

Fixed-film Anaerobic Digestion of Flushed Dairy Manure after Primary Treatment: Wastewater Production and Characterisation

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The characteristics of flushed dairy manure after primary treatment at a Florida dairy were monitored daily over a 1-year period along with cow numbers and water use, with the aim of developing fixed-film anaerobic digestion of flushed dairy manure wastewater (FDMW). Primary treatment included screening through a mechanical separator followed by sedimentation. The FDMW exhibited average total solids (TS), volatile solids (VS) and total chemical oxygen demand (COD) levels of 3580, 2210 and 3530 mg l^{-1} , respectively, for a milking herd average of 359 cows and a wastewater production of 502 m³ d⁻¹. Analyses of FDMW parameters and manure excretion estimates indicated that 53% of the TS, 40% of the VS and 58% of the total COD remained in the wastewater after screening and sedimentation. The separation of fibrous solids increased the COD to VS ratio of the FDMW. Wastewater temperature was found to significantly affect both the separation of TS, VS, and total COD, and the partitioning of wastewater parameters between the particulate and soluble phases. Higher FDMW temperatures tended to reduce TS, VS, and total COD removal efficiencies. The ratio of soluble COD to dissolved VS increased with temperature, while the ratio of particulate COD to volatile suspended solids decreased, indicating that significant levels of biological activity occurred in the sedimentation basin at higher temperatures. The VS measurement does not accurately determine the methane potential of components in FDMW compared to COD measurements. In spite of the fact that 60% of the VS is removed by screening and sedimentation, the major portion of the methane potential remains in the wastewater since the solids that are removed are relatively non-degradable fibres and do not contribute to methane production. Model equations were developed to predict total COD levels in flushed dairy manure wastewater as a function of fresh flushwater usage per animal unit for similar dairy operations, providing an important design parameter for implementation of fixed-film anaerobic digestion systems.

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1. Introduction

The management, treatment and disposal of liquid and solid manure at dairy operations are receiving increased attention. In Florida, governmental regulation of waste disposal activities at dairies is cited as having a negative impact on dairy producers (Tefertiller *et al.*, 1998). The anaerobic digestion of manure, however, is known to have the potential to produce energy, reduce odours, mineralise nutrients, inactivate weed seeds and lower pathogen levels (Davis, 2001; Hayes *et al.*, 1980; Powers *et al.*, 1999; Wilkie *et al.*, 1995; Wilkie, 2003). In

Florida, the typical use of large volumes of flushwater for dairy manure collection means that conventional anaerobic digestion using complete-mix or plug-flow technologies is neither practical nor economical since the dilute manure streams would require excessively large digester volumes and higher heat inputs in order to achieve the hydraulic retention times (HRT) and operating temperatures required for stable digestion (HRT>15 days at 35 °C).

Primary treatment (screening, sedimentation, or both) of flushed dairy manure is widely practised in the dairy industry since it is required to improve operation of

some wastewater irrigation systems (Brodie, 1989). In addition, screening and sedimentation to remove solids are useful in reducing the organic loading rate to anaerobic lagoons in order to extend their capacity and reduce the frequency of sludge removal. However, even after removal of solids, it is the liquid fraction of flushed dairy manure that contributes most to odour concerns beyond the farm boundary (Mackie *et al.*, 1998).

One practical alternative is to apply high-rate anaerobic digestion technology, such as fixed-film digestion, to treat the flushed dairy manure wastewater (FDMW), which is defined as the liquid fraction of flushed dairy manure after solids are removed. Fixedfilm digestion can be applied at ambient temperatures and at much shorter hydraulic retention times (HRT ≤ 3 days) than allowed by conventional technologies (Wilkie, 2000). Anaerobic treatment can reduce the odour potential and pathogen levels in FDMW, while conserving plant nutrients and producing energy. A major impediment to this approach is the general lack of data characterising the liquid fraction of flushed dairy manure. In an effort to fill this gap in understanding, a yearlong study was undertaken to characterise flushed dairy manure wastewater after mechanical separation and sedimentation. Such solids separation methods represent typical pretreatment steps for developing an appropriate influent for a fixed-film anaerobic digester.

2. Literature review

2.1. Flushed dairy manure production and characteristics

Studies of FDMW at other dairies indicate a wide range of characteristics, due primarily to the amount of flushwater used, the use of recycled water for flushing, and the use of organic bedding materials. Over a 180day period, Moore and Miner (1985) measured total solids (TS) concentrations, after separation of solids using a stationary inclined screen, varying from 10 000 to $32\,000\,\mathrm{mg}\,l^{-1}$ at a 120-cow dairy using from 15.7 to 34 m³ d⁻¹ of flushwater, which included some recycled water. They were able to attribute the wide range of TS concentrations to the introduction of rainfall runoff into the wastewater. Stowell and McKenney (1998) measured the TS content in recycled flushwater, after solids separation in an Agpro® separator, and found it to range from 9600 to $33300 \,\mathrm{mg}\,l^{-1}$, while the volatile solids (VS) ranged from 6200 to $24\,800\,\mathrm{mg}\,l^{-1}$. Hills and Kayhanian (1985) measured the TS in a composite sample of FDMW from a 500-cow dairy using around $762 \hat{l} A U^{-1} d^{-1}$ [1 animal unit (AU) = 1000 kg live animal mass] of recycled flush wastewater and found it to average $11\,000\,\mathrm{mg}\ l^{-1}$. Most of the solids were found in the first minute of a 3.5 min flushing event.

Williams and Frederick (2001) measured total chemical oxygen demand (COD-T) and VS of FDMW from a 200-cow dairy (estimated to be 225-cow equivalent due to confinement of dry and replacement heifers) using around 2260 l AU⁻¹ d⁻¹ of partially recycled flushwater and reported their averages as 3000 and 3200 mg l^{-1} . respectively. Chastain et al. (2001) measured TS and VS for FDMW after mechanical screening and sedimentation at a 54-cow Jersey-herd dairy, which used 22 700 l d⁻¹ of fresh flushwater, and found levels of 11470 and 8705 mg l^{-1} , respectively. NRCS (1992) reported the TS, VS, and COD-T levels in milk house and parlour wastewater as 6000, 4200, and $5000 \,\mathrm{mg}\,l^{-1}$ respectively. Not all of these studies report the cow densities and wastewater production levels corresponding to their characterisation data and they fail to report on the soluble versus particulate fractions of the TS, VS, and COD which are critical to evaluating the wastewater as a potential influent to fixed-film anaerobic digestion.

Specific wastewater production levels on dairies using hydraulic flushing are dependent on the amount of flush water use. Flushed dairy manure wastewater production based on AU were calculated to be, at a minimum, $1010 l AU^{-1} d^{-1}$ for Florida dairies using clean flushwater (Van Horn *et al.*, 1993). But commonly, wastewater production levels are $2090 l AU^{-1} d^{-1}$ at dairies that use flushing systems (Van Horn *et al.*, 1993).

2.2. Impact of screening and sedimentation

Two major impediments to estimating the organic strength and quantity of FDMW are imprecise excretion estimates for dairy cows and lack of data for VS and COD-T removal efficiencies of separation processes. The ASAE data on dairy manure production has been shown to be low (Morse et al., 1994; Safley et al., 1984; Van Horn et al., 1994, 1998) for dairy cows with high dry matter (DM) intake. Methods to estimate excretion rates based on DM intake have been developed (Van Horn et al., 1998) but do not directly estimate COD-T excretion rates. The VS and COD-T removal efficiencies of screening and sedimentation processes reported in the literature have ranged from 50–73% and 41–67%, respectively (Barrow et al., 1997; Chastain et al., 2001; Fulhage & Hoehne, 1998; Graves et al., 1971; Hegg et al., 1981; Powers et al., 1995). Unfortunately, most studies on separation have focused on TS removal only and no studies were found which examine the fate of soluble versus particulate fractions and their relative degradability.

