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Biochemical methane potential of dairy manure residues and separated fractions: An Australia-wide study of the impact of production and cleaning systems

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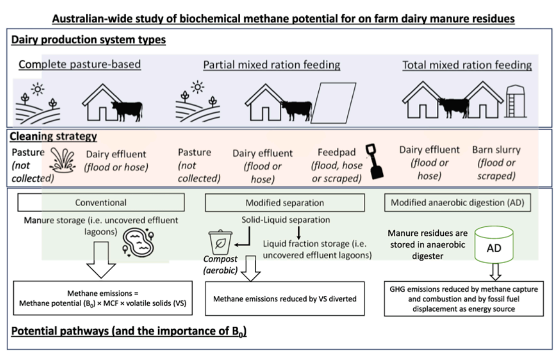
HIGHLIGHTS

- First report of biochemical methane potential (B_0) for grazing dairy effluent.
- B_0 of grazing dairy effluent is 161 $L_{CH4}\cdot kg_{VS}^{-1}$.
- B_0 of intensive dairy (barn) effluent is 202 $L_{CH4}\cdot kg_{VS}^{-1}$.
- Manure solids content is affected by cleaning method; and amount by capture extent.
- Mechanical manure separation can reduce fugitive methane losses from effluent ponds.

GRAPHICAL ABSTRACT



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ABSTRACT

This study investigated biochemical methane potential (B_0) of manure residues and solid–liquid separation fractions from Australian dairies. This is important for country-specific sector emissions and biogas potential estimates. A range of samples were collected from 12 farms across 4 Australian states, and B_0 was measured. A first B_0 value for grazing dairy effluent is reported, at 161 $L_{CH4}\cdot kg_{VS}^{-1}$. The B_0 of manure residues from intensive dairies with total mixed ration feeding was not significantly different, at 202 $L_{CH4}\cdot kg_{VS}^{-1}$. Passive solid–liquid separation decreased B_0 with potential fugitive methane losses. Mechanical separation preserved B_0, allowing organic matter diversion to reduce fugitive methane emissions. Cleaning method at a dairy significantly influenced residue total solids content, important for solid–liquid separation and selection of anaerobic digestion technology. Overall, B_0 for Australian dairy residues was estimated at 76.2 million m_N^3 methane per annum, with a total energy content of 2.8 petajoules·annum^{-1}.

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1. Introduction

Dairy farming is a major agricultural sector, with a significant global importance for food production and responsibility to minimise environmental impacts (Soteriades et al., 2020). The dairy industry is also important in Australia, comprised of 4,618 dairy farms with an average herd size of 300 cows, generating yearly about 8.9 billion litres of milk and 4.7 billion Australian dollars in farmgate value (Dairy Australia, 2021). One key aspect of dairy farming is manure management, important for sustainability and minimising negative environmental impacts (Laubach et al., 2015; Sudmeyer, 2021).

Substantial amounts of manure residues are produced by dairy farming annually, and if these are not properly managed, their nutrient contents such as nitrogen and phosphorus can adversely affect surface waters (Gourley et al., 2012; Jackson, 2020) and groundwater. Furthermore, dairy manure storage as an effluent or slurry, such as in uncovered effluent holding ponds, can be a significant source of fugitive methane (CH_4) as a potent greenhouse gas (Laubach et al., 2015). Manure-management CH_4 was identified as an important mitigative target to address climate change (Smith et al., 2007).

Dairy manure residues can however also be viewed as a valuable resource. For example, its nutrient content has long been used on-farm to reduce synthetic fertiliser use, and to potentially enhance soil health (Rayne & Aula, 2020). Additionally, anaerobic digestion (AD) is a mature bioprocess technology that converts organic matter (such as in manure) into progressively simpler metabolic intermediates and ultimately into CH_4 and carbon dioxide (CO_2) in biogas (Batstone & Jensen, 2011). Organic matter is degraded by anaerobic digestion via four main biological steps: hydrolysis, acidogenesis, acetogenesis, and methanogenesis (Weiland, 2010). Biogas from AD can be captured and used as a renewable energy source (Abbasi et al., 2012; Weiland, 2010), both to prevent fugitive CH_4 emissions from manure management, as well as enable the displacement of fossil fuel energy (Abbasi et al., 2012). In this way, AD could be one of the most efficient technologies to reduce the carbon footprint of dairy manure management (Belflower et al., 2012).

Dairy production types can have important implications for manure management, its associated environmental practices (Soteriades et al., 2020), and AD options. Specifically, with pasture-based dairies common to Australia, New Zealand and Ireland (Moscovici Joubran et al., 2021), cows graze in paddocks for most of their daily feeding. Pasture-based (PB) systems are often seen as sustainable from an environmental and welfare perspective (Moscovici Joubran et al., 2021), with reduced feed inputs and greenhouse gas (GHG) emissions from manure management (Latham, 2010). However, PB systems can also have environmental challenges, including potential for overgrazing, erosion, and manure nutrient accumulation and leaching in paddocks if not properly managed (Rojas-Downing et al., 2017). Additionally, extreme weather conditions such as drought and floods can restrict the grazing capability and accessibility of land, causing farmers to adjust their PB herd size in response to such conditions rather than milk demand. As a consequence, Australian dairy production is increasingly considering and adopting intensive feeding systems (Watson & Watson, 2015), where a majority proportion of the daily ration is fed to cows on feedpads or housed in barns or freestalls (Dairy Australia and Agriculture Victoria, 2023; Tait et al., 2021). These are commonly termed partial mixed ration (PMR) where some grazing still occurs, or total mixed ration (TMR) systems without significant grazing. Intensive systems can be more efficient in terms of feed conversion and milk production (Fontaneli et al., 2005) and can improve commercial performance and climate resilience (Dairy Australia and Agriculture Victoria, 2023; Tait et al., 2021). However, production types (i.e., PB vs. PMR vs. TMR) also affect manure capture. For example, with PB, most of the cow's daily manure output is excreted directly onto pastures and is therefore not captured. Only a minor proportion of manure excreted is captured during milking with some feed, milk spillage and cleaning chemicals, then producing a dilute effluent with the typically large amount of cleaning water used (Tait et al., 2021). In contrast, intensive dairies capture a much larger proportion of daily excreted manure on feedpads and/or housing floors. This additional manure then needs to be carefully managed as a potential point source of nutrients, and may increase fugitive CH_4 from manure-management (Williams et al., 2020). However, additional manure capture can also represent a higher biogas energy potential.

