Converting Gin and Dairy Wastes to Methane

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Abstract. *Alternatives to gin trash and manure disposal would benefit both the cotton ginning and dairy industries. Anaerobic digestion produces both methane gas and a class A soil amendment. Gin and dairy wastes were combined in the solid phase portion of a two-phase anaerobic system to determine the combinations of temperature, rewetting interval, and mixture ratio that maximize potential methane gas production and minimize process completion time. No significant volatile fatty acid formation occurred after leachate pH approached 7.0, indicating process completion. This took approximately three weeks when temperatures were above 32°C (90°F), mixture ratios were below 5:1 (gin to dairy waste, dry mass basis), and the solid phase was wetted twice daily. Ten percent of the mass was converted to soluble chemical oxygen demand (COD), which has potential for conversion to methane in the second phase.*

Keywords. *Anaerobic digestion, Cotton gin trash, Manure, Methane production, Pathogen reduction.*

Recently passed concentrated animal feeding operation (CAFO) regulations restrict nutrient land application rates, in some cases limiting traditional manure management practices. This has led New Mexico dairy producers to consider more costly alternatives like composting and anaerobic digesting. The New Mexico State University Bioenergy Taskforce convened to study "current technologies applied to the recovery of methane from the anaerobic digestion of animal manures and evaluate them in the context of dairy practices in New Mexico" (Hanson et al., 2003). The two-phase anaerobic digester was one of the more promising systems considered in the context of desert dairies because of its minimal water requirement. A commercial-sized two-phase system is under construction that will demonstrate the feasibility of the technology using manure from a New Mexico dairy. Cotton gin trash is mixed with the manure to raise the C:N ratio and reduce pathogen populations. This study was conducted to obtain system parameters for designing the commercial-sized two-phase anaerobic digester.

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Dairy Byproducts

The dairy industry is facing increasing pressure to dispose of manure in ways that reduce odor, limit public exposure to pathogens, and reduce the likelihood of contaminating ground and surface water with nutrients. For example, recent federal regulations designed to protect the waters of the U.S. from nutrients and wastes apply to all animal feeding operations, including dairies. Liquid manure, wastewater, and even storm water contacting the production area must be contained. Biochemical oxygen demand and fecal coliform bacteria must not be allowed to enter waters of the U.S. Soil and any material applied to it (manure, litter, process wastewater, or residues thereof) must be analyzed for nitrogen and phosphorus content, so annual application rates are consistent with the expected nutrient uptake of the coming crop (EPA 2004).

New Mexico has the largest average dairy herd size (1,700 head) in the nation, and is currently ranked seventh in the nation for milk production. New Mexico dairy producers operate in a dry, temperate climate and typically house animals in open corrals, where about 40% of the manure is deposited. Approximately 45% to 50% of the manure is deposited along feed lanes. These unpaved corrals and feed lanes are typically scraped using heavy equipment. The remaining 10% to 15% of manure is deposited in the milking parlor, where cows are normally milked three times a day. There, from 95 to 130 L (25 to 35 gal) of water per cow per day is used for washing and flushing. Manure from the milking parlor (and feed lanes if they also have a flushing system) is typically stored in a lagoon after solids separation. However, 85% to 90% of the manure produced on most New Mexico dairies is dry (5% to 15% moisture) and typically dry stacked (piled into mounds) before land application. Estimated daily manure excretion for a New Mexico lactating cow is 6.85 kg (15.1 lbs) dry mass (Hanson et al., 2003).

Cotton Gin Byproducts

Cotton ginning involves the removal of both seeds and foreign matter from cotton lint. One 227 kg (500 lb) bale from spindle-picked cotton starts as 680 kg (1500 lb) of seed cotton. The balance is approximately 363 kg (800 lb) of seed and 91 kg (200 lb) of trash. Two-thirds of the gin trash in the U.S. comes from stripper-harvested cotton, where an average of 907 kg (2,000 lb) of seed cotton per bale is brought to the gin, 318 kg (700 lb) of which is trash. Cotton seed is usually fed to cattle or crushed for oil. While the market for cotton seed is well established, gin trash is more difficult to sell. The trash, mostly leaf material, stems, and hulls, may be disposed of by spreading on soil, feeding to livestock, or solid waste disposal. Roughly half the gins in the U.S. spend money disposing of this resource (Thomasson, 1990). Annually, 15 to 20 million bales of cotton are produced in the U.S., resulting in over two million metric tons of cotton gin trash, over 50% of which is volatile matter (Holt et al., 2000).