2.3. Implications of wastewater characterisation for anaerobic digestion

The VS in dairy manure is not highly degradable compared to that of other animal manures due to the high digestion efficiency of the rumen system in cows, along with their fibrous diet. Hill (1991) estimated the ultimate degradability of whole manure VS for dairy cows at only 23% compared to 63% for both swine and poultry. Data from plug-flow digesters indicate a higher VS reduction for whole dairy manure. Mattocks and Moser (2000) evaluated two farm-scale plug-flow mesophilic digesters treating scraped dairy manure at two 1000-cow dairies and estimated that VS reductions were 35% at a 20 d HRT and 42% at a 22 d HRT. Hayes et al. (1980) found an overall VS reduction of 40.6% for a pilot-scale plug-flow digester treating dairy manure at a 30 d retention time and 35 °C. Since plugflow digesters may accumulate solids, the estimates for VS reduction may be high but may approach a reasonable estimate of the ultimate biodegradable fraction of the VS.

The non-degradable fibrous fraction of dairy manure VS impedes the anaerobic digestion process by affecting the pumping characteristics of the manure, by allowing scum formation, and by displacing reactor volume and reducing the active volume for biogasification. Furthermore, in fixed-film digesters, coarse fibrous solids may contribute to media plugging and short-circuiting of wastewater around the media bed. Since solids separation tends to remove a VS fraction that is high in fibrous solids, it also tends to remove the non-degradable portion of the VS, leaving the more degradable fraction in the FDMW.

Previous studies have compared the anaerobic digestion of whole and separated dairy manure (Haugen & Lindley, 1988; Hills & Kayhanian, 1985; Lo et al., 1983; Pain et al., 1984). From bench-scale studies in mixed reactors operated at 30 °C and HRTs from 6 to 16 d, Lo et al. (1983) concluded that removing 50% of the manure as fibrous solids would not significantly reduce biogas production, while requiring only 3/8 of the reactor volume needed for whole manure digestion. In farm-scale studies using mixed reactors operated at 15 and 20 d HRTs and 32-34 °C, Pain et al. (1984) found that separation of 52% of dairy manure VS resulted in a more digestible fraction that produced only 30% less biogas than whole manure and, further, suggested that improvements in separation might allow removal of only the non-degradable solids. In bench-scale, 40 d batch studies at 35-37 °C, Haugen and Lindley (1988) found that the lowest methane production came from the fraction of dairy manure retained on a 2.36 mm screen. The above studies involved dairy manure from dairies that did not use extensive flushing water.

For dairies with flushing systems, the concentration of non-degradable fibres in separated solids should be enhanced since higher volumes of water will tend to wash the finer more degradable solids into the wastewater. Based on bench-scale studies in mixed reactors operated at a 10 d HRT and 35 °C, Hills and Kayhanian (1985) estimated that 85% of the potential methane was left after filtering flushed dairy manure through a 2 mm screen. However, they found that sludge from 30 min bench-scale settling retained 54% of the methane potential of the unseparated flushed dairy manure. A thorough characterisation of FDMW, which examines the fluctuations in soluble and particulate fractions under field-scale conditions, can help the design and operation of ambient temperature fixed-film digestion by matching the appropriate reactor volume with the proper organic loading rate and hydraulic retention

3. Materials and methods

3.1. Dairy Research Unit

The University of Florida Dairy Research Unit is located about 19 km northwest of campus in Hague, Florida (lat. 29°44′N, long. 82°23′W). The average ambient temperature is 21·1 °C. During this study (July 1995 to June 1996), the facilities included a 175-cow freestall barn, an additional 200-cow freestall barn, and a double-eight herringbone milking parlour, which were all hydraulically flushed. The milking herd was confined to the freestall barns. Dry cows, culled cows, prepartum cows, bred heifers and calves were not housed in the freestall barns.

The confined milking herd averaged 359 Holstein cows and ranged from 337 to 373 cows, with an average weight of 590 kg. The herd was fed a total mixed ration consisting of maize silage, sorghum silage, alfalfa hay, whole cottonseed, cottonseed hulls, soya bean meal, maize meal, citrus pulp and minerals. The average daily dry matter intake was 24·1 kg cow⁻¹, consisting of 27·3 kg forage (30% DM), 2·7 kg dry forage, and 13·2 kg concentrates. Cows were milked three times daily and milk production averaged 27·3 kg cow⁻¹.

3.2. Manure-handling system

Barn alleys were flushed with well water from two 41700 l storage tanks through four (11400 l/min) popup flush valves (Agpro[®] Model SF 12×20 MK II; Agpro Inc., Paris, TX), which were operated manually for approximately 1 min per alley three times a day.

The milking parlour was flushed from a 12 100 *l* storage tank with two flush valves 9–12 times per day for 30 s. The milking parlour apron was equipped with an 'udder washer' and, together with the milking parlour washdown wastewater and the alley flushing water, the manure flowed to a wastewater collection channel. In summer, misters in the freestall barns cooled the cows and also contributed additional water to the waste stream. The freestalls were bedded with sand. In addition to flushing, the alleys were scraped once per day with a skid-steer loader fitted with a tyre scraper.

The manure-handling system is illustrated in Fig. 1. The flushed dairy manure initially flowed down the collection channel to a sand-trap (emptied once per day), where some of the sand was recovered for reuse as bedding. After the sand-trap, the flushed dairy manure flowed through a channel to a mechanical separator (Agpro® Model 36G) equipped with a stainless-steel bar-screen, with a 1.5 mm screen spacing, which removed large fibrous solids. The inclined separator was fed at the bottom and the separated wastewater fell through the screen into a gutter, which directed the liquid fraction to a sedimentation basin. Belt-driven polyvinyl chloride paddles pushed the fibrous solids up over the screen, where they were deposited onto a concrete pad. Leachate from the stacked solids also flowed into the sedimentation basin.

Following the mechanical separator, the wastewater flowed across the $76.5 \,\mathrm{m}^3$ settling basin (nominal depth of 50 cm) and then over a weir into a sampling pit. The FDMW then flowed into an adjacent primary storage pond with a volume of $1970 \,\mathrm{m}^3$. After the primary storage pond, the FDMW flowed through a culvert to a secondary storage pond, which had a volume of

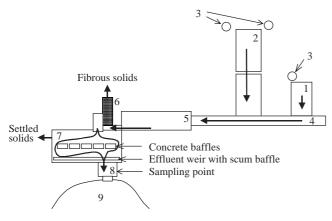


Fig. 1. Schematic layout of University of Florida Dairy Research Unit manure-handling system; heavy arrows indicate flow of flushwater and manure solids: 1, milking parlour; 2, freestall barns; 3, flushwater holding tanks; 4, wastewater collection channel; 5, sand-trap; 6, mechanical separator; 7, sedimentation basin; 8, sampling pit; 9, primary storage pond

2100 m³. From there, it was pumped to an anaerobic lagoon, which had a volume of 30 500 m³. From the anaerobic lagoon, the liquid was irrigated directly onto cropland. The sedimentation basin was emptied once or twice a week. Solids removed by the separator and from the settling basin were spread directly onto croplands. The rainfall catchment area flowing into the sedimentation basin was 3716 m².

3.3. Sampling and analysis

Daily milking cow numbers were obtained from farm records. Cow weights, dry matter intake, milk production and quality were obtained from monthly dairy herd improvement records. The daily wastewater flow was determined from the daily water use readings of a metered well (Model: 4 inch McPropeller MF100; McCrometer, Hemet, CA) after subtracting estimates of consumptive uses (Table 1), which included potable water use, water removed in milk, water intake by the unconfined herd and evaporation. It was assumed that the water intake by the milking herd in the confinement barns was mostly conserved in urine and faeces and, therefore, this was not considered as a consumptive use.

