CARBON REDUCTION AND RESOURCE RECOVERY FROM ON-FARM DAIRY WASTE IN AUSTRALIA

A Thesis submitted by

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ABSTRACT

The aim of this thesis is to develop circular manure management strategies to reduce environmental risks and improve the sustainability of the Australian dairy farming sector. Circular technologies target the beneficial recycling of resources contained in dairy manure residues, such as nutrients and organic matter, for bioenergy production. Measurements are performed on liquid manure residues (effluent) from various dairy farms across Australia and reveal material amounts of nutrients, but there are no significant differences in nutrient concentrations amongst different dairy production system types (i.e. grazing vs. intensive dairies). However, there are notable differences in total nutrient capture rates (recovery potential) between different systems. Unfortunately, measurements confirmed dairy effluent is usually heavily diluted, making its transport, further processing, and beneficial use less viable. Cleaning strategies also affected dilution, requiring recovery methods such as solid-liquid separation. A modular solid-liquid separation technology is applied at full-scale at a commercial dairy. Without chemicals (lime and flocculant), only 25.9% particulate matter and 33.4% organic particle matter are recovered into the solid fraction, but the filtrate is more usable for irrigation. The solid fraction is also stackable and easily transportable for further processing and reuse. Lime and polymer flocculant enabled nitrogen and phosphorus recovery into the solid fraction, at ~54% and up to 91%, respectively. This provides circular options for farmers. A first biochemical methane potential of grazing dairy effluent is reported, specifically 161 $L_{CH}{}^{4}\cdot kg^{-1}$ volatile solids. Important effects of on-farm manure separation were also evaluated. This is important to evaluate renewable biogas recovery potential and is essential for sector emissions accounting. Overall, the data and findings of the thesis were invaluable to understand closed-loop system options, estimating recovery potential, and evaluating approaches to reduce manure-management greenhouse gas emissions across Australian dairy farms.

CERTIFICATION OF THESIS

I, Torben Grell, declare that the PhD thesis entitled *Carbon reduction and resource recycling from on-farm dairy waste in Australia* is not more than 100,000 words in length, including quotes, tables, figures, appendices, bibliographies, references, and footnotes.

This thesis is the work of Torben Grell, except where otherwise acknowledged, with the majority of the contribution to the papers presented as a thesis by publication undertaken by the student. The work is original and has not previously been submitted for any other award, except where acknowledged.

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STATEMENT OF CONTRIBUTION

Publication 1:

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Torben Grell contributed 85% to this paper. Collectively, Serhiy Marchuk, Sasha Jenkins, Bernadette K. McCabe, and Stephan Tait contributed the remainder.

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DEDICATION

Overall, I would like to dedicate this thesis to our planet in hope for a long-lasting, sustainable future. This thesis is also for the loved ones who have left the planet recently, namely my good friend Ian Williams and my Oma Ri. I miss you; your planted seeds are germinating, hopefully in cow poo. The memories of our joint past are unforgettable, and I appreciate every minute of having you around me. Ultimately, we look into the future, and this piece of hard work is also for my little rowdies, Lia and Ellie.

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ABBREVIATIONS

ACCU	Australian Carbon Credit Units
AD	Anaerobic digestion
AMPTS	Automated methane potential test set
AUD	Australian Dollar
BMP	Biochemical methane potential
C	Carbon
Ca	Calcium
CEP	Covered effluent pond
CH_4	Methane
CO_2	Carbon dioxide
COD	Chemical oxygen demand
CSTR	Continuous Stirred Tank Reactor
DDOR	Dairy Declaration Organization
DM	Dry matter
e.g.	For example (lat.: exempli gartia)
EC	Electrical conductivity
ERF	Emission Reduction Fund
et al	and others (lat.: et alia)
FAO	Food and Agriculture Organization
GC	Gas chromatograph
GHG	Greenhouse gas
H_2	Hydrogen
ha	Hectares
HRT	Hydraulic retention time
IPCC	Intergovernmental Panel for Climate Change
K	Potassium
kg	Kilogram
kL	Kilo litre
m^3	Cubic meter
MCF	Methane conversion factor
Mg	Magnesium
mL	Millilitres
mm	Millimetre
N	Nitrogen
N_2O	Nitrous oxide
NGER	National Energy and Greenhouse Reporting
NH_3	Nitrate
NSW	New South Wales
OM	Organic matter
P	Phosphorus

PB	Pasture-based
PF	Plug-flow
PMR	Partial mixed ration
PRP	Pasture/Range/Paddock
QLD	Queensland
TKN	Total Kjeldahl Nitrogen
TMR	Total mixed ration
TN	Total nitrogen
TOC	Total organic carbon
TP	Total phosphorus
TS	Total solids
UASB	Upflow Anaerobic Sludge Blanket
USQ	University of Southern Queensland
UWA	University of Western Australia
VDI	Verband Deutscher Ingenieure
VFA	Volatile fatty acid
VIC	Victoria
VS	Volatile solids
vs.	against, opposed to (lat.: versus)
WA	Western Australia
WFPS	Water filled pore space
WHC	Water holding capacity
WHO	World Health Organization
μm	Micrometre

CHAPTER 1: INTRODUCTION

This chapter introduces the motivation and background for the thesis investigations to provide an appropriate level of introductory understanding and context. It presents a summary of the significance of the global and national dairy industry, including important operational, management, and environmental challenges relevant to the thesis investigations. Deductively, it considers opportunities for technical solutions to recover and recycle resources from dairy manure residues, reduce environmental risks, and facilitate sustainable, practical, and cost-effective closed-loop dairy farming approaches.

1.1. The significance of the global and Australian dairy industry

The dairy industry is an integral part of the global agricultural economy because it creates food for the increasing world population and generates substantial economic value. For example, in 2020, the global dairy market was valued at 827.4 billion U.S. dollars (USD) and is expected to grow to approximately 1,128 billion USD by 2026 (Statistica, 2022). The global dairy cattle population in 2021 was estimated at approximately 1.5 billion, producing around 746 million kilolitres (kL) of raw milk (FAO, 2023). Worldwide, the dairy sector contributes 27% of the total global value-added from livestock and 10% from agriculture (DDOR, 2019).

The dairy industry is also a key agricultural sector in the Australian economy. In Australia, 4,618 dairy farms with an average herd size of 300 cows produce about 8,858 million litres of milk per year and generate 4.7 billion Australian dollars (AUD) in farmgate value (Dairy Australia, 2021a). Despite its modest contribution of less than 2% to global milk production, Australia holds the fourth position in global dairy trade, with 36% of its yield exported into international markets (Dairy Australia, 2021a). This importantly positions the dairy industry as Australia's third-most significant rural industry (Dairy Australia, 2021a).

Most Australian dairy farms are located in Victoria (about 67%), followed by New South Wales (NSW) (11%), followed by the other states and territories jointly making up the remainder (22%) (Dairy Australia, 2021a). The on-farm Australian dairy industry is predominantly pasture-based (PB), meaning the milking herd mostly grazes pasture paddocks for their daily diet (Aarons et al., 2020; Bargo et al., 2003). However, with the concentration of the dairy industry in Victoria and southern NSW and associated and emerging climate, environmental, and commercial challenges (e.g., bushfire risk, persistent wet weather, licensed water costs), there has been a progressive shift towards intensive production systems, including in roofed housing (e.g. freestalls or barns) and feedpads (Dairy Australia and Agriculture Victoria, 2023). With intensive systems, a majority part or all of the daily feed of the milking herd is supplied as a formulated ration (Hofstetter et al., 2014). These are respectively termed partial mixed ration (PMR) and total mixed ration (TMR) systems. These progressive changes in dairy production have important implications for livestock manure management, as outlined in the section that follows.

1.2. On-farm dairy manure residues: Environmental risks and recovery opportunities

Dairy farms in Australia (and elsewhere in the world) commonly produce effluent comprised of manure, urine, wash water, and any cleaning chemicals. Dairy effluent contains valuable nutrients like nitrogen (N), phosphorus (P), and potassium (K), as well as organic matter (i.e. carbon) that can be used for renewable biogas energy or as a soil amendment (Abbott et al., 2018). If not carefully managed, these nutrients and organic matter can cause ground and surface water pollution, eutrophication, and the loss of aquatic biodiversity in waterways (FAO & WHO, 2023). For instance, a water quality improvement plan from the Department of Water and Environmental Regulation in the Australian state where the research in Chapter 4 was conducted, identified the dairy sector as one of the biggest nutrient sources in local waterways (White, 2012). In addition, improper dairy effluent disposal and storage contributes to the release of fugitive greenhouse gases, such as methane and nitrous oxide (N_2O) (Laubach et al., 2015), which exacerbate climate change (IPCC, 2006).

Livestock production contributes to Australia's emissions, predominantly with methane (CH_4) and N_2O, accounting for 56% and 73%, respectively (Sudmeyer, 2021). Globally, these emissions (based on the CO_2 equivalents) originate 39% from enteric fermentation, 10% from manure storage (emphasis of the current work), 6% from processing, and 45% from feed production, which includes agricultural operations, manure, fertilizer, and chemical application to soil (Grossi et al., 2019).

Currently, the dominant manure management systems in the Australian dairy industry are simple, low-cost holding ponds (79%), followed by sump and dispersal systems (14%), with 5% of dairy farms draining or irrigating their effluent directly to pasture paddocks (Watson & Watson, 2015). Effluent holding ponds in the dairy sector are predominantly used for wet weather storage when effluent cannot be land applied. Anaerobic lagoons, which are often confused with secondary storage ponds, refer to the first pond in a two-pond system. Their primary function is to treat effluent in an anaerobic environment before it moves to secondary ponds for further processing or storage. However, if not carefully designed and managed, they could result in an increased risk to surface water bodies and groundwater (Houlbrooke et al., 2004; Laubach et al., 2015).

For these reasons, the sustainable management of livestock manure residues is an important challenge that dairy farmers face in Australia and around the world. It is essential to acknowledge that the trend towards intensification in dairy farming has a significant impact on manure collection and management (Dairy Australia and Agriculture Victoria, 2023). In PB systems, approximately 80% of daily manure output are excreted in open paddocks (Christie et al., 2018). However, with the intensification of the industry, there is a noticeable trend towards a more centralised approach to manage manure from centralised point sources (Dairy Australia and Agriculture Victoria, 2023). In such intensive systems, a significant amount of the daily manure excretion is collected from impermeable surfaces, such as concrete floors (Watson & Watson, 2015). This concentrated residue collection can further increase environmental risks when the industry intensifies and highlights the importance for recovery strategies (Dairy Australia and Agriculture Victoria, 2023). As a result, this topic has attracted ongoing research interest with the objective of developing sustainable strategies and practices. This is especially important considering the potential for high-yielding, intensive production systems to attract negative attention from the public, policymakers, and industry stakeholders. These stakeholders will increasingly inquire about how such systems are protecting the environment and addressing issues like greenhouse gas emissions and nutrient and water pollution risks.

Considering these risks and opportunities, the possibility of recovering, recycling, and reusing resources in manure residues is an important topic (Fyfe et al., 2016). Especially with the industry transition into intensification, nutrient as well as carbon recovery technologies can play an important role for a sustainable dairy industry.

1.3. Effluent treatment and recycling: From waste to resource

To recover resources in dairy effluent in practical and cost-effective ways, circular technologies for waste treatment are required (Williams et al., 2020). Such circular technologies at a dairy aim to reduce waste, improve efficiency, and enhance sustainability through closed-loop resource recovery (Chapter 5). Circular technologies enable dairy farmers to manage and utilize their waste more effectively, reduce environmental impact, and recover valuable resources (Chapters 2 and 5). This has the potential to lead to a win-win-win scenario for profitability, environment, and food production. Environmental impacts of dairy production can be reduced by decreasing nutrients and organic matter in effluent being stored or land applied (Hjorth et al., 2010). Especially, leaching potential to groundwater, run-off potential to surface water, and greenhouse gas emissions such as CH_4 or N_2O (Hjorth et al., 2010). Thereby offering the potential to create an alternative soil amendment and decrease global dependencies on synthetic fertiliser and energy (O'Brien & Hatfield, 2019).

This is important as the dairy industry currently faces economic challenges, such as the rising price of synthetic fertilisers. The escalating fertiliser costs documented by Schnitkey (2022), creates economic pressures in maintaining crop productivity, livestock operations, and ultimately milk production. In this context, the possibility of recycling and reusing nutrients in livestock effluent as an organic fertiliser alternative provides a promising solution to both environmental and economic challenges.

The second benefit is the possibility of on-farm energy production from waste. This would enable farmers to decrease their energy dependency on fossil fuels, while ultimately becoming more self-sufficient. This is especially important, considering the net-zero policy, as biogas can be stored and use when wind or sun is not available (Tauseef et al., 2013).

By transforming livestock effluent into nutrient-rich fertiliser and renewable energy, farmers can reduce input costs while potentially increasing their profits. In contrast, this can contribute to a more robust and resilient Australian dairy sector, capable of withstanding pressures from fluctuating fertiliser and energy prices and other market uncertainties such as climate impacts (Guzman-Luna et al., 2022). This also aligns with global efforts to target climate change and protect the environment for future generations (Ouikhalfan et al., 2022).