Two-Phase Anaerobic Digestion

Pohland and Ghosh (1971) proposed a two-phase anaerobic process that separates cellulose degradation and volatile fatty acid (VFA) formation from methanogenesis. This results in a higher quality gas (by excluding the respiration gases of the acidifiers from the methane production reactor) and a lower capital and operating cost (smaller anaerobic vessel, less water, and more rapid cycling).

A pilot system was tested for treating New Mexico dairy manure (fig. 1) both alone and in combination with gin trash. Pumps recirculated water from the sump area beneath the solid phase pile through spray nozzles above the pile. As water drained through the pile, it picked up VFA. Recirculating the leachate kept the pile moist and maintained a high leachate VFA concentration. Previous studies operating this system found a correlation between VFA concentration and pH (Yu et al., 2002). Therefore, when the pH of this leachate fell to 5.8, it was fed to two fixed-film upflow anaerobic filters (UAF) containing established colonies of methanogenic bacteria. As fresh leachate was pumped into the bottom of the UAF, the liquid displaced off the top flowed by gravity back to the sealed reactor containing the solid phase. This process continued until leachate pH reached 7.00, corresponding to a low VFA content. Macias-Corral et al. (2005) found that one dry ton of combined gin trash and cow manure produced 87 m^3 (3072 ft^3) of methane gas. The UAF product gas was 73% to 86% methane. (The solid phase produced smaller amounts of gas from 35% to 74% methane.) This gas production was 10.6 m^3 per m^3 digester volume, 35% higher than that attained with manure alone. The process reached completion in 45 days, as determined by pH level. Substantial reductions in weight and volume of feedstock were also achieved. The resulting soil amendment was 2.32% nitrogen, 0.38% phosphorus, and 1.14% potassium. Total nitrogen was twice the level typically achieved by aerobic composting, most likely due to ammonia retention during the initial anaerobic treatment (Macias-Corral et al., 2005).

Biosecurity Benefit

Consistent and near-complete inactivation of both total and fecal coliforms was observed in leachate from the two-phase anaerobic digester when cotton gin trash was mixed with manure, but not when digesting manure alone (Riordan, 2003). This was confirmed by laboratory studies in which either untreated or sterilized manure was mixed with either pure cellulose or gin trash and then seeded with enterohemorrhagic *E. coli* (EHEC) strain O157:H7 bacteria. In the sterile and non-sterilized treatments containing gin trash, EHEC was below detection after 12 days, and remained below detection for the remainder of the trial. The level of EHEC reduction observed in both treatments with cotton gin trash corresponded to the loss of fecal coliform detectability by culture on selective media. Conversely, in both treatments without gin trash EHEC could be detected throughout the trial (Riordan et al., 2005).

Objective

Demonstrating the feasibility of two-phase anaerobic digestion in the context of New Mexico dairy waste management requires operating a full-scale prototype system. This study was conducted to obtain system parameters for designing a full-scale prototype producing both energy and a class A soil amendment (less than 1,000 most probable number *E. coli* or *Salmonella* colonies per gram). Of special interest was the impact of operating temperature, leachate recirculation interval, and mixture ratio on solid phase VFA productivity and completion time. These critical variables influence methane productivity and system design.

Methodology

A complete prototype system operated at New Mexico State University (NMSU) provided full-scale system methane production data but did not have provisions for varying

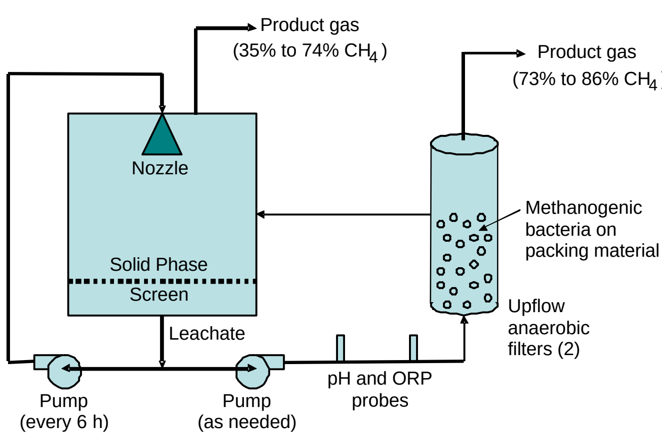

Figure 1. The two-phase biofermentation process separates cellulose degradation and acid formation in the solid phase from methane production in the upflow anaerobic filters. The closed system is operated as a batch process with no effluent discharged.

the solid phase operating temperature. A smaller-scale study was conducted at the USDA-ARS Southwestern Cotton Ginning Research Laboratory adjacent to NMSU to discover optimum operating parameters for the solid phase portion. Chemical oxygen demand (COD) data from both studies was used to predict potential methane production for the optimization study. Both studies traced fecal coliform bacteria populations as an indicator of pathogens. (A separate study by Riordan (2003) focused specifically on pathogen mortality attributable to gin trash.)