Daily potable water use was estimated from an average of monthly potable water records obtained when a local municipality supplied this water. The water removed in milk was determined from dairy herd improvement records by subtracting protein, fat, and sugar contents (assumed 4.4% sugar) from average monthly milk yield, multiplying by the monthly herd average, and computing the average daily water removed in milk for the year. Water intake for prepartum and dry cows, bred heifers, and culled cows was estimated to average 103 l cow⁻¹ d⁻¹ (Van Horn *et al.*, 1993). The average cow numbers for these herds were determined from farm records. Evaporation for the

Table 1
Estimates of average daily consumptive uses of water that did not contribute to the wastewater flow

Consumptive use	$m^3 d^{-1}$
Potable water use	3.79
Water leaving in milk	8.34
Water intake, prepartum and dry cows $(61.3 \text{ cows } @ 103 l \text{ cow}^{-1} \text{ d}^{-1})$	6.31
Water intake, bred heifers (56.8 cows @ 103 l cow ⁻¹ d ⁻¹)	5.85
Water intake, culled cows (100 cows @ 103 lcow ⁻¹ d ⁻¹)	10.30
Evaporation, confined herd (359 cows @ 49·21cow ⁻¹ d ⁻¹)	17.66
Total	52.25

confined milking herd was estimated at $49 \cdot 2 l \cos^{-1} d^{-1}$ (Van Horn *et al.*, 1993). The estimate of the average daily consumptive water use was $52 \cdot 25 \,\mathrm{m}^3 d^{-1}$ or less than 10% of total water use. The daily FDMW volume was determined by subtracting the estimate of the consumptive water use from the daily water meter readings.

Daily rainfall measurements were read manually on a rainfall gauge. However, linear regressions of all wastewater parameters on daily catchment rainfall volume did not yield significant effects (data not shown). This could be due to the limited runoff from small rainfall events, as well as the addition of manure from unconfined animals during large rainfall events, which effectively masked any dilution effect. Thus, catchment rainfall was not added to the daily wastewater flow.

Wastewater temperature was recorded once daily during collection of 24 h composite samples. Since this temperature did not represent the average temperature of the FDMW over the 24 h sampling period and due to the large variations in manual temperature readings, soil temperature at a depth of 10 cm was chosen as the best available estimate of average daily wastewater temperature. Daily soil temperature was averaged from archived weather data recorded at a Gainesville, FL weather station (AWARDS, 1996). A linear regression of average daily soil temperature versus the daily wastewater temperature reading was highly significant with a regression coefficient of 0.99 (data not shown).

Samples of FDMW were taken after mechanical separation and sedimentation of fibrous and settleable solids. A dipper-type automatic sampler (Model: 8392-300; Phipps & Bird, Richmond, VA) collected 200 ml samples into a cooler at 15 min intervals to obtain a daily composite sample (approximately 19.21). Daily sub-samples were obtained after mixing the composite sample and stored at 4 °C. Duplicate measurements of total solids (TS), volatile solids (VS), fixed solids (FS), total chemical oxygen demand (COD-T), pH, alkalinity, and electrical conductivity (EC) were performed according to standard methods (APHA, 1995). Samples for COD-T analyses were blended for 2 min to permit representative sub-sampling. Samples for total suspended solids (TSS), soluble COD (COD-S), and volatile suspended solids (VSS) were prepared by centrifuging 30 ml samples at 12 000 min⁻¹ for 30 min. A portion of the primary supernatant was recovered for COD analysis to determine COD-S. The remaining supernatant was discarded and the pellet re-suspended in 30 ml of de-ionised water, followed again by centrifuging at 12 000 min⁻¹ for 30 min. Discarding supernatant, re-suspending the pellet, and centrifuging were repeated a third time and then the pellet was analysed for TS and VS in accordance with standard

methods (APHA, 1995) to give values for TSS and VSS. Dissolved solids (DS) and dissolved volatile solids (DVS) were calculated as the difference of TS and TSS, and VS and VSS, respectively. Dissolved fixed solids (DFS) were calculated as the difference between DS and DVS. Particulate COD (COD-P) was calculated as the difference of COD-T and COD-S.

Daily sample analyses and wastewater volumes were combined with cow numbers and average weight to determine the daily production of each parameter on an AU basis in order to allow comparison to existing manure production data. Total excretion of raw manure was estimated from Van Horn *et al.* (1998).

3.4. Statistical methods

The averages of duplicate analyses from daily composite samples were treated as replicate observations from a normally distributed sample population. Data were entered in a Microsoft Excel 97 spreadsheet and ratios and AU production levels were calculated from the daily averages, after which, means, standard deviations, ranges, and numbers of data points were calculated. Therefore, the ratio of the average levels does not precisely equate to the average of the ratios of paired samples. Linear regressions were performed in Excel and the significance of regression coefficients was tested using the Student's *t*-test. The appropriate variance and effective degrees of freedom were determined and the results were subjected to a two-tailed *t*-test at a significance level α of 0·01, unless stated otherwise.

4. Results

The FDMW production and characterisation data for the sampling period from July 1995 to June 1996 are plotted in Fig. 2. Considerable variation exists in the data for the first 8 months of the study. After January 1996, however, the data appear to be more consistent. Since flushing was controlled manually, this variation may be attributed to changes in management and labour practices through the fall of 1995, which were resolved in January. Levels for TS, TSS, and COD-T also varied during the first 8 months of the study, with occasional peaks that were more than five times their base levels. Volatile solids and VSS levels follow the trends for TS during this same period. However, COD-S levels, which are independent of the suspended solids content of FDMW, were less variable during this period. The variability in the wastewater characteristics was probably caused by intermittent losses of particulates from the sedimentation basin during the first 8 months, due to 462 A.C. WILKIE ET AL.

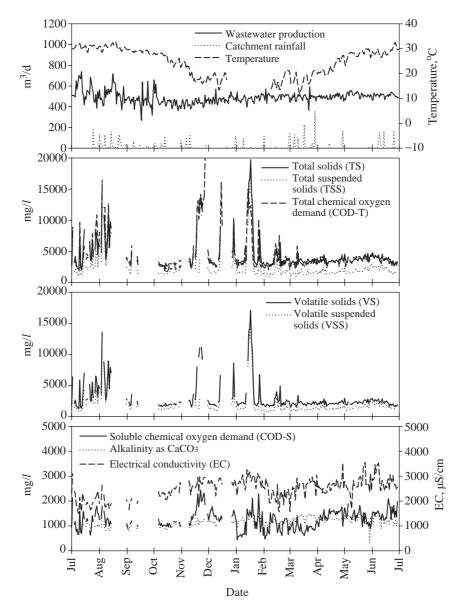


Fig. 2. Flushed dairy manure wastewater production and characterisation data

a combination of poor sedimentation basin outflow design and infrequent removal of the solids from the basin.

In order to improve the operation of the sedimentation basin, placement of baffles (precast concrete median barriers) and the installation of new effluent weirs with scum baffles (*Fig. 1*) were completed by March 1996 and, after this point, the large fluctuations of TSS levels ceased. The periodically high levels in wastewater characteristics over the first half of the study are not realistic when compared to estimates from as-excreted levels. To characterise FDMW, therefore, only data from the latter 4 months of the study were utilised.

However, large variations in wastewater parameters are possible on-farm and this presents a sampling challenge in characterising such a waste stream.

Table 2 summarises the levels for cow numbers, water use, wastewater production, and FDMW parameters for the final 4 months of the study. After solids removal by mechanical separation and sedimentation, the FDMW exhibited an average TS level of $3580 \,\mathrm{mg}\,l^{-1}$, an average VS level of $2210 \,\mathrm{mg}\,l^{-1}$, and an average COD-T level of $3530 \,\mathrm{mg}\,l^{-1}$.