The general purposes of circular technologies can be categorised as followed (See Chapter 2 for a full description):

1) Nutrient recovery and reuse: The aim is to recover valuable nutrients from manure residues, such as N, P, and K. This might keep them out of waterways, including groundwater and finally reuse them beneficially as fertilisers. Technologies such as solid-liquid separation, anaerobic digestion, and composting allow these nutrients to be captured, processed, and reused. This might reduce the dependency on synthetic fertilisers and improving soil health via the addition of carbon in soil amendments.
2) Renewable energy production: Another objective is to convert waste into bioenergy. A way to achieve that is through the production of biogas via anaerobic digestion. Biogas can be used to power the farm, thus reducing dependency on non-renewable energy sources. The residual digestate from this process can also be used as a nutrient-rich fertilizer.
3) Reduced environmental footprint: Dairy farms that manage effluent more effectively can lower the risk of nutrient runoff into nearby water bodies and therefore prevent potential eutrophication. Additionally, by adding no or minimal amounts of organic loading to effluent ponds, fugitive CH_4 levels can be reduced, contributing to the mitigation of climate change.
4) Water recycling: Large amounts of water are used in the dairy industry and circular technologies can achieve water recycling within farm operations. This is likely to lead to fewer freshwater inputs and minimizing the demand on local water resources.
5) Economic viability: Ultimately, these technologies supposed to contribute to the farm`s economic sustainability by reducing input costs (such as for energy, fertilisers). Therefore, potentially creating new revenue streams (i.e., selling excess recovered energy or nutrient-rich fertilisers/soil amendments).

1.4. Focus of study

The key research and development challenge addressed in the thesis investigations is that dairy farmers currently lack practical, effective, and economically feasible options for circular resource recovery from on-farm manure residues. The thesis investigations seek to clarify novel and closed-loop concepts and options that also address current on-farm practicality issues as well as environmental concerns (e.g. emissions abatement). Australian dairy farmers desire solutions that work well and are affordable to manage, and farmers are often inherently aware of but currently unable to utilise the energy and nutrient resources in their on-farm manure residues.

To identify and clarify technology options to capture, reduce, and recycle resources from on-farm dairy residues, the following specific objectives are addressed by the thesis investigations:

1. To understand the variation in physico-chemical characteristics of effluent generated from different dairy production systems and how this might influence recovery strategies. This is addressed in Chapter 4 (Draft; Paper 1) of the thesis.
2. To optimize carbon and nutrient recovery from dilute dairy effluent through chemically enhanced solid-liquid separation techniques. This is addressed by the investigations outlined in Chapter 5 (Paper 2; Published) of the thesis.
3. To evaluate the biogas production potential of manure residues from diverse dairy production systems in Australia by measuring their biochemical methane potential. This is addressed by the investigations outlined in Chapter 5 (Paper 3; Published) of the thesis.

CHAPTER 2: LITERATURE REVIEW

This chapter provides an overview of existing literature on effluent management systems, dairy effluent characteristics, biochemical methane potential, and resource recovery technology options. The current state of knowledge is summarized, whereas challenges and key gaps are highlighted. Firstly, the most common dairy production systems, namely PB, PMR, and TMR (Section 2.2.), are briefly explained with their corresponding manure management (Section 2.2.1). This is to provide a general understanding of the dairy farming industry and subsequently highlights the challenges in dairy manure management. The focus is to gain control over the manure residue streams for the beneficial recycling of nutrients and organic matter (carbon). To identify efficient and practical recovery strategies, the physiochemical characteristics of manure residues is explained in detail (Section 2.2.2.). Consequently, solid-liquid separation is then introduced as a potential solution to gain control over effluent and to enable efficient nutrient and organic matter recovery (Section 2.2.3). Finally, the benefits and opportunities of chemically enhanced solid-liquid separation are outlined for increasing separation and recovery efficiency (Section 2.2.4).

Chapter 2.3. explores the details of biochemical methane potential and begins with fugitive methane emissions released by dairy manure management (Section 2.3.1). This will clarify the difficulties and implications of on farm greenhouse gas emissions. Section 2.3.2 then discusses anaerobic digestion as a potential solution for biological waste treatment, carbon abatement, and energy recovery. This section will not only describe the anaerobic digestion process but also highlights its potential for carbon emission reduction. Therefore, contributing to climate change mitigation and renewable energy generation. Consequently, practical technological solutions for establishing anaerobic digestion in the Australian dairy sector are listed in Section 2.3.3. The biochemical methane potential of the Australian dairy industry is then addressed in Section 2.3.4. This will provide a comprehensive understanding of recovery potentials and how they fit into the national context of Australia.

Composting of separated dairy solids is next introduced as another possible alternative for carbon abatement and resource recovery (Section 2.4). This is to demonstrate practical compost techniques, with their advantages and disadvantages as an alternative or addition to anaerobic digestion.

Finally, the literature review closes with Section 2.5, summarising the significant gaps and forming the present thesis objectives. The purpose of Chapter 2 is to provide clarity on the research challenges that remain currently unsolved and will be addressed in this work.

2.1. Dairy production systems, manure management, manure residue characteristics, and potential recovery strategies

Manure management and associated environmental challenges are expected to be influenced by the type of dairy production system (Soteriades et al., 2020). PB systems, in which cows source most of their daily feed from grazing paddocks, have a long history in many parts of the world (Aarons et al., 2020), and are still dominant in Australia and New Zealand. Grazing systems are often seen as more sustainable from an environmental and welfare perspective compared to intensive feeding systems, as they can reduce the need for feed inputs, reduce greenhouse gas emissions from manure management, and facilitate favourable welfare outcomes (Latham, 2010). However, PB systems also have their own environmental challenges, which can include the potential for overgrazing, erosion, and nutrient accumulation if not properly managed (Rojas-Downing et al., 2017a). Additionally, extreme weather conditions such as drought and flood could restrict the grazing capability and accessibility of land, influencing herd size and productivity (Godde et al., 2021).

Intensive production systems use TMR or PMR feeding systems (Chapter 1) which can be more efficient in terms of feed conversion and milk production (Fontaneli et al., 2005), but typically also capture more manure as a potential point source of nutrients and organic matter pollution (Powell et al., 2005). This can therefore have higher greenhouse gas emissions (carbon footprint) from manure management than PB (Williams et al., 2020). This can be caused by the larger proportion of the daily manure output from the milking herd being collected during feeding and milking, which is often stored in uncovered effluent ponds. In contrast, with PB, manure is excreted directly onto pastures.

In PB, only a small portion of the manure is captured and available, specifically from the milking area, and a large amount of water is typically used for cleaning the milking shed, resulting in a highly dilute effluent with implications for resource recovery potential (Birchall et al., 2008; Tait et al., 2021a). Thus, the production system has an impact on the amount and characteristics of manure being collected, which also influences its ability to be further processed for recovery and beneficial reuse of nutrients and bioenergy (Birchall et al., 2008; Powell et al., 2005).

2.1.1. Overview of dairy production and manure management systems

Manure management systems are commonly designed to match specific farm requirements. This includes existing infrastructure, production systems (e.g. PB vs. intensive) and the maintenance capabilities of the farm staff (Birchall et al., 2008; Tauseef et al., 2013). In table 1, a general overview of typical manure management systems relevant to dairy farms globally, with their definitions according to the IPCC guidelines (IPCC, 2019), is presented. Manure management practices can range from leaving manure as is (e.g. direct deposition onto Pasture/Range/Paddock areas) to a more complex combination of removal, storage, treatment, and land-application (IPCC, 2019). These practises can influence the potentials for nutrient recovery or even anaerobic digestion for biogas energy production (Birchall et al., 2008; IPCC, 2006). Each system has an individual combination of processes, with associated implications for GHG and resource recovery potential (water, nutrients and organic matter). This includes the use of solid storage with differences like covering and compacting, the addition of bulking agents or additives, or the use of anaerobic digesters with varying degrees of leakage control, as demonstrated in Table 1 (IPCC, 2019; IPCC, 2006). However, not all of the manure management systems in Table 1 are prominent in Australian dairies. For example, there are only a handful of Australian dairies that utilise anaerobic digestion systems and aerobic treatment. Understanding these systems is the first step to address the impact of manure management on the environment, particularly in terms of CH_4 emissions. This can facilitate the identification of potential and practical resource recovery options.

Table 1 Definitions of manure management systems (adapted from (IPCC, 2019))

System		**Definition**
Pasture/Range/Paddock		The manure from pasture and range grazing animals is deposited and not managed.
Daily spread		Manure is routinely removed from a confinement facility and is applied to cropland or pasture within 24 hours of excretion.
Solid storage		The storage of manure, typically for a period of several months, in unconfined piles. Solid stores can be covered or compacted. Bulking agent or additives can be added.
Solid storage-Covered/compacted		Like solid storage, but the manure pile is a) covered with a plastic sheet and/or b) compacted to increase the density and reduce the free air space within the material.
Solid storage – Bulking agent addition		Specific materials (bulking agents like sawdust and straw) are mixed with the manure to provide structural support and allow the natural aeration of the pile.
Solid storage – Additives		The addition of specific substances to the pile to reduce gaseous emissions.
Dry lot		An open confinement area without any significant vegetative cover Manure may be removed periodically and spread on fields.
Liquid/Slurry [1]		Manure is stored as excreted or with some minimal addition of water or bedding material in outdoor ponds, removed and spread on fields. Manure is agitated before removal from the pond to ensure that most of the volatile solids (VS) is removed.
Uncovered anaerobic lagoon		Anaerobic lagoons, also called holding ponds, are designed with varying lengths of storage and operated to combine waste stabilization with storage. The supernatant water may be recycled as flush water or used to irrigate/fertilize fields.
Pit storage below animal confinements		Collection and storage of manure usually with little or no added water typically below a slatted floor in an enclosed animal confinement facility. Manure may be pumped out of the storage to a secondary storage tank or stored and applied directly to fields.
Anaerobic digester	Digesters high quality and low leakage	Animal manure with and without straw is collected and anaerobically digested in a containment vessel. Digesters are designed, constructed, and operated according to industrial technology standards for waste stabilization by the microbial reduction of complex organic compounds to CO_2 and CH_4. Biogas is captured and used as a fuel.
	Digesters with high leakage	Digesters are not designed to industrial technology standards, but still capture CH_4 for destruction or used as fuel.
Burned for fuel		The dung and urine are excreted on fields. The sun dried dung is burned for fuel.
Deep bedding		This manure managementsystem is also known as a bedded pack manure management system and may be combined with a dry lot or pasture. Manure may undergo periods where animals are present and are actively mixing the manure, or periods in which the pack is undisturbed.
Composting	In vessel[2]	Composting, typically in an enclosed channel, with forced aeration and mixing.
	Static pile	Composting in piles with forced aeration but no mixing, with runoff/leaching.
		Composting in piles with forced aeration but no mixing, without runoff/leaching
	Intensive windrow[2]	Composting in windrows with regular turning for mixing and aeration. Runoff/leaching containment
		Composting in windrows with regular turning for mixing and aeration, no runoff/leaching containment
	Composting - Passive windrow[2]	Composting in windrows with infrequent turning for mixing and aeration, with runoff/leaching.
		Composting in windrows with infrequent turning for mixing and aeration, no runoff/leaching.
Aerobic treatment		The biological oxidation of manure collected as a liquid with either forced or natural aeration. Natural aeration is limited to aerobic and facultative ponds and wetland systems and is due primarily to photosynthesis. Hence, these systems typically become anoxic during periods without sunlight.

[1] Covers on manure management systems can impact emissions of direct N_2O, CH_4 and NH_3. The cover material effects CH_4 and NH_3

[2] Composting is the biological oxidation of a solid waste including manure usually with bedding or another organic carbon source.

[3] Comparative definitions with the EMEP/EEA Air Pollutant Emission Inventory 2016

2.1.2. Characteristics of dairy manure and effluent relevant for recovery and recycling

The characteristics of excrements produced by cows depend on breed, dry matter (DM) intake, and the composition of the diet (Aarons et al., 2020; Nennich et al., 2005). Manure and urine are often collected as dairy effluent produced by normal milking and feeding operations (Tait et al., 2021a). Dairy effluent is a heterogeneous material that consists of cellulose fibers, non-cellulosic carbohydrates, lignin, organic and non-organic nitrogen (N) like proteins, urea, uric acid, and ammonium, as well as other elements such as phosphorus (P), potassium (K), calcium (Ca), and magnesium (Mg) (Le Guen et al., 2017; Longhurst et al., 2000). This composition is important because it dictates the inherent value of the effluent in terms of nutrients and biochemical methane potential for potential recovery (Section 2.3).

Importantly, dairy effluent consists mainly of wash water (especially in PB systems in Australia) and some cleaning chemicals mixed with a reduced proportion of cattle urine and dung, spilt feed, and additives deposited only by cows when they are on impermeable surfaces such as in the milking shed, dairy holding yards, or intensive feeding surfaces for short periods of time in a day (Birchall et al., 2008). Consequently, dairy effluent is usually pretty dilute with a low DM content of 0.5-1.2% (Birchall et al., 2008). This is a challenge for many closed-loop technologies because the efficacy of nutrient and/or organic matter recovery and processing is typically reduced with dilute waste streams (See further below). Abroad, cows are more frequently housed indoors, which can greatly increase manure capture and increase the DM content of the resulting dairy effluent. These differences in effluent characteristics that can result from these differences in dairy production system types (PB vs. intensive feeding dairies) are currently a key knowledge gap influencing the evaluation of closed-loop technology options.

