernatant was obtained by allowing 20 L of digester contents to settle for 2 hours and then decanting 17.5 L of the supernatant as waste. The remaining settled volume was returned to the digester. The digester was then fed 35 L of prepared manure. The HRT was maintained at 23 days, whereas the SRT averaged 30 days based upon solids measurements of digester supernatant, filtrate, and digester content wasted.

**Methanogen activity assay and other analyses**

Digester content aliquots were taken once per week immediately after feeding and used for digester activity (DA) and methanogen activity assays (MAA). DA results were used to estimate digester biogas and methane production rates since it was difficult to prevent digester biogas leaks and obtain accurate rates with measurements from the TADU itself.

For DA testing, 50 mL of digester contents was placed in a 160-mL serum bottle which was then sparged with gas (30/70% v/v CO₂/N₂), sealed with a butyl rubber septum, and placed in a shaker table at 55 °C. Biogas production was measured daily using a wetter-barrel glass syringe, and headspace gas was analyzed using gas chromatography. The resulting biogas production rate was always essentially constant for more than six days, and the rate was determined by linear regression from a plot of cumulative biogas produced versus time (Vogel, 2004). This procedure is similar to others used for biochemical methane potential (Owen et al., 1979).
For MAAs, serum bottles were prepared as described above, except that 13 g/L of calcium acetate was added to provide a significant excess of substrate for aceticlastic methanogens. Thus, MAA methane production rate is not a function of substrate concentration, and indicates the activity of aceticlastic methanogens in the biomass. Methane production rate was determined from linear regression using initial slope of cumulative methane volume produced versus time.

Solids, fecal coliform, volatile fatty acids (VFAs), and other parameters were measured by *Standard Methods* (1998). VS destruction was determined using two methods. First, VS destruction was determined by mass balance using solids data collected after three SRTs had passed and using a steady-state assumption. Second, VS destruction over the entire operating period (over 5 months) was determined by calculating the total mass of VS added to the system, minus the VS mass change in the digester volume and VS mass removed from the system. Both methods yielded the same average VS destruction value.

The composition of serum bottle headspace gas was determined using a gas chromatograph (GowMac, Bethlehem, PA, USA) with a thermal conductivity detector and a packed column (CTR-1, Altech, Deerfield, IL, USA). All biogas and methane volumes were converted to standard temperature and pressure (0°C, 1 atmosphere), and all VFA data reported or referred to herein are in units of equivalent mg/L as acetic acid (HAc). Details of all methods can be found elsewhere (Vogel, 2004).

**Results and discussion**

The TADU system was operated for over three SRTs (105 days) before quasi-steady-state data were collected. Results described below were obtained during the seven-week, quasi-steady-state period unless otherwise noted. The effluent VFA concentration was 220 ± 80 mg/L, and was significantly lower than expected since most investigators report thermophilic VFA values in the range 500–3,000 mg/L (e.g., Vandenburgh and Ellis, 2002; Kim et al., 2002). In addition, thermophilic processes typically produce significantly higher VFA concentrations than similar mesophilic systems. In this regard, Vandenburgh and Ellis (2002) report VFA concentrations of 400–2,800 mg/L for thermophilic, and 50–500 mg/L for mesophilic digesters. The TADU system described herein produced an effluent with VFA concentrations more typical of mesophilic, rather than thermophilic systems.

The VS destruction efficiency was 49 ± 3%, with biogas containing 69 ± 7% methane. This VS destruction is approximately 20% higher than the value reported for an extensively-monitored mesophilic manure digester operated at a 30-day SRT at a similar dairy (Tinesdale Farms) in Wrightstown, WI, USA (Katers and Schultz, 2003). The mesophilic seed sludge demonstrated thermophilic aceticlastic activity immediately as indicated by MAA results. During digester start-up, the MAA increased from an initial value of 0.029 m³ CH₄/kg VS-day (39% of the ultimate quasi-steady-state value) and reached the quasi-steady-state value of 0.074 m³ CH₄/kg VS-day after approximately 100 days. These MAA values are on the lower end of the range 0.02–0.34 m³ CH₄/kg VS-day reported for aliquots of biomass from full-scale thermophilic digesters in Denmark (Ahring, 1994). The initial DA value averaged 0.004 m³ CH₄/kg VS-day, and increased to the quasi-steady-state value of 0.025 m³ CH₄/kg VS-day after 50–100 days.