Table 3 summarises the levels of wastewater production and FDMW parameters on an AU basis for the last 4 months of the study, and lists corresponding 'as

Table 2				
Characterisation of flush	hed dairy manure wastewat	er		

Parameter	Mean	SD^{a}	Range	
			Min	Max
Lactating cows (mean weight 590 kg), cows d ⁻¹	359	8.2	337	373
Water use, m ³ d ⁻¹	554	27.9	415	643
Wastewater production, m ³ d ⁻¹	502	27.9	364	592
Total solids (TS), $mg l^{-1}$	3580	436	2490	4790
Volatile solids (VS), $mg l^{-1}$	2210	327	1480	3360
Fixed solids (FS), $mg l^{-1}$	1370	148	1000	1820
Total suspended solids (TSS), $mg l^{-1}$	1810	431	877	2950
Volatile suspended solids (VSS), $mg l^{-1}$	1430	366	718	2460
Dissolved solids (DS), $mg l^{-1}$	1770	253	1160	2880
Dissolved volatile solids (DVS), $\operatorname{mg} l^{-1}$	771	220	206	1750
Dissolved fixed solids (DFS), $mg l^{-1}$	996	126	459	1373
Total chemical oxygen demnd (COD-T), $mg l^{-1}$	3530	609	2090	5830
Soluble chemical oxygen demand (COD-S), $\operatorname{mg} l^{-1}$	1310	321	609	2100
Particulate chemical oxygen demand (COD-P), $mg l^{-1}$	2220	564	840	4480
Alkalinity, mg[CaCO ₃] l^{-1}	1270	161	315	1580
Electrical conductivity (EC), μS cm ⁻¹	2670	330	1580	3540
Wastewater temperature, °C	24.2	5.03	11.7	32.4
pH	7.44	0.22	6.73	8.17

^aSD, standard deviation; sample size = 120.

excreted' levels from ASAE (1999) and estimated from DM intake (Van Horn *et al.*, 1998). Average FDMW production amounted to 2370 *l* AU⁻¹ d⁻¹. After mechanical separation and sedimentation, the wastewater contained TS, VS, and COD-T loads of 8·47, 5·23, and 8·36 kg AU⁻¹ d⁻¹, respectively.

In Table 4, the characteristics of FDMW measured in this study are calculated as a percentage of 'as excreted' estimates for this dairy operation. The TS recovered in the wastewater was estimated to be 53% of the TS that was flushed from the barns, indicating that 47% of the TS was removed by mechanical separation and sedimentation. In contrast, VS recovered was only 40% of the 'as excreted' levels, suggesting that the separator and sedimentation basin removed 60% of the VS. A higher removal of VS is expected since dissolved salts pass through separation processes. However, this difference may also be due to an over-estimation of the remaining percentage of TS due to the presence of some fine sand from the bedding, which remained in the wastewater leaving the sedimentation basin. The VS measurement of manure and FDMW is not affected by the presence of sand in the sample since sand does not burn but remains in the fixed solids fraction (ash), whose weight is subtracted from the TS measurement to determine the volatile fraction. In contrast to the VS recovery, the COD-T recovered in the wastewater after screening and sedimentation was much higher at 58% of 'as excreted' levels. As the various chemical constituents that make up the VS exhibit varying COD strengths, this suggests that solids separation tends to remove a VS fraction which has a lower COD strength, leaving a VS fraction with a higher COD strength in the wastewater.

The fractions of soluble to total levels for TS, VS, and COD are also shown in Table 4. While the dissolved fraction of TS was 50%, the dissolved fractions of VS and COD were lower at 36% and 38%, respectively. The VS represented only 62% of the TS in the wastewater, compared to 83% in the 'as excreted' manure. This indicates that a large portion of the fixed solids fraction, much of which is soluble salts and fine sand, remains in the wastewater after separation and sedimentation. Neither VS nor COD measure these soluble fixed solids and the fractions of soluble to total levels for both parameters indicate that a little more than one-third of the degradable compounds were in the soluble phase in the FDMW.

The ratios of COD fractions to corresponding solids fractions are also shown in Table 4. While the 'as excreted' COD-T/VS ratio was 1·1 g[COD] g⁻¹, the same ratio in the FDMW was 1·61 g[COD] g⁻¹. Again, this follows from the earlier observation that VS removed in mechanical separation and sedimentation exhibited lower COD strength and that the VS remaining in the FDMW had much higher COD strength. In fact, the COD strength of the dissolved VS (given by

Table 3
Comparison, on an animal unit basis, of flushed dairy manure wastewater (FDMW) characterisation to 'as excreted' values

Parameter ^a	FDN Present		As excreted	
	Mean	SD^{b}	$\overline{ASAE^{c}}$	Estimated from DM intake ^d
Animal units, AU d ⁻¹	212	4.86		
Wastewater production, $lAU^{-1}d^{-1}$	2370	124	nd ^e	nd
Total solids (TS), kg $AU^{-1}d^{-1}$	8.47	1.09	12.0	15.9
Volatile solids (VS), kg AU ⁻¹ d ⁻¹	5.23	0.81	10.0	13.2
Fixed solids (FS), kg $AU^{-1}d^{-1}$	3.24	0.36	2	2.7
Total suspended solids (TSS), kg AU ⁻¹ d ⁻¹	4.29	1.04	nd	nd
Volatile suspended solids (VSS), kg AU ⁻¹ d ⁻¹	3.40	0.89	nd	nd
Total chemical oxygen demand (COD-T), kg AU ⁻¹ d ⁻¹	8.36	1.50	11.0	14.5
Soluble chemical oxygen demand (COD-S), kg AU ⁻¹ d ⁻¹	3.09	0.77	nd	nd
Alkalinity, $kg[CaCO_3] AU^{-1}d^{-1}$	3.02	0.41	nd	nd

^aAU, animal unit (1000 kg live animal mass).

Table 4
Comparison of flushed dairy manure wastewater (FDMW) characteristics to 'as excreted' levels, soluble to total fractions, and chemical oxygen demand (COD) to solids ratios

Parameter	FDMW Present study		As excreted ^b
	Mean	SD^{a}	
Total solids (TS) recovered, % of 'as excreted' TS	53.3	6.83	na ^c
Volatile solids (VS) recovered, % of 'as excreted' VS	39.6	6.14	na
Total COD (COD-T) recovered, % of 'as excreted' COD-T	57.6	10.3	na
Volatile solids/total solids (VS/TS), %	61.5	2.64	83.0
Dissolved solids/total solids (DS/TS), %	49.9	7.52	nd ^d
Dissolved volatile solids/volatile solids (DVS/VS), %	35.5	10.2	nd
Soluble COD/total COD (COD-S/COD-T), %	37.5	9.39	nd
Total COD/total solids (COD-T/TS), g[COD] g ⁻¹	0.989	0.146	0.913
Total COD/volatile solids (COD-T/VS), g[COD] g ⁻¹	1.61	0.241	1.10
Soluble COD/dissolved volatile solids (COD-S/DVS), g[COD] g ⁻¹	1.92	0.989	nd

^aSD, standard deviation; sample size = 120.

COD-S/DVS) was the highest ratio (1.92 g[COD] g⁻¹), which indicates that the soluble compounds in FDMW have the highest COD strength.

The effect of wastewater production (AU basis) on levels of FDMW parameters for the last 4 months of the study was investigated by performing linear regressions of parameter concentrations on wastewater production. None of the regression coefficients (slopes) were significantly different than zero when evaluated using a Student's *t*-test over the range of wastewater production in the last 4 months of the study (data not shown).

The effect of temperature on FDMW characterisation data was also investigated by performing linear regressions of parameter concentrations on average daily wastewater temperatures (estimated by soil temperature) for the final 4 months, during which the temperature ranged from $11.7\,^{\circ}$ C to $32.4\,^{\circ}$ C. Table 5 shows the regression coefficients, t values, and significance for many of the wastewater parameters and some selected ratios. The regression coefficients for each of the parameters were highly significant except for VS and COD-P, which were not significantly affected by

^bSD, standard deviation; sample size = 120.

^cASAE, 1999.

^dDM, dry matter; Van Horn et al. (1998).

end, no data.

^bVan Horn et al. (1998).

^cna, not applicable.

^dnd, no data.