Dilute effluent is not cost-effective to transport for processing or beneficial reuse, except over short distances, and this strongly limits viable closed-loop options. One approach that has been extensively investigated for dairy effluent abroad is solid-liquid separation (Hjorth et al., 2011). This involves separating the dilute dairy effluent into solid and liquid fractions, which enables the recovery of valuable nutrients and organic matter, eases further transport and processing of the solid fraction, and potentially allows the use of both fractions for biogas energy and fertiliser nutrient sources. A key determining feature for separation is the particle size of dairy manure. Figure 1 shows the characteristics of dairy manure regarding the particle size distribution of total solids (TS), total nitrogen (TN), total phosphorus (TP) and K, and shows how the majority of nutrients are in particle sizes smaller than 0.125 mm, specifically 86% of N, 85% of P, and 99.8% of K (Meyer et al., 2007). In addition, particle sizes smaller than 0.125 mm contain 46% of TS (Meyer et al., 2007), which would be mostly manure organic matter and is the component of interest for biogas production (Tait et al., 2021a). Similarly, another study indicated that the majority of organic matter was found in particles >25 μm, but N and P were found in particles <25 μm (Peters et al., 2011). This is important because mechanical separation, such as via screening, can be cost-effective to run on-farm at dairies but may only be able to capture larger particles, with most of the nutrients and organic matter then not being removed or recovered to the solid fraction, except using flocculation chemicals as described below.

2.1.3. Solid-liquid separation to recover nutrients and organic matter

Solid-liquid separation treatment divides effluent into a liquid and solid fraction and can therefore recover and recycle valuable nutrients as well as organic matter (Hjorth et al., 2011). The removal of organic matter contributes to reducing CH_4 emissions from dairy effluent stored in effluent holding ponds (Amon et al., 2006; Laubach et al., 2015).

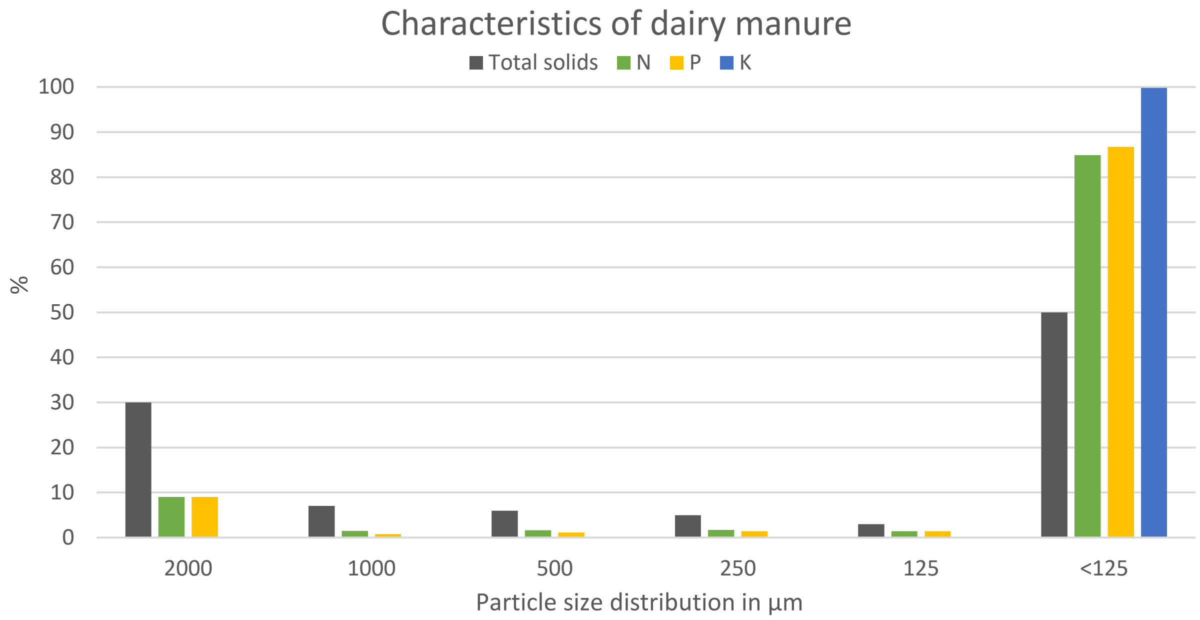

Figure 1 Characteristics of dairy manure (adapted from data in (Meyer et al., 2007; Peters et al., 2011)).

Furthermore, it minimizes the risk of ground- and surface water pollution (Houlbrooke et al., 2004), by recovering nutrients so the amount of nutrients in effluent being applied to land can be controlled and/or minimised. Overall, the separation process could have multiple benefits for the farmer because the liquid fraction contains a reduced level of particulate organic matter and P. Therefore, nutrients and DM might be easier to handle for cropland application. This is especially important in wet regions with heavy rainfall and sensitive environments (Drewry et al., 2006), where the impact of water pollution can be significant through effluent pond overflows, leaching from poorly constructed effluent ponds, over-application of effluent, or nutrient run-off. Furthermore, the liquid fraction can also contain a reduced level of heavy metals and pathogens if these are also separated out. Meanwhile, the production of solid fraction as a by-product enables an easier export for agronomic use in various farm areas, as does the possibility of composting or anaerobically digesting the solid fraction in a more compact digestion system, with dilutions potentially provided as required by adding liquid effluent.

Solid-liquid separation can also enhance the anaerobic biological process by removing indigestible material from the liquid fraction being digested and/or increasing digestion spatial loading efficiency by producing a concentrated digester feed from an originally dilute effluent (Burton, 2007). For example, this could help overcome the hydraulic loading limitations of wet mixed anaerobic digesters by having more concentrated digester feeds.

Research on dairy effluent and separation is global (Hjorth et al., 2011; Moller et al., 2007; Rico et al., 2012). However, very few Australian research studies have been conducted previously on this topic. This is important because of the predominance of PB production in Australian dairies, which is not shared with many other parts of the world (except New Zealand). A relevant study could be found in Western Australia, but it was conducted on raw pig effluent, where 80% of N & P were recovered in the solid fraction by chemical treatment with a flocculant and subsequent solids separation (Payne, 2014). Studies have observed a significant variation between separated solids from different animal manure types in commercial operations (Jorgensen & Jensen, 2009), so data on piggery effluent may not be representative of the operational performance expected for dairy effluent.

International studies to date have been mainly conducted in laboratories and controlled field environments and have focused on screw press, centrifuge, belt press, and sedimentation separation (Drosg et al., 2015; Hjorth et al., 2010; Watts et al., 2002). Some studies indicate that the separation efficiency for total N and DM was highly dependent on the DM content of the manure, while total P was only slightly affected by this factor (Moller et al., 2007). Generally, dry matter recovery increases with increasing DM concentrations in the effluent (Birchall et al., 2008). This is important because of the dilute dairy effluents expected to be typical for Australian PB systems.

Vanotti and Hunt (1999) considered that most of the equipment available for slurry separation was not efficient for nutrient removal (N, P, K) because the nutrients were mostly found in fine particle suspensions (Section 2.1). Similar results on manure showed that screening with a filter mesh of 0.1 mm was not enough to remove significant amounts of N and P (Fangueiro et al., 2010). Chemical pre-treatment (i.e. added before separation) and chemically-assisted separation may increase the capture of smaller Norg- and P-rich particles into larger particles between 25 and 1000 µm (Peters et al., 2011). However, studies on dilute effluent from Australian dairy farms were missing. Some international research has tested synthetic dairy effluent made up of cow excreta diluted in water to DM concentrations similar to those in Australian dairy effluent (Ellison & Horwath, 2021; Liu et al., 2016; Sherman et al., 2000). However, no results were found in the literature for direct testing on real dairy effluent, which may have distinct characteristics from synthetic effluent.

2.1.4. Chemically enhanced solid-liquid separation

Chemicals used for aiding solid-liquid separation are coagulants and flocculants. Coagulants added to treated water hydrolyse, forming insoluble precipitates and/or release cations that destabilize fine particles by screening/neutralizing their surface charge, thereby reducing the repulsive force to facilitate the formation of bridges between them (Crittenden et al., 2005). This is important because the typically negative net charge on organic matter particulates unwantedly encourages electrostatic repulsion and dispersion in an aqueous phase rather than aggregation, keeping fine particles apart and making their separation more difficult. Flocculation involves the aggregation of destabilized particles for which the electrical surface charge has been screened or neutralized, such as via coagulation, causing the fine particulates to clump together into larger flocs that are easier to separate.

Polymer flocculants are either cationic (positive charge) or anionic polymer (negative charge) molecules with charged functional groups to induce coagulation and a polymer chain to induce flocculation by forming bridges between individual particles (Crittenden et al., 2005). Generally, anionic polymers are preferred for environmental applications because of their extremely low aquatic toxicity compared with cationic forms. The positive charges of cationic polymers make them toxic to aquatic organisms when dissolved in water (Guezennec et al., 2015). A flocculant manufacturer had advised that very rapid hydrolysis of the chain on cationic polymer flocculants removes cationic charges and therefore could make the effect on aquatic organisms insignificant (SNF, no date). However, the use of polymer flocculants on-farm in agriculture would need to consider the ancillary effects of these chemicals, such as potential environmental impacts.

Chemical pre-treatment with polymer flocculant before separation is common to significantly increase the removal efficiency of the nutrients and manure organic matter (OM), as small particles are captured in the solid fraction (Hjorth et al., 2010; Jorgensen & Jensen, 2009). Past studies have demonstrated the possibility of reducing P in dairy effluent to low levels with the addition of alum and ferric chloride solutions, but the economics of the treatment typically did not appear to be favourable (Sherman et al., 2000). A recent study tested three different coagulants, specifically poly-aluminium chloride, ferric and alum, without mechanical separation and was able to minimize organic material, nutrients (N and P) and pathogens in the supernatant liquid fraction while obtaining sludge with properties more favourable for land application than raw dairy effluent (Mohamed et al., 2020). This study aligns with a laboratory test conducted in Spain to replicate the characteristics of dairy effluent by means of screening and coagulation–flocculation treatments (Rico et al., 2007).

Flocculation of dairy cattle manure with TS concentrations of up to 140 g kg^{-1} through the addition of a strong cationic polyacrylamide flocculant allowed the elimination of up to 90% of TS, using optimum doses of approximately 43.9 g kg^{-1} of TS (Rico et al., 2007). The effects of the flocculant on the separation efficiency increased linearly with the TS concentration (Rico et al., 2007), which is also the reason why dosages are often normalised to TS. The solid fraction retained 29.1% of total initial mass, as well as 76.1% of TS and 79.9% of VS (Rico et al., 2007). In addition, 59.4 and 87.4% of TKN and TP, respectively, were found in the solid fraction (Rico et al., 2007). However, the assessment of practical operation and economic suitability for full-scale implementations are lacking for many innovative technologies, such as chemical treatment for nutrient recovery from manure residues (Mehta et al., 2015).

2.2. Composting of separated dairy solids as a carbon abatement and recovery option

The biological process of composting can also be described as the breakdown and stabilisation of organic matter, such as manure residues (Bernal et al., 2009). This, then results in the formation of stable, humus-like substances, which can promote soil health (Bernal et al., 2009). The composting process predominantly occurs in an aerobic environment, where microorganisms (bacteria and fungi) decompose organic material in the presence of oxygen (Bernal et al., 2009). This microbiological process produces CO_2 and heat (exothermic process) and eliminates seeds and pathogens (Yuan et al., 2018). Furthermore, CH_4 and N_2O can occur during composting, if there is an oxygen deficit or nitrogen surplus, respectively (Biala et al., 2016). Their levels tend to remain relatively low in composting systems that are effectively managed (Biala et al., 2016). Mulbry and Ahn (2014) have observed that a substantial portion of the overall CO_2 and CH_4 emissions are released during the first two weeks of composting.

The end product of this decomposition process is commonly known as compost, which can serve as a soil enhancer by providing essential nutrients and stabilised OM (De Rosa et al., 2021). Its application to agricultural land is a proven strategy for soil carbon sequestration to mitigate climate change (Lal, 2004). Additionally, this can positively influence soil fertility and the retention of water in soils, ultimately contributing to food security (Lal et al., 2007). A relatively simple method to compost dairy manure directly on-farm, is static pile composting, where manure is stockpiled and occasionally mixed (Ahn et al., 2011). The CH_4 conversion factor for compost is significantly lower than for most other processing pathways (IPCC, 2006) (Table 2), meaning that fugitive CH_4 emissions from composting can be relatively low. However, the IPCC (2019) recommends confirmation of low emission profiles from composting due to limited studies being available.

The low solids concentration of dairy effluent (Table 3) poses significant challenges for on-farm handling and management and prevents composting of the material on its own. One possible solution is the solid-liquid separation of the effluent to produce a potentially compostable solid fraction (Section 2.2.3). Zhong et al. (2018) demonstrated the natural composting of separated dairy solids. The liquid fraction could be treated, stored, and then applied to land. The efficiency of separating and concentrating nutrients and carbon in the solid fraction can be enhanced by using coagulation and flocculation agents, as demonstrated by Wang et al. (2020).

The use of cationic polyacrylamide polymers (PAMs) for efficient resource recovery has become more important in the field of manure management (Ellison & Horwath, 2021; Tabra et al., 2020). For the process of flocculation, water-soluble polymers demonstrate a promising performance, as they promote the aggregation of particles (Arp and Knutsen, 2019). This leads to the formation of bigger clusters, which ultimately can be extracted by using a screen or filter from the liquid phase (Arp and Knutsen, 2019).