Solid Phase Optimization Study

Cotton gin trash (approx. 5% to 10% moisture) was combined with dry bovine manure (5% to 15% moisture, 10% to 15% sand) scraped from a dairy feed lot and dry aged from one to three months. The ratio of gin trash to manure ranged from 1:1 to 10:1 dry mass basis (varied according to the central composite experimental design, table 1). Samples (10 kg each) of this mixture were wetted to field capacity (using 0.125% Induce, a surfactant, to counter the hydrotropic property of cotton lint) and layered in 38 L (10 gal) plastic tanks 29 cm (11.5 in.) in diameter by 58 cm (23 in.) high. Each tank drained into a 19 L (5 gal) plastic bucket equipped with a recirculation pump. The pumps were activated by timers (DT17C, Intermatic, Inc., Spring Grove, Ill.) on a schedule that varied from 1 min every 2 h to 1 min every 24 h and returned leachate to the top of the pile in the tank. Each plastic tank was in a 135 L (35 gal) galvanized steel garbage can equipped with insulation and an electric heating element (fig. 2). The temperature in the tanks was thermostatically controlled (iSeries Temperature Controller, Omega, Stamford, Conn.) and ranged from 20°C to 45°C (68°F to 113°F). A central composite experimental design was selected to find the optimum combination of mixture ratio, temperature, and wetting frequency. Their influence on the response variables (time to complete the process and cumulative COD, fig. 3) was analyzed for significance and plotted as a response surface (fig. 4) using Design-Expert (Stat-Ease, 2000).

Twenty lots (six replicates at the center to determine pure error, eight corner points, and six axial points) were run in sets of four from May through December of 2002. The approximately 2 L of leachate that had accumulated in the catch bucket was discarded and replaced with 2 L of fresh water each morning. Pumps circulated the fresh water to the top of the solid phase. The water trickled down through the pile and accumulated in the buckets. The pH and NaCl in the fresh leachate were measured using handheld direct-read meters; NaCl was estimated by measuring conductivity and applying a manufacturer-supplied calibration (pHTestr and SaltTestr, Oakton Instruments, Vernon Hills, Ill.). In addition, 125 mL samples were collected and refrigerated for later COD analysis (High Plus Range COD Reagents, Hach Co., Loveland, Colo.). Sodium chloride was monitored to see if it was soluble enough to be washed out of the material before eventual land application, since dairy waste may contain dietary salt. Note that the larger pilot plant-scale reactor circulated leachate daily through a second methanogenic phase (where VFA were consumed) and then back to the solid phase. Because this small-scale trial lacked that second methane phase, it used daily fresh water replacement (pH 7.12 to 7.73) of leachate to simulate VFA consumption by the missing methanogenic bacteria. This may have resulted in P and K transport as well.

Once leachate pH levels indicated completion of VFA formation, the solid phase residue was carefully removed, aerobically composted in windrows for 60 days, and tested for soil amendment properties using standard procedures (Soil Water and Air Testing Laboratory, New Mexico State University, Las Cruces, N.M.).

Table 1. Solid phase optimization study experiment design and results.[a]

Mean Process Temperature		Wetting Interval	Mixture Ratio Trash:Manure	Completion	Cumulative COD (per 10 kg dry feedstock)	Methane Potential (per 10 kg dry)
(°C)	(°F)	(h)	(dry basis)	(days)	(g)	(L)
24.7	76.4	4	2	27	1,281	4,355
25.0	77.0	4	8	[b]	--	--
27.6	81.6	12	2	16	1,026	3,488
28.3	83.0	8	5	23	780	2,652
29.0	84.2	12	8	18	964	3,278
29.9	85.8	24	5	21	1,024	3,482
30.5	86.9	8	5	23	1,182	4,019
30.9	87.6	8	1	14	--	--
30.9	87.7	8	10	29	--	--
31.4	88.5	8	5	21	--	--
31.7	89.0	8	5	19	--	--
31.9	89.5	8	5	20	1,104	3,754
32.0	89.6	8	5	21	1,154	3,924
32.1	89.7	2	5	13	1,140	3,876
32.2	90.0	8	5	23	870	2,958
36.2	97.2	4	8	17	931	3,165
37.3	99.2	4	2	13	563	1,914
37.1	102.0	12	2	19	--	--
37.9	103.0	12	8	23	--	--
43.3	111.0	8	5	16	--	--

[a] Missing numbers are from two runs that were not analyzed.
[b] This run never reached apparent completion.