Differences in manure collection (including cleaning systems) can influence the characteristics of the collected manure residues, and this has important implications for AD technology selection. For example, because the effluent collected from PB systems is often highly diluted with a low total solids (TS) content (Grell et al., 2023; Tait et al., 2021), a larger digester size may then be required to retain the particulate organic matter in dairy manure for long enough to be converted into biogas. This is because the overall AD rate kinetics for particulate substrates can be limited by hydrolysis (Batstone & Jensen, 2011; Batstone et al., 2009). Covered anaerobic ponds are often considered for minimising capital expense when large digester sizes are required. In PMR and TMR, the additional manure and spilt feed from feedpads or barns/freestalls may increase TS in the collected manure residues. This may allow use of continuous stirred tank reactors (CSTRs), generally easier to control with heating and mixing to enhance biogas production. Importantly, TS is affected by water use for different cleaning methods, such as with (Birchall et al., 2008): hose cleaning with moderate-to-high pressure water (here termed Hose); floodwash systems where a large wave of water is released over a short period of time to wash manure off floor surfaces (here termed Flood); or scraping or vacuuming of excreted manure from surfaces as a slurry or semi-solid using machinery with minimal water use (here termed Scraped). However, some minimum amount of water may still be required to effectively clean a dairy, thereby limiting the TS content in the effluent. This could be partly why covered effluent ponds have been the most common digestion technology to date for cattle manure in the United States (AgSTAR, 2022). The connection between water use for cleaning, TS in manure residues collected, and implications for AD technology selection, have not been previously explored in the published literature.

An alternative to using low-rate covered anaerobic pond technology, is to separate out and concentrate manure from a dilute slurry/effluent by solid-liquid separation. This could help overcome the hydraulic limitations of CSTRs (Batstone & Jensen, 2011). Several solid–liquid separation systems have been commonly used for dilute dairy manure (Hjorth et al., 2010) and can be categorised as active or passive separation. Passive separation includes sedimentation in basins or holding ponds, or via trafficable solids traps with weeping walls (Mukhtar et al., 2011). With passive systems, solids settle out under gravity and are retained, often over extended time periods of weeks to months. Importantly, due to the likely prevailing anaerobic conditions and typical extended manure storage times in passive separation systems, CH_4 potential may be lost resulting in significant fugitive emissions. For example, trafficable solids traps are concrete in-ground pits with an access ramp, and a slatted weeping wall to retain manure solids and allow liquid to pass. Storage of the retained solids in the trap for extended periods as a slurry leads to anaerobic conditions and potentially substantial fugitive CH_4 emissions (Hull-Cantillo et al., 2023). In contrast, active separation refers to mechanical separation, using a centrifuge, screw press, screen, or other technology (Hjorth et al., 2010). Mechanical separation is usually rapid and the separated solids subsequently stored aerobically or composted (Zhang et al., 2022), thereby discouraging fugitive CH_4. Anaerobic digestion of the separated solid and liquid fractions has been previously explored (Rico et al., 2012; Rico et al., 2007). However, there has been limited attention given to potential CH_4 yield loss and fugitive CH_4 emissions potential from passive and active solid-liquid separation.

Biochemical methane potential (B_0) is an essential quantitative parameter used to assess CH_4 losses across manure management systems (e.g., solid–liquid separation), and also to evaluate biogas energy potential from AD. A previous *meta*-analysis of 115 articles and 2,181 cases on B_0 of dairy cattle manure found that the mean B_0 for all regions was 198 $L_{CH4}\cdot kg_{VS}^{-1}$ (Miranda et al., 2016). However, the same study found that values differed between continents (e.g., 220 $L_{CH4}\cdot kg_{VS}^{-1}$ for Asia/Middle East & the Indian Subcontinent; 195 $L_{CH4}\cdot kg_{VS}^{-1}$ for Europe; 280 $L_{CH4}\cdot kg_{VS}^{-1}$ for North America; and 100 $L_{CH4}\cdot kg_{VS}^{-1}$ for Africa) (Miranda et al., 2016). This could be due to several factors such as feed type, manure management practices, and climate. The IPCC (2006) suggests that ideally country-specific B_0 values should be measured and applied. However, there are currently no published values for B_0 of on-farm manure residues in Australian dairies. This is important because it means that Australia's National Inventory (NGER, 2022) has to date been using the IPCC default value of 240 $L_{CH4}\cdot kg_{VS}^{-1}$ for dairy cattle manure, and industry reference guidelines have been reporting international B_0 values (Birchall et al., 2008). With PB dairies still dominant in Australia, the manure from grazed feed would be expected to contain high concentrations of recalcitrant lignocelluloses. Conversely, with emerging intensive feeding systems, the manure may be influenced by a more energy-dense ration diet (Labatut et al., 2011). The current lack of B_0 values for dairy manure residues in Australia is a key gap, as it would influence country-specific estimates of dairy sector emissions and biogas energy potential. This is especially important because AD is not yet widely applied across the Australian dairy sector (Tait et al., 2021), and the lack of B_0 values affects evaluation of future opportunities.

The aim of the current study was to measure B_0 values for manure residues from dairies across Australia, including a range of production systems to evaluate manure capture, and cleaning systems to evaluate influence on TS. These data are then used to assess and discuss the potential for manure-derived CH_4 emissions, including with solid–liquid separation, to discuss AD technology suitability, and to evaluate biogas energy potential across the Australian dairy sector.

2. Materials and methods

2.1. Sample collection

Manure and effluent samples were sourced from 12 different dairy farms across four important dairy states of Australia, namely, Queensland (QLD), Western Australia (WA), Victoria (VIC), and New South Wales (NSW). In total, 29 different dairy manure residue types were collected from a diverse range of production types (6 PB, 3 PMR, 3 TMR). A third (4/12) of the investigated farms had solid–liquid separation; specifically, Farms 1 and 12 used mechanical separation, and Farms 2 and 6 used passive separation in trafficable solids traps with a weeping wall. At Farm 12, an inclined screen was used, but the separated liquid fraction drained via an inaccessible underground pipe directly into an uncovered holding pond. Consequently, only inflow effluent prior to separation and the separated solids fraction could be sampled at this farm. It is noted that the relative prominence of solid–liquid separation at the sampled farms was not reflective of the broader Australian industry but provided diversity of investigations. Detailed information of the sampled farms and their manure collection and management systems, are listed in Table 1. Systems terminology in Table 1 aligns with IPCC (2006) definitions.