PAMs are also widely used for sludge dewatering in municipal and commercial wastewater treatment facilities. As Liu et al. (2016) have shown, the use of PAMs can contribute to the reduction of pathogen levels during the separation of effluent. However, the use of PAMs for enhanced solid-liquid separation might pose the risk of introducing the cationic polymer into the wider environment, particularly in cases where sludge is applied to agricultural fields (Hennecke et al., 2018). Most studies address improper land application, the contents of trace elements, organic compounds, and pathogens, but not the content of C-PAMs. Only a limited number of studies are available focussing on the environmental impacts of manure application with PAM. One study showed high phytotoxicity of C-PAMs in solutions above concentrations of 1,000 mg L^{-1}, but no negative effect was detected when C-PAMs at the same concentrations were mixed into the experimental substrate (Tabra et al., 2020). A significant negative effect of C-PAM in growing media was only observed at concentrations above 5,000 mg L^{-1} for radicle elongation, aerial biomass, and radicle biomass (Tabra et al., 2020). There seems to be a significant lack of understanding about the fate of PAMs in soil and the effects they have on soil organisms. This represents a notable knowledge gap regarding the possible consequences of using PAMs in agricultural soils (Hennecke et al., 2018). Toxicity mitigation through composting can be a potential pathway for agricultural waste management (Kapanen & Itavaara, 2001). This is particularly important with regard to eliminating the negative effects of compounds such as C-PAM (Hennecke et al., 2018).

2.3. Biochemical methane potential of Australian dairy effluent

Manure production from milked cows has been extensively investigated in the literature, and well-defined empirical approaches are available to estimate excretion rates of cattle (Nennich et al., 2005), However, there is currently a lack of knowledge on the biochemical methane potential (BMP or B_0) for manure residues from PB dairies, and no data is available for Australian dairy effluent. Biochemical methane potential (B_0) is a measurement that determines the methane production potential from anaerobic digestion of any specific organic material under controlled conditions (VDI 4630, 2016). Anaerobic digestion (AD) occurs naturally during the decomposition of biomass, such as plant material and animal manure residues, in the absence of oxygen (Batstone & Jensen, 2011). In the IPCC guideline terminology, B_0 is often referred to as the maximum methane-producing capacity and is said to vary by diet and species in the case of manure (IPCC, 2019). Understanding the BMP and B_0 of livestock effluent is important for several reasons. Firstly, it allows the quantification of the amount of methane that can be generated from anaerobic digestion of livestock residues to evaluate biogas energy recovery opportunities. However, it is also important for assessing the environmental impact of livestock operations, with manure-management CH_4 contributing significantly to the Australian dairy sector's emissions profile (Christie et al., 2012; Christie et al., 2018; NGER, 2022). With the lack of B_0 data for dairy effluent from PB dairies, it is also not known how PB systems compare with intensive feeding systems (PMR, TMR) in terms of potential manure-management CH_4 profile as well as expected relative biogas potential. This is because substrates that are rich in lipids and easily degradable carbohydrates tend to have a higher CH_4 potential, while substrates that are more recalcitrant and made up of lignocellulosic materials (as pasture) can tend to have lower CH_4 yields (Labatut et al., 2011). This can be important because the diet of cattle could significantly affect the biodegradability of their manure and, thus, CH_4 emissions or recovery potential. Grass-fed cattle produce manure that is rich in slowly degrading lignin and cellulose, while grain-fed cattle would yield manure rich in quickly degrading lipids and acids (Labatut et al., 2011; Vidal et al., 2000).

Furthermore, proteins may play a crucial role, providing essential nutrients but potentially inhibiting digestion in high concentrations as a result of ammonia released from the digestion process (Capson-Tojo et al., 2020), but they may also be a good pH buffer (Tait et al., 2009). Dairy manure is globally known for its suitable characteristics for AD because of its nearly optimum C/N ratio of 15-30 (Godbout et al., 2010; Tauseef et al., 2013). The decomposition of organic matter during AD is influenced by the biochemical characteristics of the material and the rate of microbial growth and replacement, which can influence the retention volume (and thus size) required of a digester (Section 2.3.2). There are currently no studies on the impact of PB vs. TMR or PMR systems on manure effluent characteristics, including B_0, which leaves a gap in understanding of the implications for biogas recovery potential from these different production system types prevailing and emerging within the Australian dairy sector.

Manure residues are often managed in specifically combined systems (Section 2.2.1). For instance, liquid effluent can be flushed into an anaerobic lagoon after passing through solid-liquid separation and the solids can be composted. This reduces the organic loading rate into lagoons and therefore it is recommended to report the individual CH_4 emissions from each system to determine the overall carbon footprint (IPCC, 2019). It is possible to estimate the potential methane emissions and develop strategies to mitigate them, when the individual BMP/B_0 and MCF is known (Commonwealth of Australia, 2022). This can also provide valuable information for optimizing anaerobic digestion systems.

Anaerobic digestion is a process that captures methane from organic waste and converts it into biogas, which can be used as a renewable energy source (Abbasi et al., 2012). The knowledge of specific BMPs enables a more efficient design and operation of AD systems, maximizing methane capture and energy production (Lauer et al., 2018). Especially, the understanding of BMP from livestock effluent supports different waste management strategies.

The understanding of B_0 values of different treatment options can identify the most effective and sustainable approaches for handling manure residues to simply reduce fugitive CH_4 emissions or promote renewable energy production.

2.3.1. Fugitive methane emissions from dairy manure management

Fugitive CH_4 emissions from manure management are important, accounting for as much as 10% of total agricultural emissions in 2018 (Commonwealth of Australia, 2018). These emissions from manure management occur predominantly from manure management under anaerobic conditions, such as extended storage in uncovered lagoons (Laubach et al., 2015) commonly used in dairies across Australia (Birchall et al., 2008; Watson & Watson, 2015). There are several pathways to manage manure residues with individual emissions profiles (Aguirre-Villegas & Larson, 2017). These emission profiles are generally presented as methane conversion factors (MCF). MCF represents the proportion of B_0 that is released as fugitive CH_4 from specific management practices and environmental conditions (IPCC, 2006). Table 2 summarises MCF factors from manure across various manure management systems and those within different climate zones. The MCFs are expressed as percentages. Conventional manure management practices, such as uncovered anaerobic lagoons, liquid/slurry and pit storage, cattle and swine deep bedding, solid storage, dry lot, and daily spread, are associated with higher MCF than practices that are predominantly aerobic (e.g. composting).

Table 2 Methane conversion factors for manure management systems

		Temperate		Warm
		Moist	Dry	Range of values for tropical, montane, wet, and dry conditions
Uncovered anaerobic lagoon		73%	76%	76 – 80%
Liquid/Slurry, and Pit storage below animal confinements	1 month	13%	15%	25 – 42%
	3 months	24%	28%	43 – 62%
	6 months	37%	41%	59 – 74%
	12 months	55%	64%	73 – 80%
Cattle and Swine deep bedding (cont.)	> 1 month	37%	41%	59 – 74%
Cattle and Swine deep bedding	< 1 month	6.50%		18%
Solid storage		4.00%		5.00%
Solid storage – Covered/compacted		4.00%		5.00%
Solid storage – Bulking agent addition		1.00%		1.50%
Solid storage – Additives		2.00%		2.50%
Dry lot		1.50%		2.00%
Daily spread		0.50%		1.00%
Composting – In-vessel		0.50%		
Composting – Static pile (Forced aeration)		2.00%		2.50%
Composting – Intensive windrow		1.00%		1.5%
Composting – Passive windrow (Infrequent turning)		2.00%		2.50%
Pasture/Range/Paddock		0.47%		
Poultry manure with and without litter		1.50%		
Aerobic treatment		0.00%		
Burned for fuel		10.00%		
Anaerobic Digester (low leakage, high/ low quality gastight storage technology)		1.00 – 1.41%		
Anaerobic Digester (low leakage, open storage)		4.38%		4.59%
Anaerobic Digester (high leakage, high/ low quality gastight storage Technology)		9.59% - 10.85%		
Anaerobic Digester (high leakage, low quality technology, open storage)		12.97%		13.17%

Anaerobic digestion is somewhat unique in that it enables the controlled capture of CH_4 and its use to offset fossil fuels. However, some CH_4 is still released via leaks with anaerobic digestion, so its MCF is not zero.

2.3.2. *Anaerobic digestion: A biological waste treatment, carbon abatement, and energy recovery option*

Anaerobic digestion (AD) is a biological process by which complex organic matter is broken down or degraded into progressively more simple metabolic intermediates to ultimately produce CH_4 and CO_2 as terminal products (Batstone & Jensen, 2011). The treatment of organic waste through AD could serve as a more sustainable approach to reducing greenhouse gases and recovering renewable energy in the form of biogas (Batstone & Jensen, 2011). Agriculture residues are basically organic matter and can be a suitable feedstock for biogas production (Abbasi et al., 2012). The production of biogas through AD has been evaluated as one of the most energy-efficient and environmentally beneficial technologies for bioenergy production (Fehrenbach, 2008). Furthermore, it has been suggested to be the current best practice for manure treatment and one of the most efficient technologies to reduce the carbon footprint of dairy effluent (Belflower et al., 2012).

Anaerobic digestion can be broadly divided into four biological process steps: hydrolysis, acidogenesis, acetogenesis/dehydrogenation, and methanogenesis (Weiland, 2010) (Figure 2). Various microorganisms are involved in each of these degradation steps. Firstly, bacteria break down particulate and complex organic matter composed of long chains of complex carbohydrates, proteins, and lipids into smaller molecules like sugars, peptides, amino acids, and long-chain fatty acids by the process of hydrolysis (Weiland, 2010). Hydrolysis is generally considered to be the rate-limiting step in the anaerobic digestion of particulate material (Batstone & Jensen, 2011), such as is found in dairy manure residues. A fibre-rich diet and the inclusion of bedding material, such as straw, often provide cattle manure with a high lignocellulosic content (about 50% based on dry weight). Since lignocellulose is a highly resistant material, it is resistant to anaerobic degradation and restricts the first hydrolysis step (Batstone et al., 2009).

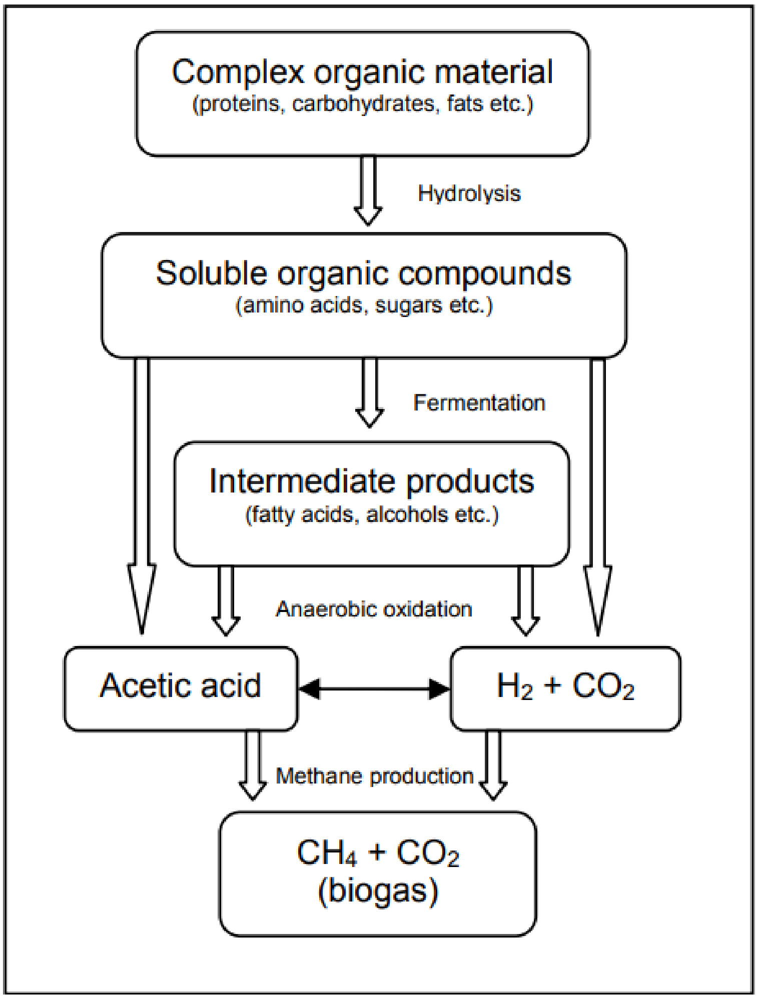

Figure 2 Overall steps of biological process anaerobic digestion (adopted from Batstone and Jensen 2011)

Acidogenesis involves the fermentation of the metabolites from hydrolysis to produce volatile fatty acids and alcohols, and syntrophic acetogenesis involves the digestion of the fermentation products into acetate (Batstone & Jensen, 2011). The generation of CH_4 is performed by two trophic groups of anaerobic archaea; one group (hydrogenotrophic methanogenesis) oxidizes H_2 and respires CO_2 ($CO_2 + 4H_2 \rightarrow CH_4 + 2H_2O$) which is coupled with syntrophic acetogenesis; the other group (acetoclastic methanogenesis) cleaves acetic acid into CH_4 and CO_2 ($CH_3COOH \rightarrow CH_4 + CO_2$) (Batstone & Jensen, 2011).

Both groups of methanogens are slow growers and require strictly anaerobic conditions, but acetoclasts are particularly sensitive to inhibition (Angenent et al., 2004; Buhlmann et al., 2019).