Table 5
Linear regression coefficients and significance of flushed dairy manure wastewater parameters on temperature

Parameter	Regression coefficient	t^{a}	Significance ^b
Total solids (TS)	25.4, mg l^{-1} °C ⁻¹	3.87	**
Volatile solids (VS)	$10.6, \text{ mg } l^{-1} {}^{\circ}\text{C}^{-1}$	1.80	ns
Fixed solids (FS)	$14.8, \text{ mg } l^{-1} {}^{\circ}\text{C}^{-1}$	6.35	**
Total suspended solids (TSS)	42.8, mg l^{-1} °C ⁻¹	6.27	**
Volatile suspended solids (VSS)	$36.9, \text{ mg } l^{-1} ^{\circ}\text{C}^{-1}$	6.39	**
Dissolved solids (DS)	-17.4 , mg l^{-1} °C ⁻¹	-4.03	**
Dissolved volatile solids (DVS)	-26.3 , mg l^{-1} °C ⁻¹	-8.16	**
Dissolved fixed solids (DFS)	8.83, mg $l^{-1} \circ C^{-1}$	4.10	**
Total COD (COD-T) ^c	40.6 , mg l^{-1} °C ⁻¹	3.87	**
Soluble COD (COD-S)	37.9, mg $l^{-1} \circ C^{-1}$	8.03	**
Particulate COD (COD-P)	$2.70, \text{ mg } l^{-1} ^{\circ}\text{C}^{-1}$	0.26	ns
Alkalinity	-6.85 , mg[CaCO ₃] $l^{-1} \circ C^{-1}$	-2.38	*
Electrical conductivity (EC)	$17.4, \mu \text{S cm}^{-1} {}^{\circ}\text{C}^{-1}$	3.00	**
рН	-0.02 , pH units $^{\circ}$ C ⁻¹	-7.27	**
COD-S/COD-T	0.73, %/°C	4.64	**
DVS/VS	-1.37 , % $'/^{\circ}$ C	-10.05	**
DFS/FS	$-0.17, \%/^{\circ}C$	-1.39	ns
COD-S/DVS	0.125 , g[COD] $g^{-1} \circ C^{-1}$	9.01	**
COD-P/VSS	-0.046 , g[COD] $g^{-1} \circ C^{-1}$	-5.73	**

^aCoefficients with absolute t values greater than critical t values are significant; corresponding critical t values are 2.62 and 1.98 for levels of significance α of 0.01 and 0.05, respectively.

temperature. Total solids, TSS, and VSS levels in the FDMW increased with temperature, while DS and DVS levels decreased.

Although linear regression analysis indicated that there was no significant effect of wastewater production on levels of FDMW parameters, a linear model is not the most appropriate form to measure this effect. For modelling purposes, the manure-handling system is considered as a combination of two discrete unit processes—flushing and solids removal. For a given solids removal efficiency, therefore, the process can be reduced to a dilution model. Equation (1) gives the concentration of a component as a function of the volume of dilution water added:

$$C = \frac{C_0 V_0}{(V_0 + V_A)} \tag{1}$$

where: C is the component concentration after dilution and solids separation, in mg/l; C_0 is the initial concentration of C in 'as excreted' manure, in mg/l; V_0 is the initial volume of C in 'as excreted' manure, in $l \, \text{AU}^{-1} \, \text{d}^{-1}$; and V_{A} is the fresh water volume added, in $l \, \text{AU}^{-1} \, \text{d}^{-1}$.

This dilution equation suggests that an inverse relationship is a more appropriate model for the effect of wastewater production on FDMW components. Inverting and scaling the concentration data can linearise the model equation. This allows the determination of the slope of a linear regression line through the

origin of the transformed data. This approach was used to investigate the relationship between COD-T and wastewater production (AU basis).

The concentration C_0 that the COD-T remaining in the FDMW occupied initially in the 'as excreted' manure prior to solids removal can be estimated by multiplying the 'as excreted' COD-T concentration by the percentage of COD-T recovered in the wastewater. The 'as excreted' COD-T concentration was estimated to be $103\,000\,\mathrm{mg}\,l^{-1}$ (Van Horn *et al.*, 1998) with 57.6% remaining in the wastewater (Table 4). Thus, the initial concentration C_0 in the 'as excreted' manure of the COD-T that was not removed by solids separation was about $59\,400\,\mathrm{mg}\,l^{-1}$. Using this value for C_0 , and linearising the data, resulted in a highly significant regression coefficient ($t=54.5\ versus$ a critical t=2.62 at $\alpha=0.01$).

The volume V_0 that the COD-T remaining in the FDMW occupied initially in the 'as excreted' manure can be estimated from the ratio of the wastewater COD-T levels on an AU basis (Table 3) to the initial concentration C_0 . After solving for a V_0 of $141 I AU^{-1} d^{-1}$, the form of the equation for predicting wastewater COD-T as a function of fresh flushwater use per AU was determined to be:

$$C = \frac{59400 \times 141}{(141 + V_{\rm A})} \tag{2}$$

b** Significant at the $\alpha = 0.01$ level; *Significant at the $\alpha = 0.05$ level; ns, not significant.

^cCOD, chemical oxygen demand.

where: C is the COD-T concentration after dilution and solids separation, in mg/l; and V_A is the fresh water volume added, in $lAU^{-1}d^{-1}$. This equation is valid for flushed dairy manure that undergoes solids removal with similar efficiencies (42.4% COD-T removal) and is useful for predicting the influent COD-T concentrations of FDMW treatment systems, including high-rate anaerobic digestion.

5. Discussion

5.1. Flushed dairy manure production and characteristics

Extensive characterisation of particulate and soluble fractions of wastewater from flushed dairy manure has not been previously reported. However, TS, VS and COD-T of FDMW have been reported and Table 6 compares these values to results from the present study. The mean TS concentration in FDMW in the present study of $3580 \,\mathrm{mg}\ l^{-1}$ is similar to the $3000-4000 \,\mathrm{mg}\ l^{-1}$ range of TS reported for this dairy in 1998 (Sherman *et al.*, 2000). However, this TS level is higher than the $2500 \,\mathrm{mg}\ l^{-1}$ concentration reported for this dairy in September 1994 (Barrow *et al.*, 1997) when a wastewater production level of $1670 \, l\,\mathrm{AU}^{-1}\,\mathrm{d}^{-1}$ was measured, which is below the typical level for flushed dairy manure production in Florida (Van Horn *et al.*, 1993). The

wastewater production level of 2370 l AU⁻¹ d⁻¹ in the present study (Table 6) is slightly higher than typical Florida production levels.

Levels of TS, VS, and COD-T on an AU basis in FDMW were not reported in other studies. However, if an average weight of 409 kg is assumed for a Jersey cow, data from a Jersey-herd dairy (Chastain et al., 2001) can be calculated to give an FDMW total solids level of 11.8 kg AU⁻¹ d⁻¹. The corresponding level for VS is $8.9 \text{ kg AU}^{-1} \text{ d}^{-1}$. Both of these levels are higher than the TS and VS levels found in the present study (Table 3). However, calculating production of TS and VS from Chastain et al. (2001) raw data for flushed dairy manure prior to separation gives production levels of 39.3 and $32.9 \text{ kg AU}^{-1} \text{ d}^{-1}$, respectively, which are more than double typical 'as excreted' production levels. The Chastain et al. (2001) dairy employed copious use of organic bedding material (wood shavings) and this may account for the higher levels of TS and VS in the FDMW after separation and settling.

5.2. Screening and sedimentation

The present study estimated TS, VS, and COD-T removal efficiencies of 46.7%, 60.4%, and 42.4%, respectively, and these are compared to literature values in Table 7. The TS level in the FDMW after mechanical separation and sedimentation reported by Barrow *et al.*

Table 6 Flushed dairy manure wastewater (FDMW) production and characteristics from this study compared to field-scale literature values

Details	from the second section of the section		ng/l	Source	
	iAC u	Total solids	Volatile solids	COD-T ^b	
24h composite sampling after screening and settling	2370	3580	2210	3530	Present study
Grab sampling after screening and settling	nd ^c	3000–4000	nd	nd	Sherman <i>et al.</i> (2000)
Grab sampling after screening and settling	1670	2500	nd	nd	Barrow et al. (1997)
Grab sampling after screening	nd	9600-33 300	6200-24800	nd	Stowell and McKenney (1998)
Grab sampling after screening with some recycled flushwater	nd	10 000–32 000	nd	nd	Moore and Miner (1985)
Grab sampling after screening with some recycled flushwater	2260	nd	3200	3000	Williams and Frederick (2001)
Grab sampling	nd	6000	4200	5000	NRCS (1992)
Composite sampling with recycled flushwater	762	11 500	9800	nd	Hills and Kayhanian (1985)
Composite sampling after screening and settling	1030	11 500	8710	nd	Chastain et al. (2001)
Florida dairies	1010-2090	nd	nd	nd	Van Horn et al. (1993)

^aAU, animal unit (1000 kg live animal mass).