2.2. Sampling procedure

Raw manure residue samples (i.e., prior to any separation) were typically collected as an effluent at the outflow of the dairy shed, feedpad or housing complex, during a washdown event, and were comprised of a composite of approximately evenly timed grab samples, of about 20 L in total. To prevent the settling of solids, the aggregated composite sample was stirred continuously with a paint mixer (Model 257 Universal Power Mixer, UNi-PRO, Kilsyth) before representative sub-sampling into smaller sample bottles. After sub-sampling, pH was measured with a portable pH meter (Model WP 80, TPS, Brendale). Manure samples were also collected via scraping directly off barn or feedpad floors into a central pile, collecting material from an area with an approximate 2.5 m radius. Typically, five manure piles were taken from the floor and combined in a bucket before being thoroughly mixed and a representative composite sub-sample of 0.5 kg collected. The sampling of solid–liquid separation systems broadly followed principles described by Grell et al. (2023) to ensure the inflow and outflow fractions were representatively sampled. After sampling, all sample containers were promptly sealed and placed on ice and transported cold to the laboratory for analysis. At the laboratory, samples were stored at 4 °C for no more than 2 weeks prior the B_0 measurement (Section 2.3), and were also analysed for TS, volatile solids (VS), pH, chemical oxygen demand (COD), and volatile fatty acids (VFA) (Section 2.4).

Table 1
Detailed overview of investigated dairy farms and effluent samples.

Farm	State	Milking herd	Effluent system	Sample location	Feed	Cleaning	Effluent volume ($kL\cdot d^{-1}$)
1	WA	1,400	Uncovered anaerobic lagoon and passive composting	Active Separator Flocculated[1]	PB	Flood	110
2	WA	300	Liquid/Slurry	Passive Separator	PB	Hose	25
3	WA	1,200	Daily spread	Dairy Feedpad	PMR	Hose	70
4	QLD	180	Daily spread	Dairy	PB	Hose	21
5	NSW	400	Daily spread	Dairy	PMR	Hose	11.3
			Solid storage	Feedpad	PMR	Dry Scraped	
6	NSW	250	Uncovered anaerobic lagoon and passive composting	Passive Separator	PB	Hose	15.5
				Dairy	PB	Recycled Flood	12
			Pure manure	Floor	PB		–
7	QLD	450	Uncovered anaerobic lagoon	Feedpad	PMR	Recycled Flood	40
			Pure manure	Floor	PMR		–
8	NSW	550	Uncovered anaerobic lagoon	Dairy	PB	Hose	41
9	WA	350	Uncovered anaerobic lagoon	Dairy	PB	Hose	21.6
10	VIC	440	Uncovered anaerobic lagoon	Dairy	TMR	Recycled Flood	60
			Liquid/Slurry	Barn	TMR	Wet Scraped	19
			Uncovered anaerobic lagoon	Dairy	TMR	Flood	47
11	VIC	675	Slurry storage	Barn	TMR	Wet Scraped	19
			Uncovered anaerobic lagoon	Barn	TMR	Recycled Flood	69
12	VIC	350	Passive composting	Active Separator	TMR	Wet Scraped	–

[1]Chemically enhanced separation with cationic polyacrylamide as a flocculant and hydrated lime as a coagulant-aid (Grell et al., 2023)

2.3. Biochemical methane potential test

Biochemical methane potential tests were carried out in batch using an Automated Methane Potential Test System II (AMPTS II; Bioprocess Control, Lund Sweden) equipped with CO_2 traps holding 3 M sodium hydroxide. Each AMPTS II batch digestion test had a working volume of 400 mL. All the batch tests were conducted at 37 ± 1.0 °C. Test batches were inoculated with fresh inoculum generated in-house in a 30 L lab-scale CSTR operating at 38 °C, a hydraulic retention time of 60 days, and a typical pH in the range 7.6–7.8. This inoculum digester was fed at a low loading rate of 0.25 $kg_{VS}{\cdot}m^{-3}{\cdot}d^{-1}$ with cattle manure and paunch, and sludge from a domestic wastewater treatment plant. Biogas and pH of this inoculum digester was frequently monitored to confirm on-going operational health. As per VDI 4630 (2006), the inoculum was sieved through a 2 mm mesh, resulting in a typical inoculum TS of 3.1–4.2 % and VS of 1.8–2.5 %. To confirm the viability of the inoculum, positive controls using microcrystalline cellulose were operated in parallel, and if >= 80 % of the expected B_0 was reached in these tests, the inoculum was deemed to have been viable and the test successful. As per AMPTSII supplier recommendations, the initial amount of inoculum vs. substrate added to each test batch was fixed at a respective VS ratio of 3:1, to provide an excess of microbial biomass in the tests. The tests were deemed to have been completed when daily CH_4 production was below 1 % of the cumulative CH_4 production across the test (VDI 4630, 2006). A negative control containing only inoculum was also run in parallel to subtract background CH_4 production from each of the treatment batches. The AMPTS normalised the measured CH_4 volume data back to 0 °C, 1 atm, and 0 % humidity, which are the conditions at which gas data are reported below. The number of manure residue types tested required five separate test batches, because of a limited number of AMPTS digestion bottles available. Since inocula can vary somewhat between different test batches (as assessed by the positive controls), rate kinetics data obtained from the batch tests were carefully interpreted accordingly. As per conventional methods, B_0 was normalised to substrate VS added to each treatment batch. In this case, added substrate VS was taken to be equal to measured VS in the substrate added plus measured VFAs in the substrate added (Section 2.4). This accounted for the expected VFA loss, which can be substantial during the standard oven drying step of VS determination (Section 2.4).

2.4. Analytical methods

Total solids (TS) and VS were analysed using Standard Method 2540G (APHA, 1995). Chemical oxygen demand and VFA were determined using Merck Spectroquant test kits (catalogue numbers: 1.14555.0001; 1.91797.0001, and 1.01809.0001) with a Spectroquant Pharo 100 spectrophotometer (Merck, Germany). For COD, the samples were shredded and quantitatively diluted in a kitchen blender before analyses, and VFAs were analysed from the supernatant following centrifugation (Sigma 2-16P) at 5,000 rpm (2,665 × g) for 10 min.