There are multiple digestion technologies available for livestock residues (Angelidaki et al., 2018). Digestion technologies are high-rate or low-rate. High-rate digestion systems, such as the anaerobic membrane bioreactor, UASB, or IC reactor, attempt to keep effective microorganisms by decoupling hydraulic retention time (HRT) from solid retention time via filtration or granular sludge. High-rate systems are often used for highly degradable organic matter feedstocks. Particularly, dairy manure can produce substantial amounts of organic acids by fermentation at short retention times and high organic loading rates (Coats et al., 2011), and it may be possible to utilise these organic acids subsequently in a high-rate AD system. Low-rate digestion technologies include covered effluent ponds (CEP), continuous stirred tank reactors (CSTR), and unmixed plug-flow (PF) reactors (Lauer et al., 2018). CEP is the most common digestion technology for cattle effluent in the United States (AgSTAR, 2022). CEP operate under ambient temperature conditions and are lined with an impermeable plastic cover to trap methane, typically designed for low-spatial-loading liquids with a solids concentration of up to 2% (Angelidaki et al., 2018). Due to a relatively long hydraulic retention time (HRT) of 35–60 days, CEP requires large footprints (Batstone & Jensen, 2011). Solid matter can be eliminated prior to digestion in a CEP to reduce sludge and/or floating solids accumulation under the cover (Angelidaki et al., 2018). Typical inputs and feeds for covered-effluent pond systems include dairy and swine manure (livestock manure) collected by flush systems. Emerging variants of CEP involve mixing and heating the feedstock, which may be effective in speeding up digestion (Ziganshin et al., 2016). However, this approach does not allow for the extended sludge/particulate manure solids retention of unmixed ponds, and as a result, it could become limited by hydraulic loading similar to CSTRs (Ziganshin et al., 2016).

CSTR is the predominant AD technology in Germany (Weiland, 2010) and consists of temperature-controlled reactors in which agitators mix the digestion gently. The HRT for heated digester types is at a minimum of 20 to 30 days to prevent washout, and as such, the solids content must be high enough (between 3 and 10% to minimise retention volume and thus cost) (Weiland, 2010). Solids can be eliminated both before and after digestion (Diaz et al., 2016). In the livestock industry, CSTRs are utilised in dairy, beef, and swine production.

Plug-flow digesters are horizontal tanks with intermittent feedstock addition into the reactor, pushing material along the length of the digester in a "plug" (Liebetrau et al., 2019). Without the use of agitators, a greater proportion of solid material (TS of 10 to 14%) is required to maintain a homogeneous mixture in the digester; otherwise, sand and grit settle out rapidly and occupy useful digester space (Li et al., 2021b). Comparable to those of complete-mix digesters, the HRT ranges from 20 to 30 days, but a plug-flow digester can benefit from first-order digestion rate kinetics typically observed for manure particulates (Adar, 2020; Batstone & Jensen, 2011). Plug-flow digesters are widely used on dairy farms in the United States (Tauseef et al., 2013). Manure suitable for CSTRs and PF is predominantly gathered by scrape collection techniques to prevent dilution with water (Angenent et al., 2004; El-Mashad & Zhang, 2010).

One of the key challenges associated with the AD of dairy effluent is its typically dilute form (Section 2.2.2). This can limit the AD in a low-rate technology such as CSTRs because of the long HRT required for hydrolysis of the particulates in the effluent, which translates into very large digester volumes because of the low DM content in the effluent. Solid-liquid separation can help solve these challenges by producing biomass with an increased DM content. Moller and Hansen (2007) highlighted the ability of pre-separation to increase the economic efficiency of AD as it concentrates VS and thus intensifies the AD process, as well as enhancing the economics of biomass transport to a centralized AD facility (i.e., carting less water).

The explanation is that methane yield on a VS basis is expected to be similar to that of non-separated manure, but the VS content is 5 to 10 times higher in a separated solid fraction than in non-separated manure. The separation efficiency can therefore influence the potential methane yield of the total separated biomass (Moller et al., 2007), if only the solid fraction is processed via AD. The same authors analysed the methane potential of the solid fraction of cattle and pig manure (Moller et al., 2007). A study by Amaral, Kunz et al. (2016) using a simple separation method based on settlement of swine effluent showed that the settled sludge fraction corresponding to 20–30% of the raw manure volume will produce 40–60% of the total methane yield. The methane potential of the settled sludge fraction was about 2 times higher on a wet mass basis than the supernatant fraction (do Amaral et al., 2016). A more detailed study on dairy effluent was established in Italy by Rico in 2012, where the different fractions were compared with each other (Rico et al., 2012). It was found that the liquid fraction has a higher methane potential than the solid fraction on an organic dry matter basis; however, it is anticipated that the interpretation of their results may have been influenced by the method by which these authors measured the VS in their liquid fraction. Specifically, volatile organic matter losses during the drying step of VS determination can incorrectly inflate methane yield normalised to VS because of the lower measured VS. It is expected (but yet to be confirmed) that methane yield on a VS basis should be similar for the liquid fraction and the solid fraction. (Rico et al., 2012) mentioned that the liquid fraction had yielded 90% of B_0 after 21 days, whereas the manure and the solid fraction required 48 and 52 days to reach this same percentage, respectively. This is important because it may indicate potential differences in degradation rates for the solid and liquid fractions due to compositional differences in their organic matter content.

The degradation rate also influences the economic feasibility of AD, especially by operation and design (Adar, 2020; Angelidaki et al., 2018). The use of polymer flocculant to enhance separation and VS proportion to the solid fraction can increase methane yield from digestion of the solid fraction. However, as highlighted above, the conditions of separation and use of chemicals could also influence the composition of organic matter in the solids fraction, thereby also influencing methane yield on an organic dry matter basis. This presents a key research gap that requires further exploration.

2.4. Overall summary of gaps to be addressed by the thesis

The understanding of nutrient availability in dairy effluent is the first challenge. This includes the differences in effluent characteristics of PB and intensive dairy systems. Synergies or differences in effluent characteristics, produced by these systems, can indicate potential effectiveness of recovery strategies and circular technologies applied across different dairy production systems. This understanding can improve the decision-making process for sustainable nutrient and manure management in the dairy industry. The gabs in literature as outlined in Section 2.2, indicate a need to investigate differences in effluent characteristics comparing PB vs. TMR or PMR systems. There is a scientific lack in researching the potential environmental impacts and most sustainable management strategies related to different production systems. To fully take advantage of resource recovery opportunities it is crucial to investigate the various dairy production systems and how they impact the environment.

The second challenge lies in the generally dilute nature of dairy effluent, more specifically the nutrient and organic particle sizes that are tiny and require a fine separation to enable effective recovery and beneficial reuse. This is especially the case for dairy effluent collected during the milking operation from PB dairies with a high dilution of water.

Technology options are required to facilitate transportation and provide farmers with a flexibility to control these resources. A potential recovery strategy is solid-liquid separation to enable the efficient recovery of nutrients and OM as resources from dilute effluent. A well-known challenge for solid-liquid separation of slurry or effluent are the fine particle size distribution of its OM and nutrient contents. Commercially available technologies, such as a mechanical screen or belt filter press faces problems to separate those small particles. The use of chemically enhanced separation approaches by the use of cationic polymers can increase the recovery of nutrients and organic matter. However, this approach appears to be ineffective at removing dissolved nutrients, while mobile nutrients are more challenging for the environment. There is a need to test practicality at full scale and gain scientific experience with chemically enhanced separation techniques for dilute dairy effluent treatment. This represents a clear research opportunity and would address an important data gap by clarifying solid-liquid separation options for the dilute dairy effluent typical of PB systems across Australia. This might enable effective recovery of nutrients and OM into useable liquid and solid fractions.

The main objectives that arise from these literature gaps can be summarized into two overarching goals: (1), to identify differences or synergies in effluent characteristics and nutrient concentrations between various dairy production systems. This could provide insight into potential relationships which could make resource recovery more efficient and (2) enable control over carbon and nutrient capture and recovery from dilute dairy effluents, specifically focusing on solid-liquid separation techniques.

The last challenge lies in the unknown methane potential of pure pasture based dairy cattle, where detailed data is missing. This introduces complexity around the potential to recover CH_4 from dairy residues across Australia. There is also an uncertainty regarding the potential impact of forage on the production of CH_4 between different production systems.

There are currently no published studies available that have measured B_0 for on-farm manure residues across the Australian dairy sector, neither for PB systems in general. This leaves an important uncertainty in predicting biogas energy recovery potential as well as estimations for evaluating fugitive methane emissions for GHG accounting in the Australian dairy sector. Australia's National Inventory (NGER, 2022) currently uses the IPCC default factor B_0 of 240 $LCH_4 \cdot kgVS^{-1}$ for dairy cattle manure. However, the IPCC (2006) also suggested that specific country values for B_0 should be measured and applied, especially for livestock because of varying diets and species. The section also highlighted the potential influence of various production systems, including different diets and manure management systems (e.g. solid-liquid separation) on B_0, thus their influence on sector emissions and biogas potential. This is a key research gap that requires further exploration.

To address the key knowledge gaps and challenges, a set of dedicated thesis research objectives was formulated and presented in Section 1.4.

CHAPTER 3: GENERAL METHODS

The thesis investigation involved extensive sampling and analysis of manure effluent from real commercial dairy farms. The complexity of conducting such sampling should not be understated. It is important to acknowledge that commercial farm systems hold a higher degree of variability compared to controlled laboratory conditions. This includes fluctuations in effluent stream composition and consistency, diversities in farming practices, and the influence of seasonal variations. Hence, a straightforward, 'one-size-fits-all' approach to sampling, data collection, and analysis is rarely effective in such contexts. For this reason, the current chapter outlines general principles that were adopted for the investigations of the thesis (Section 3.1). The main aim is to ensure representative sample collection to accurately represent the dynamic effluent stream.

In addition, theoretical approaches were used to estimate manure nutrient and VS productions for cross-validation of the sampling and analysis data throughout the thesis. The procedures used for these theoretical estimates are briefly outlined in this chapter (Section 3.2).

The Z-Filter was a solid-liquid separation technology that was directly investigated in Chapter 5 and from which samples were collected for investigations in Chapters 4 and 6, and so was a key component across the entire thesis. The author actively participated in the practical installation and commissioning of a Z-filter trail at a dairy farm in Western Australia. Because of its importance to the thesis investigations and for clarity, the Z-filter and peripheral farm infrastructure are outlined in Section 3.3 of this chapter.

3.1. On farm effluent sampling

Representative sample collection was achieved by combining approximately twenty subsamples, each measuring a significant quantity (one litre each). Detailed information on farms, manure collection, and management systems is listed in Chapters 4, 5, and 7. Care is taken to collect effluent at the outflow of the dairy shed, feedpad, or housing complex progressively across a whole washdown event, comprising a composite of time-proportional grab samples (total about 20 L). The selection of the location to sample took into consideration safe access and being able to source a sample that is representative of the whole flow (e.g. avoiding solids segregation). To prevent the settling of solids, the aggregated composite sample is usually stirred continuously before representative sub-sampling occurs into smaller sample bottles. Manure samples were also collected via scraping directly off a barn and feedpad floor into a central pile, collecting material from an area with a significant area of approximately 2.5 m radius. This was to ensure that enough material was collected for a representative sample. Typically, five manure piles were collected from the floor and combined in a bucket before being thoroughly mixed and a significant representative sub-sample collected (about 0.5 kg).

3.2. Statistical approaches for data validation

Nennich et al. (2005) developed regression equations to predict excretion of total manure, total DM, N, P, and K for lactating cows. The predictors used in the regression equations for lactating cows included milk yield, percentages of protein and fat in milk, dietary concentrations of crude protein and neutral detergent fibre, and intakes of nutrients. The paper by Nennich et al. (2005) ultimately simplified the regression equations for excreted amounts as relationships only with the milk yield of lactating cows as follows (Nennich et al. (2005)):

1. Manure excretion [$kg \cdot d^{-1}$] =

(Milk yield [$kg \cdot d^{-1}$] $\cdot$ 0.0874 ($\pm$ 0.007) + 5.6 ($\pm$ 0.3)

2. N excretion [$g \cdot d^{-1}$] =

(Milk yield [$g \cdot d^{-1}$] $\cdot$ 2.82 ($\pm$ 0.42)] + 346 ($\pm$ 18.1)

3. P excretion [$g \cdot d^{-1}$] =

(Milk yield [$g \cdot d^{-1}$] $\cdot$ 0.781 ($\pm$ 0.230)] + 50.4 ($\pm$ 8.6)

4. K excretion [$g \cdot d^{-1}$] =

(Milk yield [$g \cdot d^{-1}$] $\cdot$ 1.476 ($\pm$ 0.7207)] + 154.1 ($\pm$ 24.5)

These are total daily excretion rates, which can be multiplied by the fraction of daily time that the cows spend on surfaces from which manure is collected to determine manure capture rates. These estimates are indicative only of actual capture rates expected to fluctuate due to individual commercial farm operations and the ongoing challenges with dynamic effluent flows.

3.3. The Z-Filter as a solid-liquid separation technology

The Z-Filter is installed and operating at the single largest dairy in southwest Australia, carrying a herd of 1,400 animals and producing 11.5 million litres of milk and 36,500 cubic meters of manure effluent per year. The dairy previously used a conventional manure management system consisting of an effluent pit and four unlined, uncovered effluent holding ponds on an area of 10 hectares. Before the trial, effluent was typically applied over an 8-hectare sacrificial paddock near the milking shed, with little agronomic benefit. The Z-Filter is a mechanical solids separation device that functions like a belt filter press, which is explained in detail in Chapter 5. Figure 2 presents a flowsheet illustrating the Z-Filter ("Z"), pumps and ancillary equipment installed for the trial and as part of the on-going normal operation of the Z-Filter at the farm (Chapter 5).