^bCOD-T, total chemical oxygen demand.

^cnd, no data.

Separation type		Removal efficie	Source	
	Total solids	Volatile solids	Chemical oxygen demand	
Screening and settling	46.7	60-4	42.4	Present study
Screening and settling	55.3	54.3	nd^a	Barrow <i>et al.</i> (1997)
Screening and settling	70	72.9	nd	Chastain et al. (2001)
Screening only	8–16	nd	3–12	Hegg et al. (1981)
Screening only	32.1	nd	nd	Barrow <i>et al.</i> (1997)
Screening only	45.5	50.1	nd	Fulhage and Hoehne (1998)
Screening only	55-74	57–75	41–68	Graves et al. (1971)

66.5

nd

62.8

48

Table 7
Separation removal efficiencies for flushed dairy manure estimated in the present study compared to field-scale literature values

Screening only Settling only

(1997) was lower than levels measured in the present study and this could account for the higher levels of TS removal found in their study. In both screening and sedimentation, there appears to be a trend that VS removal rates are higher than TS removal rates. Since soluble salts, which make up a significant fraction of the TS and yet are not a part of the VS, are left in the liquid phase after separation, a higher VS removal is expected. Except for the results reported by Chastain *et al.* (2001), there is also a trend that COD removal is lower than either TS or VS removal. This again suggests that solids separation removes a VS fraction that has a lower COD strength and, therefore, leaves a VS fraction in the FDMW that has a higher COD strength.

60.9

nd

5.3. Solids and chemical oxygen demand fractions

The ratio of COD to solids fractions directly measures the potential energy content of the components in these fractions. The COD-T to TS fraction is highly variable since dissolved salts and sand included in the TS measurement do not contribute to the COD. Fresh manure exhibited a COD-T to TS ratio of 0.913 g[COD] g⁻¹ (Van Horn *et al.*, 1998), while the present study measured this ratio at 0.989 g[COD] g⁻¹ in FDMW (Table 4). Comparable ratios for FDMW from the literature include: 1.15–1.52 g[COD] g⁻¹ after screening (Hegg *et al.*, 1981); 0.83 g[COD] g⁻¹ in milk house and parlour wastewater (NRCS, 1992); and 0.90 g[COD] g⁻¹ after field screening and 60 min laboratory-scale sedimentation (Chastain *et al.*, 2001).

The ratio of COD-T to VS should be more consistent as sand and salts do not influence the VS measurement. However, this was not found to be the case, possibly due to differences in bedding material and separation efficiencies. For fresh manure, the COD-T to VS ratio was 1·1 g[COD] g⁻¹ (Van Horn *et al.*, 1998), while the present study measured this ratio at 1·61 g[COD] g⁻¹ in

FDMW (Table 4). Comparable ratios for FDMW from the literature include: 0.94 g[COD] g⁻¹ after screening (Williams & Frederick, 2001); 1.19 g[COD] g⁻¹ in milk house and parlour wastewater (NRCS, 1992); and 1.02 g[COD] g⁻¹ after field screening and 60 min laboratory-scale sedimentation (Chastain *et al.*, 2001). Since no other studies reported soluble COD, there are no comparable values available for the COD-S to DVS measured in this study to confirm the higher ratio found in the soluble fraction.

Chastain et al. (2001)

Barrow et al. (1997)

5.4. Effect of temperature on wastewater parameters

Both physicochemical and biological phenomena explain the effect of temperature on FDMW parameters (Table 5). The efficiencies of mechanical separation and sedimentation of manure solids are affected by temperature, as the viscosity of water changes and the solubility of salts is also affected. The solubility of most salts increases with temperature (Snoeyink & Jenkins, 1980) and this is shown by a large positive regression coefficient for the increase in DFS in the FDMW with increased temperature (Table 5). The FS also increased. However, the additional FS released by hydrolysed particulates explains this. As temperature increases, the biological activity in settled manure solids increases and the resulting gas production floats some of the particulate matter and increases the suspended solids levels in the wastewater. In addition, as temperature increases, the microbial hydrolysis of particulate matter increases and transfers some of the hydrolysis products to the soluble phase. Higher FDMW temperatures, therefore, reduce separator and sedimentation removal efficiencies, which increases the levels of FDMW parameters.

Many of the effects of temperature on FDMW parameters counter each other and are complicated by measurement limitations, including inaccuracies in the

and, no data.

VS measurements due to a loss of small volatile organic compounds during drying of the sample for TS analysis. The COD procedure, which is conducted in a sealed tube, minimises volatile losses and so is more accurate for measurement of smaller volatile compounds in the soluble phase. Indeed, while DVS levels in FDMW decreased with increasing temperature, COD-S levels increased with temperature (Table 5), suggesting that the DVS measurement was not detecting many of the soluble phase compounds. Finally, the organic strength of soluble products is affected by the profile of fermentation products, which may be influenced by temperature. For example, while acetate has a COD/VS ratio of 1.06 g[COD] g⁻¹, butyrate has a COD/VS ratio of 1.82 g[COD] g⁻¹.

Both COD-T and COD-S concentrations in the FDMW increased with temperature. While the COD-S as a percentage of COD-T increased with temperature, the DVS as a percent of VS decreased (Table 5). This suggests that either volatile components were lost during DVS determination or the DVS measurement failed to measure the increase of more reduced (higher COD strength) organic compounds that are formed at higher wastewater temperatures. The increase in COD-S/DVS ratio and decrease in COD-P/VSS ratio, as temperature increased, suggest that hydrolysis of the particulate fraction was significant at higher temperatures. The fact that VS and COD-P levels were not significantly affected by temperature indicates that competing effects were involved. None of the other studies referred to have reported the changes in FDMW parameters as affected by ambient temperature variations.

The soluble compounds found in dairy manure, which are responsible for odours, were reviewed by Mackie et al. (1998) and include volatile organic acids, amines, indoles, phenols, and sulphur compounds. Most of the phenolic compounds, mainly p-cresol, originate from urine but degradation of manure proteins during storage may produce additional phenolic compounds. Powers et al. (1999) found increased levels of phenol, acetate, and propionate in diluted manure after 1 day of storage at room temperature. Phenol has a COD/VS ratio of 2.38 g[COD] g⁻¹ and could cause significant changes in the COD-S/DVS ratio. In addition, spilled and waste milk sometimes entered the FDMW in the present study and would have impacted the COD-S concentration. In the current manure-handling system, the average retention time in the sedimentation basin would be 3.7 h based on the daily flow rate. While increased volatility of soluble components is expected at higher temperatures and often detected in increased levels of odour production, biological activity also increases the level of volatile compounds in the soluble phase by hydrolysis of particulate matter.

5.5. Predicting flushed dairy manure wastewater characteristics

Using the average milk production (27·3 kg cow⁻¹), wastewater production, and TS, VS, and COD-T production in the FDMW, a wastewater volume coefficient and wastewater strength coefficients can be calculated for the current site. The wastewater volume coefficient was found to be 51·35 kg[wastewater] kg⁻¹[milk]. This level should be similar at dairies using similar amounts of water on an AU basis. Comparable levels calculated from Van Horn *et al.* (1993) for dairies using manure-flushing systems were 26 kg[wastewater] kg⁻¹[milk] for minimum water usage levels to 53 kg [wastewater] kg⁻¹[milk] at dairies with common water usage levels.

The wastewater strength coefficients for TS, VS, and COD-T were 184, 113, and 181 kg per 1000 kg milk production, respectively. These levels should be similar at dairies with 100% confinement of the milking herd that are using fresh water for flushing (3 times per day), solids separation with similar removal efficiencies, and not using organic bedding materials. When dry cows and replacement heifers are included in the confinement barns, these strength coefficients are increased by approximately 16.7% since the dry herd is often onethird of the milking herd and consumes about half of the DM intake of the milking herd. Wastewater strength coefficients for TS and VS were also calculated from milk production versus manure solids production found in NRCS (1992), which yielded values of 290 kg TS and 247 kg VS per 1000 kg milk production. However, these levels use total manure solids production levels without solids removal and are based on low estimates of manure production that were not based on DM intake. No other comparable wastewater strength coefficients were found in the literature reviewed.