2.5. Data analysis and statistical methods

Each analyte was measured in triplicate, reporting mean values with standard deviations corresponding to variability in the analytical replicates. The validity of the sample collection was assessed by estimating expected VS in manure residue collected/captured at each farm. This was done by multiplying measured VS concentrations in the samples by daily volumes of manure residues/effluent produced by each farm determined from farm operational data (e.g., pump times, or changes in liquid hold-up volumes of flood wash water tanks), and then comparing this result to a theoretical VS production estimate by the approach in the Australian National Greenhouse Accounts (Commonwealth of Australia, 2018). Assumptions for the theoretical estimate included an average milk yield of 16.5 kg per cow per day, a daily liveweight gain of 0.016 kg for milking cows, an average weight of 550 kg for milking cows, and other default factors (Commonwealth of Australia, 2018). Based on these parameters, the theoretical total daily manure output was estimated to be 4.5 kg VS per cow. It is noted that the milk yield as well as the animal weight can vary across different production systems. However, for the level of validation sought in the current study, the average assumptions were deemed to be appropriate.

All the statistical evaluation was carried out in R statistical software (version 4.2). For the statistical analysis, farms were categorised based on production system type (i.e., PB, PMR, TMR) and cleaning type (i.e., Flood, Hose, Scraped) to test for effects on measured B_0 and TS. Assumptions of normality and homogeneity of variances were tested for the B_0 and TS data. The impact of production systems (PB vs. PMR vs. TMR) on B_0 was assessed using a 1-way ANOVA with Type III sums of squares to account for differences in sample size (i.e., farms) between factors. Only the B_0 data for raw dairy manure residues (prior to any separation) were considered in this analysis, and any data from systems utilising recycled water for cleaning were also excluded for the reasons discussed below. Cleaning was included as a random factor to account for background variance caused to B_0 due the different cleaning methods. For the TS data, it was observed that normality conditions were not satisfied, accordingly a log+1 transformation was applied, and the resulting transformed values found to be normally distributed and used instead in the subsequent analyses. Production type was included as a random factor to account for background variance caused to TS due the different production types. Post-hoc pairwise comparisons determined which treatments were statistically different, using the difflsmeans function from the lmerTest package in R and applying the Kenward-Roger approximation for degrees of freedom. Solid-liquid separation systems were investigated by using a two-tailed student *t*-test ($\alpha = 0.05$) to identify significant differences between the inflow and separated fractions. This was done individually for separation systems on particular farms and, where data availability permitted, also for clustered data for particular separation types as stated below. To investigate a potential correlation between VS/TS ratio and B_0, a Pearson correlation analysis was performed. The correlation coefficient was calculated using the cor.test ($\alpha = 0.05$) function in R, quantifying any linear association between the variables across all samples.

3. Results and discussion

3.1. Impact of production system type

The type of production system (PB vs. PMR vs. TMR) was expected to have a bearing on manure capture extent, manure residue composition, and potentially B_0 via effects of dietary differences. This is important for quantifying emissions potential and biogas energy potential. Measured characteristics (TS, VS, VFA, COD and B_0) were observed to vary between the different farms (Table 2). To contextualise the current results, the measured TS concentrations of samples (prior to any separation) varied widely from 0.6 % to 28.9 %, depending on whether the manure was collected as an effluent, a slurry, or a scrape. Measured TS concentration for the effluent samples ranged from 0.57 % to 3.07 %, which was comparable to values reported by Longhurst et al. (2000) (0.5 %-1.4 %) and Page et al. (2014) (2.8 %), as well as the mean value of 1.7 % from 19 other studies (Kupper et al., 2020). This also aligns with studies that simulated dairy effluent by diluting pure manure, ranging from 0.4 % to 3.2 % (Garcia et al., 2009; Pandey et al., 2019). Measured VS concentrations of the dairy effluent samples varied between 0.4 % and 2.3 %. This was somewhat consistent with the mean value of 7 studies reported by Kupper et al. (2020) at 0.4 %, and the VS range of a simulated effluent of 0.3 % to 2.8 % (Garcia et al., 2009; Pandey et al., 2019). Measured COD concentration of the dairy effluent samples ranged from 6,748 to 40,827 $mg{\cdot}kg^{-1}$ (on a wet basis), somewhat higher than reported elsewhere (438–23,650 $mg{\cdot}kg^{-1}$) (Birchall et al., 2008; Fyfe et al., 2016; Wang et al., 2020) and compared to COD values of the simulated effluent study of Garcia et al. (2009) (3,100–29,200 $mg{\cdot}kg^{-1}$).

Table 2
Characteristics of dairy waste relevant to manure methane and anaerobic digestion. Values given are calculated means in replicates (±standard deviation).