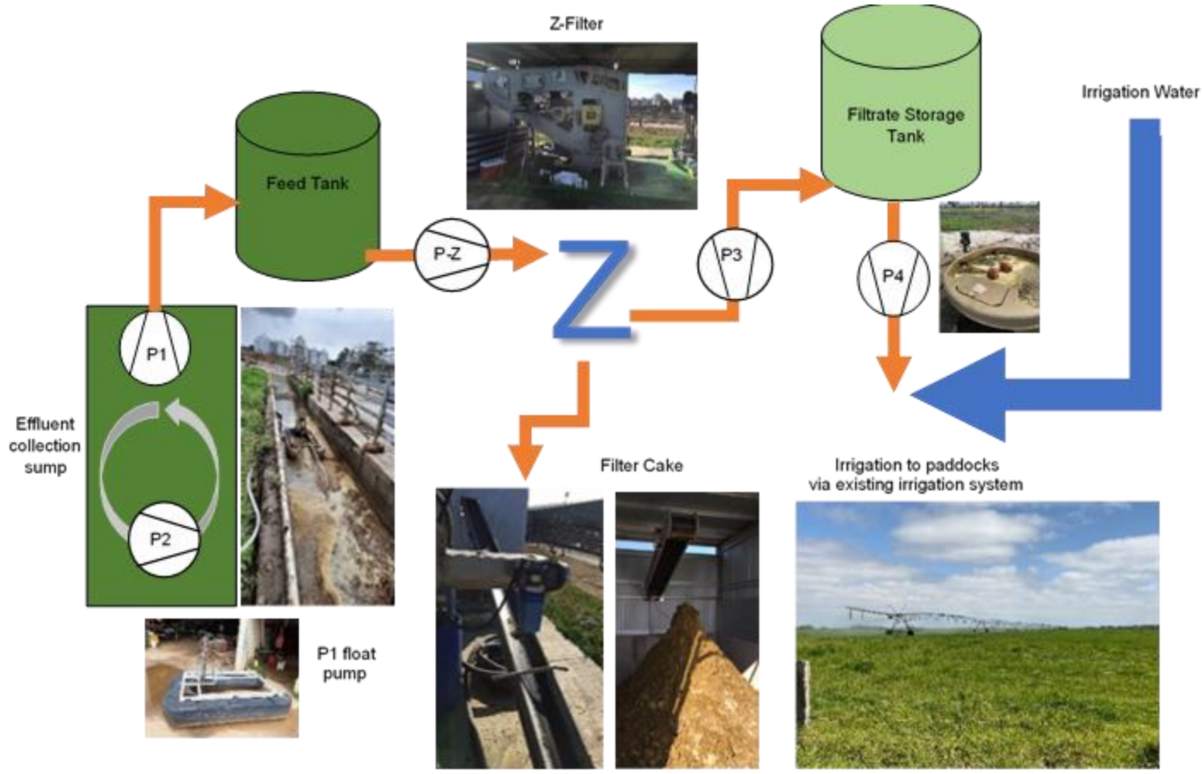

Figure 3 Structural implementation of the Z-Filter on the trial dairy farm

CHAPTER 4: PAPER 1 – IMPACT OF AUSTRALIAN DAIRY PRODUCTION SYSTEMS ON COMPOSITION AND RECOVERY OF MANURE NUTRIENTS

In late draft, aim for submission to the Journal of Environmental Management.

This research paper addresses Research Objective 1 of the thesis, "To understand the variation in physico-chemical characteristics of effluent generated from different dairy production systems" and how this might influence recovery strategies. In addition, the study adds to the existing literature compositional data for dairy manure residues, which is of importance for quantifying greenhouse gas emissions, optimising resource utilisation, and implementing sustainable manure management practices.

The study utilised the methods outlined in Chapter 3 (Section 3.1) to sample dairy manure residues from several commercial dairies across Australia, with different production systems, geographical locations, and farm operations. The composition of these residues was then analysed in detail to also investigate implications for circular technology options. While the same samples are also used in the investigations of B_0 in Chapter 5, the analyses for which results are presented in this chapter instead focus on nutrients, for which data are not presented in Chapter 5.

Impact of Australian dairy production systems on nutrient composition and recovery of dairy manure residues

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Keywords: Pasture based; Dairy effluent; Circular economy; Resource recovery; Natural resources; Fertiliser

Abstract:

The influence of dairy production systems on effluent characteristics and capture is important for nutrient management and recovery strategies. This study measured the composition (including nutrients) for several dairy effluent and manure residue streams sampled from different production systems across Australia. Twenty-two sample types were collected from 11 dairy farms across four important production states. Effluent/residue volumes and concentrations were used to estimate total nutrient capture rates, and correlations between components in the effluent/residues were investigated. No significant differences in effluent nutrient concentrations were found between the different production systems, likely due to an expected high variability within each on-farm production system type. However, total capture rates were significantly higher in intensive indoor systems than in extensive pasture-based systems ($p<0.1$), indicative of cows spending more time on concrete when indoors. Furthermore, observed correlations indicated dependent relationships among certain components in the effluent, indicating the possibility of precipitation reactions enhancing recovery. Also, nutrient forms differed between mobile and particulate phases, influencing nutrient management and recovery strategies. These findings are important as farms in Australia increasingly transition between production systems (i.e. extensive vs. intensive), changing nutrient capture rates as a point source, and influencing nutrient resource recovery opportunities.

4.1. Introduction

The sustainability of agriculture faces critical challenges into the future in terms of resource use efficiency and greenhouse gas (GHG) abatement (Rosa & Gabrielli, 2023; Willett et al., 2019). Specifically, conventional agricultural practices primarily rely on synthetic fertilizers for food crops (Rosa & Gabrielli, 2023), with synthetic nitrogen (N) fertiliser feeding 48% of the world population. However, N fertiliser production is energy intensive with substantial associated anthropogenic carbon emissions via fossil-fuel use (Ouikhalfan et al., 2022), representing 1-2% of world energy consumption via the Haber-Bosch process (Matassa et al., 2015). Phosphorus (P) fertiliser production and use also poses sustainability challenges, with synthetic P fertilizers mostly derived from non-renewable and diminishing phosphate rock (Cordell & White, 2011). Global phosphorus use is not efficient, with Brownlie et al. (2023) reporting that less than 50% of P is reused. Supply uncertainty of fertiliser trade is clearly a global risk. For example, due to the Ukraine conflict, fertiliser prices have peaked and put pressure on the global fertiliser market (Ben Hassen & El Bilali, 2022; Rabbi et al., 2023). Moreover, the largest sedimentary deposits of phosphate rock are found in northern Africa, China, the Middle East, and the United States, which may be perceived as a supply security issue by other nations (Mehta et al., 2015). Consequently, there has been an increasing global shift towards fertiliser nutrient stewardship, with some countries already having made P recovery mandatory (Brownlie et al. (2023)). The production and transport of synthetic fertilisers over extended distances (essentially an international market of trade) is also energy intensive and significantly adds to the global emissions footprint (Chai et al., 2019; Menegat et al., 2022; Ouikhalfan et al., 2022). This is especially relevant for the Australian agriculture sector, which relies heavily on global fertiliser imports (importing about 63% of Nitrogen, 33% of Phosphorus and 100% of potassium fertiliser) (FAO, 2024), and distributing these to regional areas over significant transport distances.

Dairy farming generates and captures large volumes of manure rich in nutrients and organic matter (Aarons et al., 2020; Birchall et al., 2008; Longhurst et al., 2000; Tait et al., 2021b). Manures have been proposed as an important future global source of P fertiliser (Cordell et al., 2009), and indeed has long been employed as an organic fertiliser on-farm (O'Brien & Hatfield, 2019). This has provided a renewable source of nutrients for soil amendment and effectively substituting synthetic fertilisers (O'Brien & Hatfield, 2019). However, if not properly managed, manure can pose similar increased environmental risks to synthetic fertilizers in terms of leaching, run-off, eutrophication, and GHG emissions (Edmeades, 2003; Rojas-Downing et al., 2017b). For example, typically, livestock systems employ extended storage of captured manure to enable controlled application of its nutrient content. This helps to prevent nutrient run-off and leaching losses, but effluent storage can also be a significant source of fugitive methane emissions (Kupper et al., 2020; Owen & Silver, 2015).

The characteristics (e.g. moisture content) of captured manure can vary within and across different farms (Aarons et al., 2023). For example, yard cleaning with large amounts of flood wash water generates a dilute manure effluent (TS<5%), whereas manure in other cases is chain scraped and collected as a slurry (5-15%) (Birchall et al., 2008). Regardless, a high moisture content translates into a dilute nutrient content, making transport over extended distances unviable. Thus, liquid effluent and slurry require advanced technological solutions to recover nutrients efficiently and transform them into forms that are both cost-effective and practical for transportation and beneficial re-use (Grell et al., 2023; Hjorth et al., 2010; Williams et al., 2020). This can then provide solid manure residues (TS>15%) that are stackable, and suitable for further processing, such as via composting (Birchall et al., 2008; Zhong et al., 2018), or anaerobic digestion for renewable biogas energy recovery (Abbasi et al., 2012; Batstone & Jensen, 2011; Hjorth et al., 2010; Hull-Cantillo et al., 2023; Tauseef et al., 2013; Zhang et al., 2022), or for transporting to areas where the nutrients are needed for crop growth.

Different farming production systems also influence the capture of manure. For example, the dairy industry in Australia continues to predominantly consist of grazing or pasture-based (PB) systems (Aarons et al., 2023; Aarons et al., 2020; Dairy Australia and Agriculture Victoria, 2023). In such systems, most of the manure is excreted on paddocks, and only about 9% of the excreted manure nutrients are captured and actively reused (Aarons et al. (2023)). PB systems rely on grazed natural forage, such as pasture, as the primary source of nutrition for the herd (Fontaneli et al., 2005; Rojas-Downing et al., 2017a). However, a significant trend is emerging towards more intensive production systems; such as partial mixed ration (PMR) systems where a significant portion (>50%) of feed is fed on a feed pad (Dairy Australia and Agriculture Victoria, 2023; Tait et al., 2021b; Timlin et al., 2021), or total mixed ration (TMR) systems where all of the milking herd's daily feed is fed in a shed (Dairy Australia and Agriculture Victoria, 2023; Williams et al., 2020). Intensive systems can be more efficient in terms of feed conversion and milk production (Fontaneli et al., 2005; Hofstetter et al., 2014) and can improve commercial performance and climate resilience (Dairy Australia and Agriculture Victoria, 2023; Tait et al., 2021b; Timlin et al., 2021). However, with intensive systems, cows are spending more time on impermeable surfaces (e.g. concrete) which captures a larger proportion of daily excreted manure on feedpads and/or housing floors, requiring careful management as a potential point source of nutrients and fugitive CH_4 emissions source (Grell et al., 2024; Williams et al., 2020). However, the additional manure capture might also represent a higher energy and nutrient recovery potential.

An in-depth comprehension of the components and resources contained in captured dairy manure streams across various production types is important to effectively recover valuable resources and predict if recovery strategies might be applicable across different production systems. This information needs to be gathered to assess the suitability of technologies and to capitalize on opportunities within the circular economy. According to these aspects, the main goal of this study was to analyse and compare the composition of dairy manure effluent/slurry on-farm originating from different production systems across Australia. The results are supposed to provide valuable insights into the options for manure management, the opportunities for nutrient resources and to facilitate the development of future strategies for their closed loop recovery on farms.

4.2. Methods

4.2.1. Sample collection & residue streams

Manure and effluent samples were sourced from the same sampling campaign as described by Grell et al. (2023). In total, samples were collected from 11 different dairy farms across four important dairy states in Australia: Queensland (QLD), Western Australia (WA), Victoria (VIC), and New South Wales (NSW). In total, 20 different dairy manure residue types were collected from a diverse range of production types (5 PB, 3 PMR, 3 TMR). With all three production types, manure effluent is typically produced by washing down and rinsing in the dairy or milking parlour and its associated holding yard. This effluent is predominantly in liquid form and can be diluted. With the PMR systems, a feedpad is also cleaned to remove residues, either as a wet slurry or effluent (cleaned daily) or dry (scraped weekly). With the TMR systems, most (or all) of the daily manure output of the cows is collected as an effluent or scrape from housing, in contrast to PB systems, where most of the daily manure output is instead deposited onto grazed pastures. These differences have important implications for nutrient capture and closed-loop options, as discussed further below. Three of the investigated farms had solid-liquid separation; specifically, Farms 1 and 11 used mechanical separation, and Farms 2 and 6 used passive separation with a weeping wall. At Farm 11, an inclined screen was used; however, the separated liquid fraction was drained via an inaccessible underground pipe so that only the inflow effluent prior to separation and the separated solids fraction could be sampled at this farm. Farm numbers in the tables and throughout the paper align with those reported by Grell et al. (2024). Detailed information on the farms and their manure collection and management systems is listed in Table 3, with terminology aligning with IPCC (2006) definitions.

Table 3 Detailed overview of investigated dairy farms and effluent samples

Farm	State	Milking herd	Manure management system	Sample location	Production system	Cleaning	Effluent volume ($kL \cdot d^{-1}$)
1	WA	1,400	Uncovered anaerobic lagoon and passive composting	Active Separator	PB	Flood wash	110
2	WA	300	Liquid/Slurry	Passive Separator	PB	Hose	25
3	WA	1,200	Daily spread	Dairy	PMR	Hose	70
				Feedpad			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	3.2
6	NSW	250	Uncovered anaerobic lagoon and passive composting	Passive Separator	PB	hose	15.5
				Dairy		Recycled flood	12
7	QLD	450	Uncovered anaerobic lagoon	Feedpad	PMR	Recycled flood	40
9	WA	350	Uncovered anaerobic lagoon	Dairy	PB	Hose	41
10	VIC	440	Uncovered anaerobic lagoon	Dairy	TMR	Recycled flood	60
			Liquid/Slurry	Barn	TMR	Wet-scraped	19
11	VIC	675	Uncovered anaerobic lagoon	Dairy	TMR	Flood	47
			Slurry storage	Barn	TMR	Wet-scraped	19
12	VIC	350	Uncovered anaerobic lagoon	Barn	TMR	Recycled flood	69
			Passive composting	Active Separator	TMR	Wet-scraped	n.a.