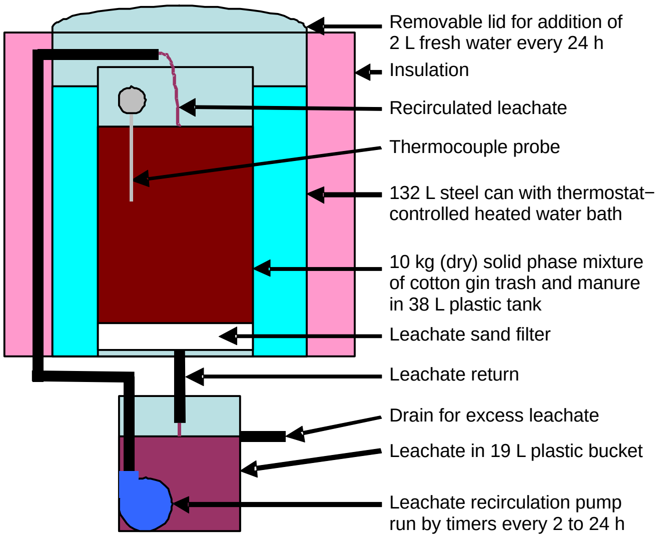

Figure 2. Solid phase portion of biphasic process as operated for the solid phase optimization study. Fresh water replaced leachate after pH and COD sampling each day.

Results and Discussion

For the time-to-completion response variable, the most significant effect was temperature (1.6% level of significance), followed by mixture ratio (3% level). Increasing the temperature from 25˚C to 43˚C (76˚F to 110˚F) decreased completion time, as did increasing the dry manure portion from 1:10 to 1:1 (table 1). A response surface showing days to completion as a function of solid phase temperature and gin trash to dairy waste mixture ratio is presented in figure 4. The response surface model had an adjusted R^2 of 0.42. Frequency of wetting (omitted from the model) had only a minor influence on time to complete acidogenesis. The mean time to completion was 20 days. Rising pH levels indicated a drop in VFA content in this system and corresponded to declining COD levels.

Figure 3 illustrates the relationship between COD and pH. For the four runs shown, operating temperature and wetting frequency are the same. The lot with equal parts gin trash and manure (triangles) had already completed VFA and COD production. The lot with 10% manure by weight (squares) was proceeding much more slowly and therefore still had a low pH (corresponding to high VFA levels in this system) and higher COD levels than the other two lots. The remaining two lots with intermediate ratios of gin trash to manure (5:1) were in the process of reaching completion, as indicated by their increasing pH and declining COD levels.

Cumulative COD production for each 10 kg (22 lb) batch of dry feedstock was largely independent of the three controlled variables. (Cumulative COD production appeared to decrease with increasing temperature, but there is no statistical significance to support this observation.) Cumulative COD removed was typically 1 to 1.2 kg, or from 10% to 12% of initial dry matter. This corresponded to a potential methane production of 0.04 to 0.05 m^3 (14 to 18 ft^3) per kg dry matter, based on the ratio developed by Yu et al. (2002) correlating methane production to COD. Yu found that methane production in fixed-film upflow anaerobic columns was proportional to feed water COD content, with yield at standard conditions averaging 0.34 m^3 methane per kg COD. Yield in the solid phase optimization study was approximately half as much as was found with the larger-scale system operated by Yu, in part because he was using pure manure. Also, VFA mass transport can be limiting, and keeping the pH level depressed in the solid phase is necessary to prevent methanogen establishment there. In this experiment, methanogenisis following rising pH levels in the solid phase may explain part of the VFA production that was not accounted for.