Farm	Sample	TS (% wet)	VS (% wet)	VFA ($mg \cdot kg^{-1}$)	COD ($g \cdot kg^{-1}$)	COD/ VS	B_0 ($L_{CH4} \cdot kg_{VS}^{-1}$)
1a	Effluent	0.56(±0.02)	0.41(±0.02)	589 (±29)	6.8 (±0.5)	1.66	165 (±3)
1a	Liquids	0.45(±0)	0.29(±0)	434 (±38)	5.0 (±0.3)	1.72	154 (±3)
1a	Solids	21.27(±0.15)	18.2(±0.21)	–	309.5 (±40.8)	1.70	187 (±2)
1b	Effluent	0.57(±0.02)	0.39(±0.02)	539 (±61)	5.6 (±0.4)	1.46	139 (±5)
1b*	Liquids[%]	0.19(±0)	0.07(±0)	267 (±18)	1.1 (±0.1)	1.48	171 (±9)
1b	Solids	19.46(±0.47)	13.22(±0.39)	–	186.8 (±15.8)	1.41	141 (±5)
2	Effluent	1.84(±0.07)	1.28(±0.04)	1,484 (±60)	20.3 (±2.5)	1.58	141 (±13)
2	Liquids	0.64(±0.01)	0.39(±0)	972 (±27)	6.5 (±0.2)	1.67	91 (±3)
3	Effluent	2.59(±0.1)	2.01(±0.07)	2,723 (±73)	29.9 (±1.4)	1.49	225 (±4)
3	Feedpad effluent	3.54(±0.02)	2.66(±0.02)	3,586 (±129)	46.1 (±1.3)	1.73	185 (±1)
4	Effluent	3.14(±0.06)	1.57(±0.04)	1,094 (±31)	31.4 (±3.4)	2.00	196 (±4)
5	Effluent	2.82(±0.13)	2.23(±0.11)	1,629 (±33)	40.8 (±3.9)	1.83	148 (±15)
5	Feedpad solids	28.85(±0.47)	23.05(±0.51)	5,143 (±158)	28.1 (±23.6)	1.22	119 (±18)
6	Effluent	1.1(±0.04)	0.73(±0.03)	967 (±56)	16.7 (±1.7)	2.30	133 (±11)
6	Liquids	0.69(±0)	0.35(±0.01)	786 (±23)	5.7 (±0.1)	1.63	102 (±2)
6	Recycled Effluent	1.05(±0.03)	0.62(±0.01)	689 (±9)	15.7 (±1.4)	2.54	98 (±16)
6	Manure	12.26(±0.05)	9.5(±0.03)	6,106 (±38)	152.1 (±9.8)	1.60	138 (±14)
6	Calf Manure	17.51(±0.06)	15.76(±0.06)	12,178 (±67)	26.7 (±15.3)	1.70	279 (±17)
7	Feedpad effluent	0.94(±0.07)	0.69(±0.07)	892 (±1)	9.8 (±0.5)	1.42	101 (±28)
7	Manure	15.22(±0.08)	11.97(±0.07)	–	–	–	155 (±1)
8	Effluent	1.39(±0.01)	0.95(±0)	1,538 (±85)	15.8 (±0.1)	1.67	197 (±3)
9	Effluent	2.11(±0.03)	1.61(±0.03)	1,455 (±58)	31.0 (±3.8)	1.92	161 (±11)
10	Recycled Effluent	1.22(±0.03)	0.74(±0.02)	892 (±51)	16.2 (±0.7)	2.21	113 (±61)
10	Barn slurry	10.89(±0.78)	8.34(±0.72)	6,559 (±177)	131.7 (±10.3)	1.58	215 (±14)
11	Barn effluent	2.82(±0.1)	1.95(±0.09)	1,795 (±111)	34.9 (±2.8)	1.79	198 (±28)
11	Calf effluent	0.46(±0.01)	0.36(±0)	330 (±8)	1.0 (±0.1)	0.28	201 (±15)
11	Barn slurry	12.76(±0.64)	9.7(±0.65)	2,591 (±34)	121.9 (±15.6)	1.26	192 (±18)
12	Barn effluent recycled	3.07(±0.05)	2.29(±0.05)	1,694 (±36)	39.6 (±5.3)	1.04	158 (±70)
12	Solids	8.78(±0.33)	7.98(±0.32)	–	109.5 (±36.5)	1.29	206 (±27)

*Sample 1b had flocculant and lime used in accordance with the conditions described by Grell et al. (2023); [%]"Liquids" refers to the liquid fraction from separation.

Measured VFAs ranged from 589 to 2,723 $mg \cdot kg^{-1}$ (on a wet basis), aligning with barn effluent (1,278–2,648 $mg \cdot kg^{-1}$) (Page et al., 2014) and the simulated effluent of Garcia et al. (2009) (i.e., 1,130 $mg \cdot kg^{-1}$). Average pH was 7.2, with a range of 6.1–7.9, comparable to values reported in the literature (7.1–8.22) (Birchall et al., 2008).

The theoretical analysis of manure capture (Section 2.5) showed that PB farms in the current study captured an average 15 ± 6 % of the daily manure VS output. This equates to a 3.5 h average time for cows spent on surfaces where manure is collected. This is generally consistent with a typical twice-daily milking with groups of cows held on concrete holding yards and returned to grazed paddocks directly after milking (Birchall et al., 2008). The results further suggested that the PMR farms captured 56 ± 6 % of daily excreted manure, aligning with cattle spending approximately the same time on pastures as on feedpads (Arnott et al., 2017). Moreover, the TMR farms captured an estimated 94 ± 5 % of the excreted manure. These results aligned with expectation, also indicating that the sampling was reasonable, and demonstrating that PMR and TMR systems enable greater manure capture than PB.

Biochemical methane potential was measured to assess CH_4 emissions potential and biogas energy potential (Section 1). The B_0 of samples across the study ranged from 91.4 to 278.5 $L_{CH4} \cdot kg_{VS}^{-1}$ ($n = 87$) with an average of 161.1(±43.6) $L_{CH4} \cdot kg_{VS}^{-1}$. Mean B_0 values were 161 (±26.9) $L_{CH4} \cdot kg_{VS}^{-1}$ for PB ($n = 7$), 166.0(±40.4) $L_{CH4} \cdot kg_{VS}^{-1}$ for PMR ($n = 5$) and 202.0(±12.3) $L_{CH4} \cdot kg_{VS}^{-1}$ for TMR ($n = 3$) (Fig. 1A). However, due to the somewhat expected variability within samples collected from commercial facilities, the effect of production type was found to be not statistically significant ($p = 0.056$). As noted in Section 2.5, the effect of cleaning type was indirectly considered as a random factor in this analysis. When a repeat analysis was conducted excluding this random factor for comparison, the results showed that some variance in the data set was visibly accounted for by the random factor, but the overall outcomes of the analysis were unchanged (See Supplementary Materials). A pairwise comparison showed that B_0 for TMR was not significantly different to that for PB ($p = 0.08$), and that the difference between PB and PMR was also not statistically significant ($p = 0.67$). This could align with the expectation that cattle spend approximately the same time on pastures as on feedpads (Arnott et al., 2017), still acquiring a significant proportion of their daily feed from pastures. However, B_0 for TMR did appear to trend towards a higher value (albeit not significantly higher), which could be worthy of further exploration in future studies to assess diet impacts. Specifically, manure from PB systems could contain more recalcitrant and poorly biodegradable lignocellulosic materials resulting in a lower methane yield. The degree of recalcitrance can affect the surface area of the cellulose that is accessible to hydrolytic bacteria (Karimi & Taherzadeh, 2016; Surendra et al., 2018). These microbes secrete extracellular enzymes (e.g. cellulases) that convert lignocellulose to monosaccharides, therefore controlling the amount of fermentable sugars available for the subsequent AD degradation pathways and ultimately methane production (Kratky & Jirout, 2011). Conversely, manure from intensive feeding systems could reflect the feed ration with more readily biodegradable carbohydrates and proteins from grain and forage, resulting in a higher methane yield (Labatut et al., 2011). For individual farms, this appeared to align with the B_0 of scraped manure for PB (Table 2, 146.6(±12.3) $L_{CH4} \cdot kg_{VS}^{-1}$) being lower than the B_0 for freshly collected barn slurry from TMR (Table 2, average across Farms 10 and 11 of 204(±14.5) $L_{CH4} \cdot kg_{VS}^{-1}$).