Fecal coliform concentration in the filtrate was relatively low, with a six-log inactivation observed for both filtrate and settled supernatant. Influent fecal coliform most probable number (MPN) values averaged 10⁷ MPN/100 mL, and average filtrate and settled supernatant values were both 10² MPN/100 mL, and less than 140 CFU/gTS. It is probable that this very good inactivation was mostly due to the thermophilic temperature and batch feeding every 2.3 days on average, rather than the ultrafiltration step. The
frequent fill-and-draw operations typical of many municipal treatment plant digesters result in short holding times for some fraction of particles, and this decreases pathogen inactivation. For example, a thermophilic digester that was treating municipal sludge with an HRT of 5.7 days and operating with fill-and-draw pumping every 2 hours produced biosolids with fecal coliforms counts as high as 2,600 CFU/gTS (Vandenburgh and Ellis, 2002). Others have described the influence of feeding intervals on fecal coliform inactivation (Farrell et al., 1988).

The ultrafilter withstood thermophilic temperatures and aggressive chemical cleaning. However, the sand bedding used at the farm entered the digester, wearing out the recirculation pump seals and abrading the ultrafilter. The initial ultrafilter flux rate increased from 140 to 230 mL/min, but began to decrease (see Figure 2). Up until 130 L of filtrate was generated, the ultrafilter was cleaned at points designated as “filter wash” in Figure 2. During these times, a 10% sulfuric acid solution was delivered to the ultrafilter casing pipe. The acid solution then permeated the surface from the outside into the ultrafilter pipe. After approximately 140 L of filtrate was removed, the ultrafilter developed holes, ostensibly from wear due to sand. Subsequently, a new ultrafilter was installed and the cleaning procedure was changed. A caustic cleaning (3.5% NaOH) followed by a water rinse and subsequent treatment with 3% phosphoric acid was used. The cleaning solutions were periodically delivered directly to the ultrafilter pipe, and flowed from the inside out (designated as “inner filter wash” in Figure 2). It was thought that this would bring the cleaning solutions in more immediate contact with foulants and lead to more complete cleaning. The operating flux rate of 60–125 mL/min (40–80 L/m²-hr) was maintained. The effectiveness of similar alkali–acid cleaning has been described by Lee et al. (2001). The alkali solution removes biological foulants, whereas the subsequent acid solution removes inorganic foulants, such as struvite.

**Conclusions**

The TADU system is a promising scheme for treating some wastes with high solids concentrations, such as dairy manure. When screened dairy manure was digested at an HRT of 23 days, the average VFA concentration in the filtrate was relatively low, the average VS destruction was 49%, and a relatively low average effluent fecal coliform concentration of 10² MPN/100 mL was produced. The relatively low effluent fecal coliform values may be important when considering digested, dewatered biosolids as animal bedding material. Typically, the cost of traditional bedding materials is significant. In light
of this, biosolids are being considered as an economical alternative to sand and other material. If used in this way, it is preferable to have biosolids with low pathogen content to help prevent mastitis and provide a more publicly appealing operation.

The titanium ultrafilter employed was cleaned with a caustic solution (3.5% NaOH) followed by a water rinse and acid cleaning (3% phosphoric acid). This sequence worked better than using an acid solution alone, and helped maintain flux rates of 40–80 L/m\(^2\)-hr. The sand used at the dairy for animal bedding damaged the pump and ultrafilter. It would be beneficial for dairy operations to change to a more compatible bedding material when TADU digestion is initiated. Ultimately dewatered TADU biosolids could be used as bedding, and sand would not be needed.

Mesophilic biomass can be used to seed thermophilic digesters. The mesophilic seed biomass employed initially exhibited 39% of the ultimate quasi-steady-state thermophilic aceticlastic activity observed, and the activity consistently increased during the start-up period. More research is required to determine long-term ultrafilter behavior, improved cleaning protocols, and TADU process economics at full scale.

References