Equation (2) uses fresh flushwater usage on an AU basis to predict the COD-T concentration in the wastewater for a dairy with 100% confinement of the milking herd and solids removal systems with similar removal efficiencies. Figure 3 shows the plot of this curve with data from the current study and from the literature. The COD-T datum from Williams and Frederick (2001) of $3000 \,\mathrm{mg}\,l^{-1}$ lies close to the predicted estimate of $3490 \text{ mg } l^{-1}$. While the $20 100 \text{ mg } l^{-1}$ from Chastain *et al*. (2001) is almost 3-fold the $7150 \,\mathrm{mg}\,l^{-1}$ COD-T level predicted by the equation, the reported datum is after screening (no COD levels after field-scale sedimentation were given). The dairy studied in Chastain et al. (2001) used large quantities of organic bedding material, which also contributes to the failure of the prediction equation. Finally, the estimated COD-T of $2470 \,\mathrm{mg}\,l^{-1}$ from Barrow et al. (1997) was calculated by multiplying the

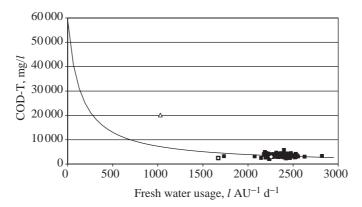


Fig. 3. Prediction of total chemical oxygen demand (COD-T) concentration of flushed dairy manure wastewater as a function of fresh water usage (AU=animal unit, 1000 kg live animal mass): —, calculated; \blacksquare , actual; \triangle , Chastain et al., 2001; \diamondsuit , Williams and Frederick, 2001; \square , Barrow et al., 1997

given wastewater TS concentration by the COD-T/TS ratio found in Table 4. This value is low compared to the predicted COD-T level of $4620 \text{ mg } l^{-1}$, though this may be due to unrepresentative sampling of FDMW after screening and sedimentation.

The existing studies in the literature rarely give cow numbers and water use levels along with the FDMW characteristics. In the present study, frequency and duration of flushing events at the dairy were controlled manually and no effort was placed in optimising flushing during the study. Automated control of flushing events can be used to increase the COD-T in the FDMW and lower freshwater consumption.

5.6. Implications of wastewater characterisation for anaerobic digestion

In the present study, 60.4% of the VS was found to be removed by screening and sedimentation (Table 4). Fixed-film anaerobic digestion of the resulting wastewater removes 40% of the remaining VS (Davis, 2001), which is equivalent to degrading 16% of the raw manure VS. If it is assumed that 40% of the raw manure VS is degradable, then fixed-film digestion of the separated wastewater is degrading 40% of the degradable VS. However, this belies the true recovery of methane (CH_4) potential since the COD of the VS in the FDMW was higher than that in the raw wastewater. Screening and sedimentation removed only 42.4% of the COD-T (Table 4). Fixed-film digestion of the FDMW has resulted in a 48% COD reduction (Davis, 2001), which corresponds to a 27.6% reduction of the COD in the 'as excreted' manure. If it is assumed that 40% of the 'as excreted' COD is degradable, then this conversion amounts to 69% of the degradable COD. Thus, the major portion of the CH₄ potential remains in the FDMW after screening and sedimentation.

Further evidence that a significant portion of the CH₄ potential remains in the FDMW is provided by the CH₄ production on a per cow basis. Vetter et al. (1990) found that a farm-scale mixed reactor processing whole manure at a 24d HRT and 37 °C produced CH₄ at a rate of $2.25 \,\mathrm{m}^3 \,\mathrm{AU}^{-1} \,\mathrm{d}^{-1}$. Also, Martin et al. (2003) found that a farm-scale plug-flow reactor processing whole manure at a 34d HRT and 35 °C produced CH₄ at a rate of $2.27 \,\mathrm{m}^3 \,\mathrm{AU}^{-1} \,\mathrm{d}^{-1}$. In July 2000, the fixedfilm digester at the present site produced biogas at a rate of $170 \,\mathrm{m}^3 \,\mathrm{d}^{-1}$ with an 80% CH₄ content (Wilkie, 2000). At that time, the digester was operating at ambient temperature (27 °C) on a 3.75 d HRT and was only processing 25% of the FDMW from 500 cows. Converted to methane, this is equivalent to a CH₄ production level of 1.84 m³ AU⁻¹ d⁻¹, which is 81–82% of the CH₄ production per animal unit reported by Vetter et al. (1990) and Martin et al. (2003). This supports the conclusion that the larger portion of the methane potential is retained in the FDMW after screening and sedimentation. Since sand is used for bedding at the dairy in the present study, a sedimentation step is required to remove additional sand from the FDMW. However, dairies using organic or mat bedding may avoid extensive settling and only screen out the non-degradable fibres such that most of the methane potential is retained in the FDMW, while the problematic solids are removed.

Generally, the effect of wastewater temperature on FDMW parameters has a positive impact on fixed-film digestion of this wastewater. Increased levels of COD-T at higher wastewater temperatures (Table 5) are accommodated by increased biological activity in the reactor. At lower temperatures, the level of COD-T in FDMW after screening and sedimentation drops and,

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for the same flow rate, this will lower the organic loading rate of the digester. Since fixed-film digestion of FDMW occurs at ambient temperatures, this tends to attenuate the organic loading rate when low temperatures are already reducing the biological activity in the digester. The COD-S proportion of COD-T also decreases as temperature falls, which lowers the COD-S loading rate (less volatile acids), again favouring the lower biological activity in the reactor at these lower temperatures. Thus, a lower biogas production at lower temperatures is not only due to lower activity but also due to lower wastewater strength as a result of increases in separation efficiencies.

6. Conclusions

Fixed-film anaerobic digestion offers a sustainable alternative to treat the liquid fraction of flushed dairy manure, providing major benefits in terms of energy production, waste stabilisation and odour control, and pathogen reduction, while conserving the fertiliser value of the wastewater. However, the lack of data characterising flushed dairy manure wastewater (FDMW) is a major impediment to implementing this approach. With the aim of developing fixed-film anaerobic digestion of FDMW, the characteristics of FDMW after primary treatment were monitored daily over a 1-year period along with cow numbers and water use. Primary treatment included screening through an Agpro® separator followed by sedimentation. Large fluctuations and high levels in total solids (TS) and chemical oxygen demand (COD) were attributed to the accumulation of solids in the settling basin and periodic losses of fugitive suspended solids into the wastewater. The installation of baffles and effluent weirs with scum baffles in the sedimentation basin significantly improved the consistency of FDMW parameters for the last 4 months of the study.

For a milking herd average of 359 cows and a wastewater production of $502\,\mathrm{m}^3\,\mathrm{d}^{-1}$, the FDMW exhibited average TS, volatile solids (VS), and total COD levels of 3580, 2210, and 3530 mg l^{-1} , respectively. Analyses of FDMW parameters and manure excretion estimates indicated that 53% of the TS, 40% of the VS, and 58% of the total COD remained in the wastewater after screening and sedimentation. The separation of fibrous solids increased the COD to VS ratio of the FDMW.

Wastewater temperature was found to significantly affect both the separation of wastewater TS, VS, and total COD and the partitioning of wastewater parameters between the particulate and soluble phases. Higher average FDMW temperatures tended to increase

levels of parameters in the FDMW by reducing TS, VS and total COD removal efficiencies. The ratio of soluble COD to dissolved VS increased with temperature, while the ratio of particulate COD to volatile suspended solids decreased, indicating that significant levels of biological activity occurred in the sedimentation basin at higher temperatures.

The VS measurement does not accurately reflect the methane potential of components in FDMW compared to COD measurements. In spite of the fact that 60% of the VS is removed by screening and sedimentation, the major portion of the methane potential remains in the wastewater, since the solids that are removed are relatively non-degradable fibres and do not contribute to methane production. Model equations were developed to predict total COD levels in FDMW as a function of fresh flushwater usage on an animal unit basis for similar dairy operations, providing an important design parameter for implementation of fixed-film anaerobic digestion systems.