To contextualise these results with the relevant literature, measured B_0 values in the current work fell within the range of the *meta*-analysis results of Miranda et al. (2016). However, the reported average B_0 of Miranda et al. (2016) for the Asia/Middle East and India region (220 $L_{CH4} \cdot kg_{VS}^{-1}$) was higher than the current results, which could be partly due to differences in production across this region (Section 1). The B_0 values measured in the current study were lower than the default value in IPCC (2006) of 240 $L_{CH4} \cdot kg_{VS}^{-1}$. This is important for sector emissions estimates and biogas energy assessments, as further discussed below (Section 3.4).

3.2. *Impact of dairy cleaning strategy*

Cleaning strategy influenced effluent characteristics and particularly TS, which is important for AD technology selection as well as for the efficiency of solid–liquid separation to divert VS away from effluent

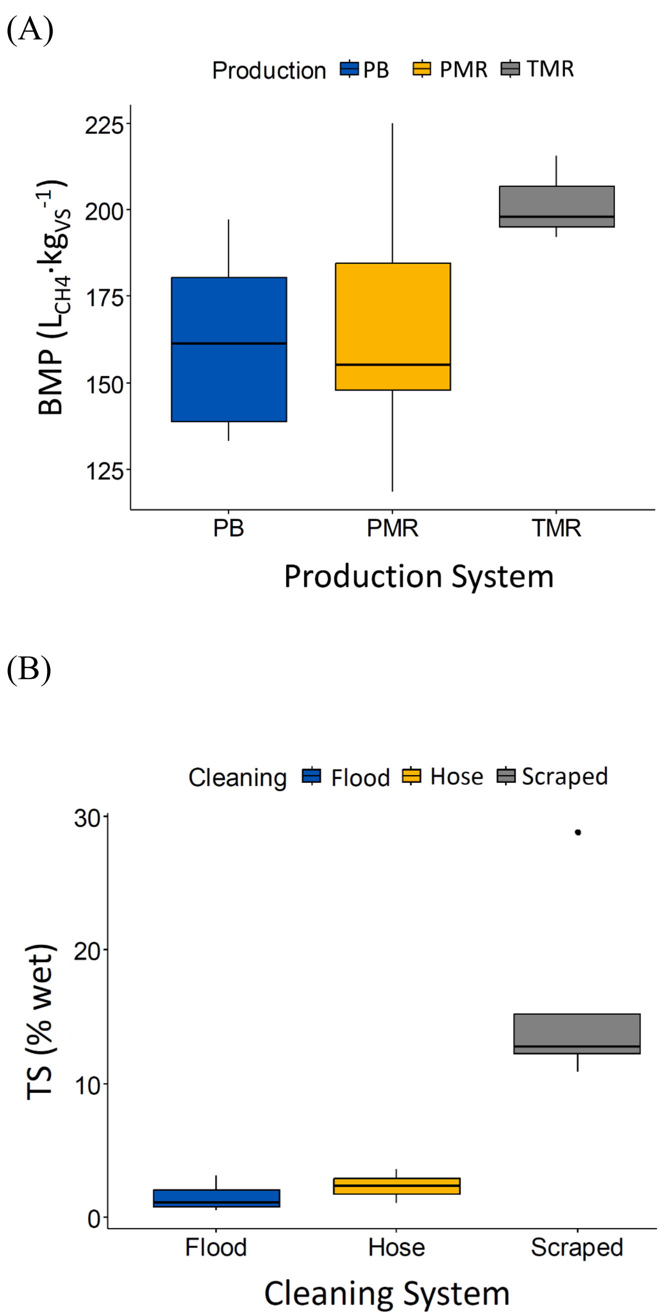

Fig. 1. Summary statistics as box-and-whisker plots, showing (A) TS in manure/effluent as affected by cleaning method, and (B) B_0 of dairy effluent as affected by production type. Outliers are also shown as single data points in the case of TS, which were excluded from the statistical analysis.

ponds. Mean TS in dairy effluent was observed to be 1.46(±1.04)% for Flood (n = 7), 2.32±(0.86)% for Hose (n = 8), and 16.0±(7.35)% for Scraped (n = 5) (Fig. 1). Yard scraping can be performed daily, collecting fresh manure, or less frequently (e.g., weekly), collecting semi-dried and partially biodegraded manure, and may partly explain the observed relatively larger standard deviation. This was also the reason why dry scraped manure from Farm 5 had to be excluded from the statistical analysis as an outlier, being abnormally dry (TS = 28.8 %), and likely unattractive for AD. The Shapiro-Wilk test indicated TS data did not follow a normal distribution (p = 0.000022), probably due to skewness in the data with substantially higher TS in Scraped than in Flood or Hose. This was resolved by a log+1 transformation of the TS data (normality assumption, p = 0.019) (See Supplementary Materials).

The Type III ANOVA showed significant effects of cleaning type on TS ($p < 0.05$). As expected, there were significant differences in TS between Hose and Scraped ($p < 0.05$), and between Flood and Scraped ($p < 0.05$), with Scraped using minimal water. However, a post-hoc pairwise comparison also revealed a significant difference (p = 0.0432) between Hose and Flood, indicating a possible influence of water use efficiency; albeit that mean TS of Hose and Flood was similar, indicating that some minimum amount of water is required for effective liquid cleaning of a dairy. Due to the restricted number of replicates, the analysis could not assess the separate effects of production type and cleaning type, nor any interactive effects. However, production type was indirectly considered as a random factor (Section 2.5). When a repeat analysis was conducted excluding this random factor effect, the results showed the overall effect of cleaning type was still significant, but the pairwise effects between Flood and Hose were no longer significant (See Supplementary Materials). This indicated an important background effect of production type, possibly due to differences in manure capture (Section 3.1). Implications for manure management and AD options are further discussed in Section 3.4.