Mechanical separation (Grell et al., 2023)

4.2.2. Sampling procedure

The methodology used for sampling, previously described in detail by Grell et al. (2024) aimed at sourcing samples that were representative of the effluent at each farm, despite expected variability. In short, about 20 L each of dairy effluent (i.e., prior to any separation) was typically collected as a composite time-proportional grab sample from the dairy shed, feedpad, or housing complex during a washdown event (Grell et al., 2023). The liquid samples were then mixed to prevent the settling of solids during sub-sampling. Semi-solid manure samples were collected via scraping directly off barn or feedpad floors, as well as by the methods previously described in detail by Grell et al. (2023). After sampling, all sample containers were promptly sealed, placed on ice, and transported cold to the laboratory for analysis. At the laboratory, samples were stored at 4°C and analysed without delay for electrical conductivity (EC), and elemental concentrations of aluminium (Al), calcium (Ca), copper (Cu), iron (Fe), potassium (K), magnesium (Mg), sodium (Na), phosphorus (P), sulphur (S), zinc (Zn), and total nitrogen (TN) (Section 4.2.3).

4.2.3. Analytical procedures

Standard methods were used for the determination of pH and electrical conductivity (EC) (APHA, 1995). Total Nitrogen (TN) was analysed using the Kjeldahl standard method (APHA, 1995), with the resulting ammonia measured on a Lachat flow injection analyser as per the Lachat QuickChem Method 31-107-06-1-A. Samples were diluted 1:6 before digestion with potassium persulfate and 1:20 after dilution with milli-Q water to bring the sample into measurement range.

Total elements, including P, Na, Al, Ca, Cu, Fe, K, Mg, S, and Zn, were measured by inductively coupled plasma optical emission spectroscopy (ICP-OES) using a Perkin Elmer Optima 5300DV (Perkin Elmer Corp., Norwalk Ct, USA). For ICP-OES measurements on liquid samples (influent or filtrate), 4 mL of liquid sample were pre-digested with 2 mL of nitric acid and 0.5 mL of H_2O_2 before being made up to 20 mL for analysis. For ICP-OES measurements of the solid fraction, the samples were dried at 70ºC and finely ground. 0.15 g dried material was then accurately weighed and pre-digested with nitric acid and perchloric acid before being made into a 10 mL solution for analysis.

Total carbon (C) and nitrogen (N) in a pre-dried (70ºC) solid fraction were measured by an Elementar Vario Macrocombustion Analyzer (Hanau) according to the Dumas method (Etheridge et al., 1998).

4.2.4. Data analysis and statistical methods

Each analyte was measured in triplicate, with the results given below reporting mean values with standard deviations to quantify variability in analytical replicates. All the statistical evaluation was carried out in R statistical software (version 4.2). For the statistical analysis, farms were categorised based on production type (i.e., PB, PMR, TMR). Any outliers for measured analytes were identified using the interquartile range method. A one-way analysis of variance (ANOVA) was performed to evaluate the influence of production type on measured concentrations. Additionally, the same statistical approach was used to determine total manure residue capture rates in $g \cdot (d \cdot cow)^{-1}$, calculated via effluent volume. TS data for further analysis in the current work were sourced from Grell et al. (2024), and wherever this was done, it was clearly cited to the original source. The analysis employed Type III sums of squares to address any differences in sample sizes across the different production type categories. Prior to the ANOVA tests, the assumptions of homogeneity of variances and normality of residuals needed a log transformation. The Levene's test therefore was executed to evaluate the equality of variances among each production type category. Meanwhile the normality of the residuals of the ANOVA model was assessed using the Shapiro-Wilk test. In order to determine significant differences both across the production groups and in terms of effluent characteristics, post-hoc pairwise comparisons were performed using Tukey's Honest Significant Difference (HSD) test for all parameters.

To investigate a potential correlation between analytes, 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.

In order to evaluate the proportion of the daily manure nutrient output captured by each production system type by expected N, P, and K in manure residue samples was estimated and compared to theoretical estimates by the method of Nennich et al. (2005). This was done by multiplying measured nutrient concentrations by the estimated daily volume of manure residues and effluent produced by each farm and then comparing this result to a theoretical nutrient production (Commonwealth of Australia, 2018). Assumptions for the theoretical estimate included an average milk yield of 16.5, 22.5 and 30 kg per cow per day for PB, PMR, and TMR, respectively (Dairy Australia and Agriculture Victoria, 2023; Nennich et al., 2005). Based on these parameters, the theoretical daily manure (TS) and nutrient (N, P, K) output was calculated per cow. In combination with an estimated averaged time on the yard of 3 hours per day for PB systems, the proportions of captured manure were compared with the calculated values by Nennich et al. (2005). It was important to cross-validate the data obtained from commercial facilities during operations.

4.3. Results

4.3.1. Statistical description of dairy manure residues

The datasets in Table 4 display the chemical characteristics of manure residues collected across the various farming systems (i.e., PB, PMR, and TMR). Whereas figure 4 summarizes the statistics for grouped effluent measurements across the three different production system types (PB, PMR, and TMR) and the correspondent PB filtrates of the solid liquid separation (individual concentration in Table 5). Estimates of error indicated in all tables and figures are standard deviations in population or group replicates.

The measured EC values range from 2.4 $\mu S \cdot cm^{-1}$ to 12.46 $\mu S \cdot cm^{-1}$. This variation suggests differences in the salinity of the effluents, which could impact their suitability for irrigation because of the risk of salt accumulation in soils. In terms of macronutrients, TN values showed fluctuations ranging from 221.4 $mg \cdot L^{-1}$ to 973.7 $mg \cdot L^{-1}$, therefore highlighting the distinct nitrogen loading capacities and dilution extents present in these systems. This is important for recovery, as further discussed below (Section 4.4). The P concentrations in the effluent vary between 51.1 and 228.7 $mg \cdot L^{-1}$, indicating opportunities for P recovery strategies, as demonstrated by Grell et al. (2023) in Chapter 5. K concentrations range from 254.3 to 978.2 $mg \cdot L^{-1}$, demonstrating their potential as a rich source of this essential macro nutrient. The dissolved state of K makes it easily accessible to plants but also require careful handling to ensure optimal usage and prevent nutrient overloading.

Table 4 Chemical characteristics of dairy residue streams

Farm	Residue stream	System	EC [μS·cm^{-1}]	TN [mg·L^{-1}]	P [mg·L^{-1}]	K [mg·L^{-1}]	Al [mg·L^{-1}]	Ca [mg·L^{-1}]	Cu [mg·L^{-1}]	Fe [mg·L^{-1}]	Mg [mg·L^{-1}]	Na [mg·L^{-1}]	S [mg·L^{-1}]	Zn [mg·L^{-1}]
1	Dairy effluent	PB	-	221.4 (±11)	61.5 (±9.4)	153.8 (±18.7)	4.8 (±1.4)	97.9 (±14.4)	0.2(±0.1)	10.9 (±2.6)	76.2 (±11.2)	136.2 (±22.3)	25.3 (±4.5)	1(±0.2)
2	Dairy effluent	PB	4.35 (±0.03)	590.5 (±43.4)	104.4 (±0.7)	382.6(±1.5)	77.7 (±4.2)	478.9 (±18)	0.5 (±0)	92.6 (±6)	178.1 (±2.4)	452 (±1.7)	78.1 (±1.6)	2.7(±0)
3	Dairy effluent	PMR	3.9 (±0.02)	716.9 (±11)	142 (±10.2)	511 (±16.2)	37.9 (±5.1)	367.5 (±23.2)	0.9 (±0.1)	60.4 (±10.7)	232.9 (±12.1)	101.7 (±3.9)	95.9 (±8)	3.8 (±0.3)
	Feedpad effluent	PMR	4.22 (±0.02)	809 (±53.7)	158.3 (±6.7)	662.8(±20.7)	63.9 (±6.9)	454.6 (±26.6)	0.9 (±0.1)	94.8 (±8.1)	274.1 (±8.9)	131.9 (±3.8)	106.4 (±6.2)	3.7 (±0.3)
4	Dairy effluent	PB	2.61 (±0.02)	500.5(±133.6)	154.5 (±14.6)	539.2 (±20.1)	741.1 (±99.9)	367.9 (±27.2)	1(±0.1)	1018.8 (±112.7)	330.1 (±26.7)	266.5(±0.6)	72.6 (±8.6)	3.8 (±0.4)
5	Dairy effluent	PMR	2.98 (±0.02)	677.1 (±59.4)	228.7 (±3.8)	298.1 (±2.7)	56 (±3.1)	479.2 (±15.7)	1.2 (±0.1)	81.2 (±4)	173.1 (±3.5)	276.1 (±2.8)	104.7 (±5.5)	5.2 (±0.8)
	Feedpad solids	PMR	4.78 (±0.03)	113,351 (±2735)	1789.9 (±82.8)	3252 (±130)	794.7 (±24.2)	3934.1 (±80)	9.5 (±0.5)	1397.1 (±60.8)	1484.6 (±68.8)	1047.8 (±48.3)	1082.6 (±63.7)	42.2 (±1.8)
6	Dairy effluent	PB	3.33 (±0.01)	417 (±97.5)	102.9 (±14)	525.6 (±41.7)	58.2 (±13.3)	291.6 (±26.2)	0.4 (±0.1)	81.4 (±16.2)	151.4 (±5.3)	241.8 (±22.2)	64.7 (±5.1)	2(±0.4)
	recycled effluent	PB	3.86 (±0.03)	400.6(±62.8)	147.9 (±58.2)	581.3 (±16.4)	65 (±25.4)	342.1 (±109)	0.5 (±0.2)	101.1 (±38.9)	189.5 (±46.1)	210.7 (±1.8)	59.5 (±15.5)	3.1 (±0.9)
7	Dairy effluent	PMR	2.4 (±0.01)	308.4 (±26.4)	80.4 (±5.6)	254.3 (±4.4)	30 (±4)	150.2 (±9)	0.3(±0)	46.8 (±5.5)	111.6 (±5.3)	109.9 (±1.9)	39.3 (±1.9)	2(±2.3)
9	Dairy effluent	PB	4.52 (±0.01)	768.8 (±57.9)	143.2 (±9.9)	553.6 (±30.5)	132.1 (±10.2)	441.2 (±29.2)	1(±0.1)	47.7 (±5.2)	219.6 (±13.4)	145.3 (±8)	96.2 (±7.8)	5.5 (±0.8)
10	Dairy effluent	TMR	5.18 (±0.01)	523.1 (±16.2)	88.2 (±3.9)	537.8(±3.1)	46.8 (±6.8)	204.4 (±12)	0.5 (±0)	66.1 (±10.2)	134.7 (±4.6)	368.6 (±2.9)	41.5 (±3)	1.5 (±0.1)
	Barn slurry	TMR	12.46 (±0.01)	383.3 (±50.5)	51.1 (±2.9)	289.5 (±4.9)	29.3 (±5.4)	117 (±15.7)	0.4 (±0.1)	37 (±8.4)	72.1 (±3.8)	163.2 (±2.5)	36.3 (±4.7)	1.2 (±0.2)

Farm	Residue stream	System	EC [µS·cm^{-1}]	TN [mg·L^{-1}]	P [mg·L^{-1}]	K [mg·L^{-1}]	Al [mg·L^{-1}]	Ca [mg·L^{-1}]	Cu [mg·L^{-1}]	Fe [mg·L^{-1}]	Mg [mg·L^{-1}]	Na [mg·L^{-1}]	S [mg·L^{-1}]	Zn [mg·L^{-1}]
11	Dairy effluent	TMR	4.27 (±0.01)	655.6 (±42.6)	134.9 (±19.4)	610.6 (±26.2)	306.4 (±61.1)	302.4 (±51.2)	1 (±0.2)	149.6 (±34.3)	174.5 (±19.4)	183.1 (±3)	92.3 (±16.6)	4.4 (±1.2)
	Barn slurry	TMR	6.72 (±0.01)	535.6 (±36)	116.9 (±6.5)	343.8 (±12.3)	161.2 (±11.4)	228 (±19.4)	1 (±0.1)	112.6 (±10.2)	130.9 (±7.5)	100.9 (±3.1)	74.2 (±7.4)	2.1 (±1.8)
12	Dairy effluent	TMR	6.04 (±0.02)	973.7 (±53.2)	187.1 (±4.2)	978.2 (±20.9)	115.4 (±1)	373.6 (±6)	1.3 (±0)	105.2 (±0.9)	245.5 (±3.1)	419.1 (±8.7)	111 (±2.4)	4.2 (±0.1)

Table 5 Chemical characteristics of resulting filtrates from separated dairy effluent