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References

APHA (1995). Standard Methods for the Examination of Water and Wastewater, 19th Edn. American Public Health Association, Washington, DC

ASAE (1999). Manure production and characteristics. ASAE D384.1 DEC98. In: ASAE Standards 1999, pp 663–665. American Society of Agricultural Engineers, St Joseph, MI

AWARDS (1996). Agricultural Weather Automatic Retrieval Display System, http://www.agen.ufl.edu/~wethr. University of Florida, Gainesville, FL

Barrow J T; Van Horn H H; Anderson D L; Nordstedt R A (1997). Effects of Fe and Ca additions to dairy wastewaters on solids and nutrient removal by sedimentation. Applied Engineering in Agriculture, 13(2), 259–267

Brodie H (1989). Irrigation systems for dairy manure. In:
Dairy Manure Management, NRAES-31, pp 195–203.
Northeast Regional Agricultural Engineering Service,
Cornell University, Ithaca, NY

Chastain J P; Vanotti M B; Wingfield M M (2001). Effectiveness of liquid–solid separation for treatment of flushed dairy manure: a case study. Applied Engineering in Agriculture, 17(3), 343–354

Davis J A (2001). Bacterial and viral indicator and pathogen reduction in the liquid fraction of flushed dairy manure by

- anaerobic digestion using a demonstration-scale fixed-film digester. MS Thesis. University of Florida, Gainesville, FL
- **Fulhage C D; Hoehne J A** (1998). Performance of a screen separator for flushed dairy manure. In: Fourth International Dairy Housing Conference (Chastain J P, ed), pp 130–135. American Society of Agricultural Engineers, St Joseph, MI
- Graves R E; Clayton J T; Light RG (1971). Renovation and reuse of water for dilution and hydraulic transport of dairy cattle manure. In: Livestock Waste Management and Pollution Abatement: Proceedings of the International Symposium on Livestock Wastes, pp 341–344. American Society of Agricultural Engineers, St Joseph, MI
- Haugen V J; Lindley J A (1988). Biogas production after solid-liquid separation of dairy manure. Transactions of the ASAE, 31(6), 1782–1786
- Hayes T D; Jewell W J; Dell'Orto S; Fanfoni K J; Leuschner A P; Sherman D F (1980). Anaerobic digestion of cattle manure. In: Anaerobic Digestion (Stafford D A; Wheatley B I; Hughes D E, eds), pp 255–285. Applied Science Publishers Ltd., London
- Hegg R O; Larson R E; Moore J A (1981). Mechanical liquid–solid separation in beef, dairy and swine waste slurries. Transactions of the ASAE, 24(1), 159–163
- Hill D T (1991). Steady-state mesophilic design equations for methane production from livestock wastes. Transactions of the ASAE, 34(5), 2157–2163
- Hills D J; Kayhanian M (1985). Methane from settled and filtered flushed dairy wastes. Transactions of the ASAE, **28**(3), 865–869
- Lo K V; Bulley N R; Liao P H; Whitehead A J (1983). The effect of solids-separation pretreatment on biogas production from dairy manure. Agricultural Wastes, 8(3), 155–165
- Mackie R I; Stroot P G; Varel V H (1998). Biochemical identification and biological origin of key odor components in livestock waste. Journal of Animal Science, 76(5), 1331–1342
- Martin Jr J H; Wright P E; Inglis S F; Roos K F (2003). Evaluation of the performance of a 550 cow plug-flow anaerobic digester under steady-state conditions. In: Animal, Agricultural and Food Processing Wastes IX: Proceedings of the Ninth International Symposium, pp 350–359. American Society of Agricultural Engineers, St Joseph, MI
- Mattocks R P; Moser M A (2000). Fate of incoming solids to measure manure digester performance. In: Animal, Agricultural and Food Processing Wastes: Proceedings of the Eighth International Symposium (Moore J A, ed), pp 187–193. American Society of Agricultural Engineers, St Joseph, MI
- Moore J A; Miner J R (1985). Wastewater characteristics of a flushing dairy. In: Agricultural Waste Utilization and Management: Proceedings of the Fifth International Symposium on Agricultural Wastes, pp 606–612. American Society of Agricultural Engineers, St Joseph, MI
- Morse D; Nordstedt R A; Head H H; Van Horn H H (1994).

 Production and characteristics of manure from lactating dairy cows in Florida. Transactions of the ASAE, 37(1), 275–279
- NRCS (1992). National Engineering Handbook Part 651: Agricultural Waste Management Field Handbook. US Department of Agriculture, Natural Resources Conservation Service, Washington, DC
- Pain B F; West R; Oliver B; Hawkes D L (1984). Mesophilic anaerobic digestion of dairy cow slurry on a farm scale: first

- comparisons between digestion before and after solids separation. Journal of Agricultural Engineering Research, **29**(3), 249–256
- Powers W J; Montoya R E; Van Horn H H; Nordstedt R A; Bucklin R A (1995). Separation of manure solids from simulated flushed manures by screening or sedimentation. Applied Engineering in Agriculture, 11(3), 431–436
- Powers W J; Van Horn H H; Wilkie A C; Wilcox C J; Nordstedt R A (1999). Effects of anaerobic digestion and additives to effluent or cattle feed on odor and odorant concentrations. Journal of Animal Science, 77(6), 1412–1421
- Safley Jr L M; Barker J C; Westerman P W (1984). Characteristics of fresh dairy manure. Transactions of the ASAE, 27(4), 1150–1153, 1162
- Sherman J J; Van Horn H H; Nordstedt R A (2000). Use of flocculants in dairy wastewaters to remove phosphorus. Applied Engineering in Agriculture, 16(4), 445–452
- Snoeyink V L; Jenkins D (1980). Water Chemistry. John Wiley & Sons, New York, NY
- **Stowell R R; McKenney A** (1998). Solids content analysis of dairy farm flushwater in storage and at critical points of the waste stream. ASAE Paper No. 98-4120. American Society of Agricultural Engineers, St Joseph, MI
- **Tefertiller K R; Jauregui C; Olexa M T** (1998). Impact of the regulatory environment facing Florida dairy farmers. Circular 1208. Florida Cooperative Extension Service, University of Florida, Gainesville, FL
- Van Horn H H; Bray D R; Nordstedt R A; Bucklin R A; Bottcher A B; Gallaher R N; Chambliss C G; Kidder G (1993). Water budgets for Florida dairy farms. Circular 1091. Florida Cooperative Extension Service, University of Florida, Gainesville, FL
- Van Horn H H; Wilkie A C; Powers W J; Nordstedt R A (1994). Components of dairy manure management systems. Journal of Dairy Science, 77(7), 2008–2030
- Van Horn H H; Newton G L; Nordstedt R A; French E C; Kidder G; Graetz D A; Chambliss C F (1998). Dairy manure management: strategies for recycling nutrients to recover fertilizer value and avoid environmental pollution. Circular 1016. Florida Cooperative Extension Service, University of Florida, Gainesville, FL
- Vetter R L; Friederick D J; Huntington P (1990). Full scale anaerobic digester and waste management system for a 300 cow dairy. In: Agricultural and Food Processing Wastes: Proceedings of the Sixth International Symposium, pp 236–249. American Society of Agricultural Engineers, St Joseph, MI
- Wilkie A C (2000). Fixed-film anaerobic digester: reducing dairy manure odor and producing energy. BioCycle, 41(9), 48–50
- Wilkie A C (2003). Anaerobic digestion of flushed dairy manure. In: Proceedings—Anaerobic Digester Technology Applications in Animal Agriculture—a National Summit, pp 350–354. Water Environment Federation, Alexandria, VA
- Wilkie A C; Riedesel K J; Cubinski K R (1995). Anaerobic digestion for odor control. In: Nuisance Concerns in Animal Manure Management: Odors and Flies (Van Horn H H, ed), pp 56–62. Florida Cooperative Extension Service, University of Florida, Gainesville, FL
- Williams D W; Frederick J J (2001). Microturbine operation with biogas from a covered dairy manure lagoon. ASAE Paper No. 01-6154. American Society of Agricultural Engineers, St Joseph, MI