The use of recycled effluent for flood wash cleaning can save considerably on freshwater use at dairies but may recycle aged manure. This would be important for emissions or biogas energy potential. To clarify this, the ratio of VS/TS was used as a typical sensitive indicator of ageing effects (Gopalan et al., 2013). A statistically significant positive correlation was found between VS/TS ratio of all samples and B_0 (Pearson coefficient r = 0.451, p = 0.014) (Fig. 2). Moreover, for specific sites, B_0 for calf manure from Farm 6 with a high VS/TS ratio of 0.90 was high at 278.5 $L_{CH4}·kg_{VS}^{-1}$. This indicates a higher proportion of biodegradable VS in samples with a higher VS/TS ratio, and that minimal ageing and CH_4 yield losses had occurred. Further, when recycled effluent was used at Farm 6 (PB), the effluent had a VS/TS ratio 0.58 and a low B_0 of 98.8 $L_{CH4}·kg_{VS}^{-1}$, whereas when fresh water was used instead for cleaning, a higher VS/TS ratio of 0.66 and a higher B_0 of 132 $L_{CH4}·kg_{VS}^{-1}$ were observed. Similar observations were noted for Farm 7 (PMR) and Farm 10 (TMR), likely due to manure ageing/extended storage in liquid effluent systems. Implications for emissions and biogas energy potential are discussed in Section 3.4.

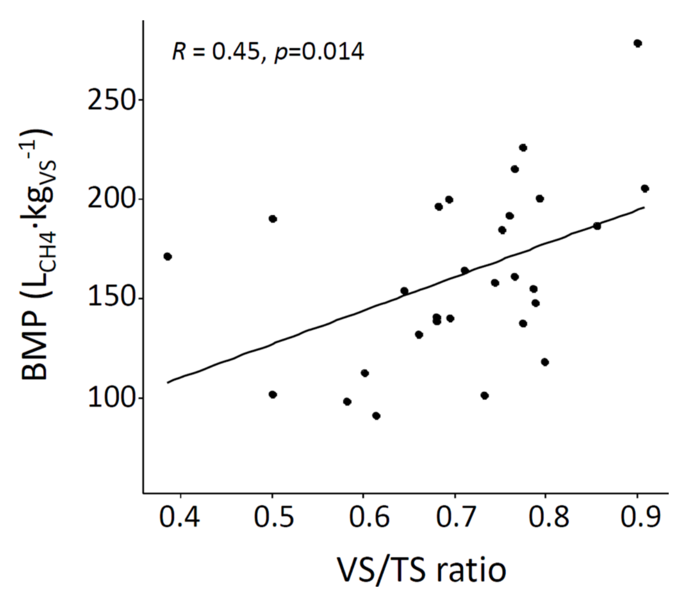

Fig. 2. Correlation plot between B_0 and VS/TS ratio (for all samples) tested in the current work. Note that measured VS was used in this case, without adding measured VFAs.

3.3. Influence of solid–liquid separation

Solid-liquid separation at all PB farms showed significant removal of TS, VS, and COD from the effluent ($p < 0.05$). However, on the day of sampling, the solids traps with weeping walls were visibly filled with accumulated solids, indicating infrequent clean-outs as confirmed by the dairy farmers. This would promote anaerobic biodegradation in these solids traps and exacerbate volatilisation losses/fugitive CH_4 emissions (Hull-Cantillo et al., 2023). For example, the B_0 values of the inflow effluent and freshly scraped manure at Farm 6 were comparable (Table 2), but B_0 of the liquid fraction from passive separation was much lower (Table 2). Observations for Farm 2 with passive separation were the same. In contrast, for the PB Farm 1 with mechanical separation, B_0 of the liquid fraction was similar to that of the effluent inflow prior to separation, indicating a preservation of specific CH_4 yield and minimal volatilisation losses. When flocculant was used at this same farm to facilitate separation (Sample 1b, Table 2), B_0 of the separated liquid fraction (171 $L_{CH4}{\cdot}kg_{VS}^{-1}$) was notably higher than that of the effluent inflow prior to separation (139 $L_{CH4}{\cdot}kg_{VS}^{-1}$) (See Supplementary Material). Moreover, the solids fraction from mechanical separation at Farm 12 had a notably higher B_0 than that of Farm 1 (Table 2), aligning with a comparatively higher B_0 of the effluent prior to separation at Farm 12. This could reflect compositional differences in organic matter between effluents from different production types (Section 3.1) but also compositional differences between separated fractions. Prior investigations have typically reported higher B_0 values for separated liquid fractions as compared to solids fractions (Rico et al., 2012; Rico et al., 2007). However, in the current work, VFAs were included in the VS amount against which B_0 values were normalised (Section 2.3), expected to be important for liquid fractions with high VFA as compared to measured VS. When this is done, B_0 values for liquid fractions were similar to that of the solids fractions, as expected from a predominantly lignocellulosic and carbohydrate-based substrate.

The amount of time required for completion of the biochemical methane potential tests varied between treatments (Table 3), which could be reflective of the separation of different organic matter components into different streams. Time to completion was similar for the effluent inflow and the separated liquid fraction. However, time to completion was notably shorter for the liquid fraction than for the solids fraction (Table 3, e.g., compare solid and liquid fractions at Farm 1). Similar observations were made when flocculant was used for separation, specifically at Farm 1 (Sample 1b), and likely reflects differences in particulate and organic matter compositions. The comparatively shorter time to completion for filtrate samples may be attributed to more rapidly biodegradable particulate matter, either being smaller with a greater accessible surface area for hydrolysis or being of a more readily biodegradable make-up. It is noteworthy that three of the farms from which only raw effluent or slurry was sampled, solid–liquid separation equipment was present but had fallen into disrepair, or were not functioning properly, and hence were not sampled for this study. This was reflective of the typical high maintenance and management efforts required by such systems but may also indicate a sub-optimal selection of separation technologies to match effluent TS at the sampled sites (Section 3.1). This generally discourages farmers from operating separation systems proactively. However, with anticipated biogas energy benefits from AD, the correct selection, adoption and proactive management of solid–liquid separation systems on-farm may become incentivised and reinvigorated.

Table 3
Times taken for completion of the biochemical methane potential tests to attain B_0 (Time to completion).