Sodium was removed from the solid phase during the process (leachate sodium went from over 10,000 to under 2,000 ppm). Composting the solid phase residue resulted in a soil amendment that was 36.39% organic material (dry mass basis). Elements (ppm) were: 99.63 Mg,

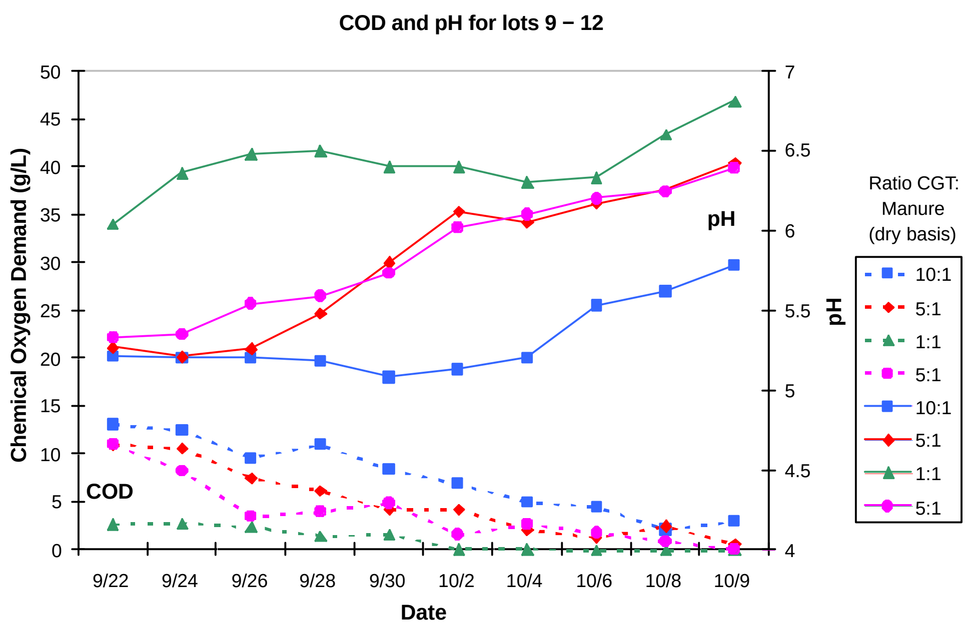

Figure 3. Plot showing correlation between acidity (pH) and chemical oxygen demand (COD) in the leachate over time for four digester runs. Process temperature and wetting frequency were constant; ratio of cotton gin trash to manure (dry basis) varied.

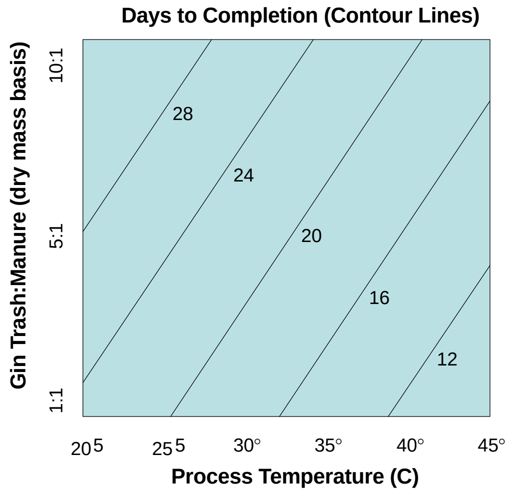

Figure 4. Response surface showing days to completion (diagonal lines) as a function of solid phase temperature and cotton gin trash to manure mixture ratio.

328.66 Ca, 90.81 Na, 449.0 P, and 654 K. Sodium absorption ratio (SAR) was 1.13, confirming that the sodium chloride initially in the manure could be flushed out.

CONCLUSIONS

Based on solid phase optimization study results, more rapid completion (and hence smaller solid phase piles) can best be accomplished through temperature control (adding insulation and/or supplemental heating). Maintaining the dairy waste proportion above 20% dry basis also contributes to more rapid process completion. Provided the pile is wetted twice daily, more frequent pumping of leachate onto the solid phase did not appear to appreciably accelerate the process. Based on pilot plant operation results, it is possible to produce 87 m^3/ton high-methane content (72%) gas in a two-phase reactor. The two-phase process, with its smaller volume, is potentially more economical to build (10.6 m^3 product gas per m^3 digester volume) and to operate in an arid region (15% water by volume) than a plug flow digester, and is far less sensitive to the high sand content of New Mexico dairy manure. The resulting soil amendment has twice the nitrogen content it would have if composted aerobically. Pathogen inactivation trials indicate that the gin trash contributes to complete disinfection in as little as eight days. Even pasteurized gin trash is effective at eliminating both indicator organisms and *E. coli* O157:H7. The resulting soil amendment is therefore safer for food crops applications than residue from processes that only digest manure. And reducing manure volume may lower transportation costs associated with delivering it to distant fields in compliance with CAFO regulations.

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