Farm	Sample	Batch	Test completion time (days)
1a	Effluent	1	8
1a	Liquids		8
1a	Solids		23
1b	Effluent		10
1b	Liquids%		6
1b	Solids		18
2	Effluent	2	14
2	Liquids		11
3	Effluent		16
3	Feedpad manure		16
4	Effluent		15
5	Effluent	3	15
5	Feedpad manure		18
6	Effluent		13
6	Liquids		8
6	Recycled Effluent		23
6	Manure		17
6	Calf Manure		18
7	Effluent		8
7	Manure	1	17
8	Effluent	4	8
9	Effluent	5	13
10	Barn effluent		11
10	Recycled Effluent		18
11	Barn slurry		11
11	Barn effluent		17
11	Calf effluent		17
12	Barn slurry		16
12	Barn effluent recycled		21

3.4. Implications for emissions abatement and biogas energy recovery

An important interplay was expected between manure capture extents in PB vs. PMR vs. TMR (Section 3.1), manure-management emissions potential, and biogas energy potential. An increased proportion of manure was captured by PMR and TMR as compared to PB (Section 3.1). This is important because a greater manure capture can increase CH_4 losses and fugitive emissions from effluent storage/ageing for extended periods (Section 3.3). For example, uncovered effluent ponds (wherein effluent is typically stored) have a methane conversion factor (MCF) of 0.7–0.8, whereas the MCF is comparatively much lower for manure deposited onto pastures (MCF = 0.01–0.02) (Commonwealth of Australia, 2018; IPCC, 2006) or for aerobic post-processing and storage of the separated solids fraction (MCF = 0.02) (Commonwealth of Australia, 2018). This means that if manure VS is diverted (e.g., via solid-liquid separation) away from extended effluent storage and then land applied with/without prior aerobic processing, a theoretical emissions saving can be achieved for the diverted VS, proportional to the difference in the MCF factors above. A greater manure capture also increases the opportunity for biogas recovery via AD. To demonstrate, a daily VS excreted of 4.5 kg_{VS} per head (Section 2.5) can be multiplied by 1.44 million cows in Australia (Section 1), then multiplied by the manure proportion not voided on pastures (0.2) (Christie et al., 2018), and then multiplied by an average methane yield of 161 $L_{CH4}{\cdot}kg_{VS}^{-1}$ for grazing dairy effluent. This amounts to an estimated 76.2 million m_N^3 methane per annum with a total energy potential of 2.82 PJ·annum^{-1}. If the proportion of manure capture was to increase to 50 % (a potential future scenario of mixed PB and intensive dairies), this total energy potential could increase to 7.04 PJ·annum^{-1}, suggesting the potential influence of intensification on biogas energy potential.

For the relatively low TS from Flood (Section 3.2), or with cases where water use efficiency cannot be further improved to increase TS, covered anaerobic pond technology may be most cost-effective for AD (Section 1) despite a typical large size and spatial footprint. In contrast, a higher TS in scraped manure residues (Section 3.2) or the solids fraction from solid–liquid separation (Section 3.3), may provide an appropriate TS to address hydraulic limitations of CSTR digestion technology with better control of biogas production via heating and mixing (Section 1). Above-ground CSTRs are the dominant AD technology in Germany (Weiland, 2010). Separation into a solid fraction has the added advantage of condensing manure VS into a much smaller mass/volume, resulting in more practical and cost-effective transporting, such as for further processing via centralised AD. For example, a mass balance for the separation at Farm 1 demonstrated a mass ratio of filtrate to solids of approximately 0.6:99.4 without flocculant and lime (Sample 1a) and approximately 2.4:97.6 with flocculant and lime (Sample 1b). The use of lime and flocculant may be important to shift methane yield more towards the solids fraction. Conversely, the separated liquid fraction may instead be considered for AD in a covered effluent pond, or even sludge blanket reactors or anaerobic filters. These latter technologies are suited to feedstocks with lower TS (Batstone & Jensen, 2011). A smaller covered pond could then be suitable, because of a relatively faster degradation rate of the liquid fraction (Section 3.3), and because of a reduced organic and solids loading resulting from solids removal by the separation step. However, the current results indicated that mechanical separation would likely be preferred over passive separation with infrequent clean-outs (Section 3.3), because the former preserves B_0, whereas the latter decreases B_0 to likely result in fugitive methane emissions. For example, the mechanical separation at Farm 1 without flocculant (Sample 1a) achieved a VS removal efficiency of 29 %. Based on measured VS and B_0, the separated liquid fraction contained 67 % of the total methane yield in the effluent inflow prior to separation, and the separated solids fraction contained about 28 %. When lime and flocculant were used at this farm (Sample 1b), VS removal increased to 81 %, so that now only 27 % of the total CH_4 yield in the inflow remained in the filtrate, and 71 % reported to the solids fraction. This indicates the potential to abate manure management emissions or to make manure organic matter available for biogas energy recovery.

Future research is recommended using B_0 data from the current study to update dairy sector emissions estimates, using detailed life-cycle assessments that consider all value-chain emission sources, as well as up to date statistics on the proportions of PB, PMR vs. TMR. This would also be important to understand carbon abatement potential, and biogas energy potential.

4. Conclusion

This study measured biochemical methane potential (B_0) for dairy manure residues and solid–liquid separation fractions, important for emissions and biogas estimates. A first B_0 is reported for grazing dairy effluent ($161 L_{CH4} \cdot kg_{VS}^{-1}$), found to be not significantly different from B_0 for intensive dairies ($166–202.0 L_{CH4} \cdot kg_{VS}^{-1}$). Intensive dairies capture more excreted manure, increasing potential emissions but also biogas energy, specifically estimated for Australia for an all-grazing scenario (current) at $2.82 PJ \cdot annum^{-1}$ or a mixed-grazing-intensive-dairy scenario at $7.04 PJ \cdot annum^{-1}$. Mechanical separation preserved B_0 and could abate fugitive manure management methane. B_0 values in this study are recommended for potential updates to Australia's country-specific values.

CRediT authorship contribution statement

Torben Grell: Conceptualization, Methodology, Investigation, Formal analysis, Visualization, Writing – original draft. **Peter W. Harris:** Methodology, Investigation, Formal analysis, Writing – review & editing. **Serhiy Marchuk:** Methodology, Investigation, Formal analysis, Writing – review & editing, Supervision. **Sasha Jenkins:** Funding acquisition, Resources, Investigation, Writing – review & editing, Supervision. **Bernadette K. McCabe:** Conceptualization, Methodology, Funding acquisition, Resources, Writing – review & editing, Supervision. **Stephan Tait:** Conceptualization, Methodology, Funding acquisition, Resources, Investigation, Formal analysis, Writing – review & editing, Supervision.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.biortech.2023.129903.

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