Farm	Residue stream	System	EC [µS·cm^{-1}]	TN [mg·L^{-1}]	P [mg·L^{-1}]	K [mg·L^{-1}]	Al [mg·L^{-1}]	Ca [mg·L^{-1}]	Cu [mg·L^{-1}]	Fe [mg·L^{-1}]	Mg [mg·L^{-1}]	Na [mg·L^{-1}]	S [mg·L^{-1}]	Zn [mg·L^{-1}]
1	Filtrate (Z-Filter)	PB	-	227.2 (±17.9)	60.3 (±9)	152.6 (±19.1)	4.5 (±1.2)	93.1 (±12.2)	0.2 (±0)	10.1 (±2.4)	74.9 (±10.5)	136 (±22.3)	24.2 (±4.4)	0.9 (±0.2)
2	Filtrate (Weeping wall)	PB	3.34 (±0.02)	223 (±37.6)	54.4 (±6.8)	192.1 (±12.2)	20.6 (±3.6)	210.2 (±31.3)	0.2 (±0)	27.8 (±4)	88.2 (±8.6)	312.2 (±19.4)	35.6 (±4.4)	1.2 (±0.2)
6	Filtrate (Weeping wall)	PB	5.09 (±0.01)	407.6 (±24.4)	69.1 (±5.1)	800.3 (±15.9)	18.3 (±2.0)	183 (±15.4)	0.2 (±0)	29.1 (±3)	121.8 (±5.4)	240.5 (±5.1)	58.4 (±1.6)	0.9 (±0.1)

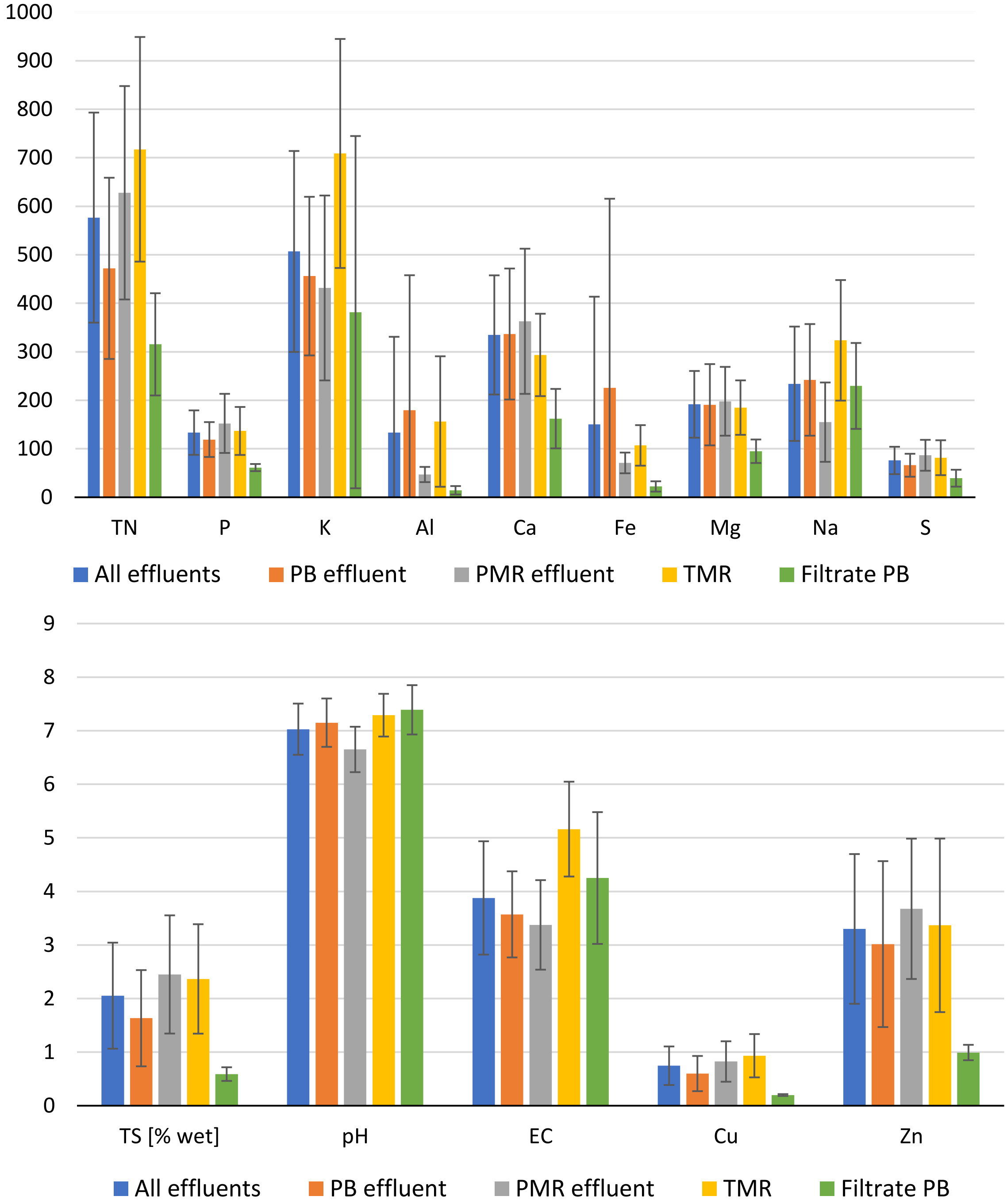

Figure 4 Measured effluent characteristics grouped by production system, including data for Filtrate from solid-liquid separation treatment of effluent. The Units of measure for macro and micro nutrients are $mg \cdot L^{-1}$, and for EC is $\mu S \cdot cm^{-1}$

Among the three production types (PB, PMR, and TMR) no statistically significant differences could be identified for the measured concentrations of liquid/slurry manure residues at a 5% significance threshold. This occurred despite different forage intakes, with cows in PMR and TMR systems receiving a formulated ration that is generally different from grazed pastures. However, P-values showed different levels of significance, which required further investigation. The results indicate that the p-values obtained are high when comparing PMR and TMR, indicating a lack of significant differences. Nonetheless, a notable disparity in p-values arises when evaluating the statistical significance of PMR or TMR in relation to PB. This may be traced back to contrasting dietary systems between PB and TMR, with PMR being between those.

While the nutrient concentrations of effluent streams only provide indications, it is crucial to understand the total nutrient capture rate on a per-cow basis. Thereby the quantity of the discharged residues is taken into consideration and a more inclusive perspective on the management of nutrients within each system can be provided. The evaluation of total capture rates allows for the assessment of the exact nutrient yield from each animal. It enables the identification of potential discrepancies that may not be noted when only examining concentrations.

Table 6 presents a comparative analysis of nutrient capture rates by presenting mean values and corresponding standard deviations for TS, TN, P, and K—across the three production systems. Statistical significance between systems by pairwise comparisons and ANOVA tests, with corresponding p-values, is provided in Table 4.

The statistical analysis of the deviation on N across the different production systems resulted in a p-value of 0.185. Thus, the outcome demonstrates neither a statistically significant difference in the means among the various production systems. Following the initial analysis, subsequent post-hoc Tukey's HSD showed a significant difference between the PB and TMR systems ($p=0.044$). However, no major differences were observed between the PMR and TMR systems ($p=0.64$).

The variations between the PB and PMR systems (p=0.11) were just not distinctive at the 0.1 significance level.

The ANOVA test for P showed a F-statistic of 3.06, and a p-value of 0.133. These results express that there is no significant difference in the means of P across the individual production systems. However, the post-hoc tests revealed a significant difference between the PB and PMR systems (p=0.039), suggesting a notable distinction in phosphorus capture rates between these systems. The comparisons between TMR and PMR (p=0.497), and PB and TMR (p=0.096), did not show statistically significant differences.

Ultimately, the variance analysis conducted for K yielded an F-statistic of 3.89, accompanied by a p-value of 0.08. Although the p- value indicates no level of significance, subsequent post-hoc analysis revealed a statistically weak difference between the PB and TMR systems (p= p=0.059). Nevertheless, the PB and PMR systems (p=0.542) as well as the PMR and TMR systems (p=0.169) did not show significant statistical variations in the average K levels.

Table 6 Total capture rates and p values of comparison (TS values adopted from Grell et al. (2024))

System	Total solids [$kg \cdot d \cdot cow^{-1}$]	Nitrogen [$g \cdot d \cdot cow^{-1}$]	Phosphorous [$g \cdot d \cdot cow^{-1}$]	Potassium [$g \cdot d \cdot cow^{-1}$]
PB	1.45(±0.44)	35.8(±13.8)	8.6(±3.3)	32.7(±18.0)
PMR	2.50(±0.80)	138.0(±50.1)	13.3(±4.2)	42.0(±27.7)
TMR	6.05(±0.14)	115.7(±67.4)	21.7(±13.2)	112.4(±71.92)
Pairwise comparison		P-values		
PB vs PMR	0.163	0.110	0.039	0.542
PB vs TMR	0.0001	0.044	0.096	0.059
PMR vs TMR	0.003	0.640	0.497	0.209
ANOVA	0.0001	0.185	0.133	0.080

4.3.2. Correlations in Dairy Effluent Component Concentrations

The analysis of statistical correlations within only liquid dairy effluent (excluding slurry, to avoid confounding effects of moisture content) showed numerous interconnections that may be crucial for understanding of resource recovery opportunities in a closed-loop system. The correlations and their level of significance are presented in a heatmap (Figure 5). All Pearson correlations subsequently described in this section were found to be significant at the 5% significance threshold. Positive correlations were observed between TS data from Grell et al. 2014 and many elements, including Cu (r=0.90), Mg (r=0.82), S (r=0.88), P (r=0.77), TN (r=0.78), and Zn (r=0.76). These observed correlations suggest that these elements are associated with particulate matter found in the effluent. Furthermore, there was a modest positive correlation between TS and Ca (r=0.70) and a much weaker positive correlation between TS and K (r=0.57), indicating that Ca is partly associated with particulate matter, and K is generally found in mobile or soluble forms (Syrchina et al., 2022). This is also suggested by an observed positive correlation between EC and K (r=0.77).

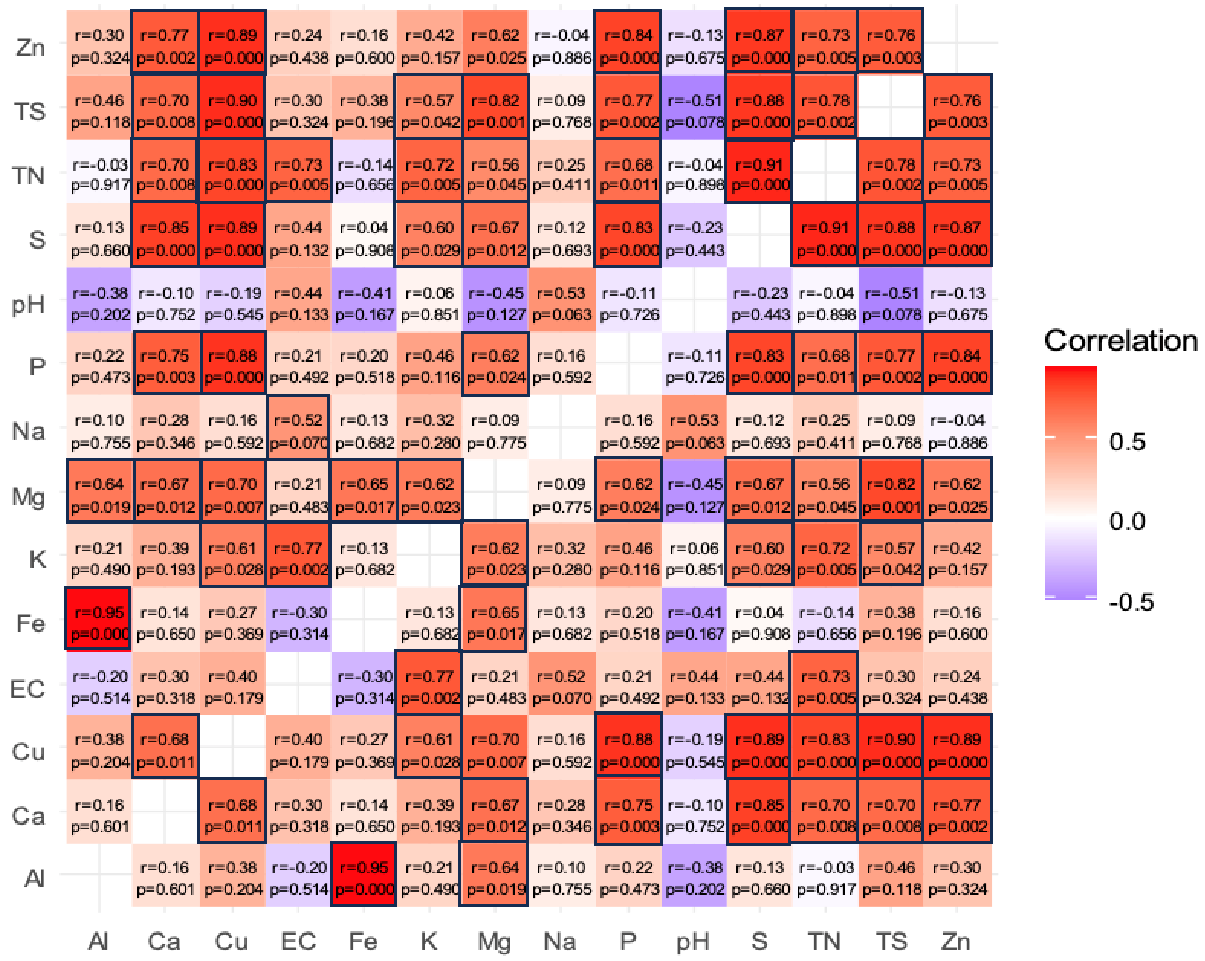

Figure 5 Correlation heatmap of nutrients in liquid/slurry manure residues, including significance values

EC is a measure of effluent's capacity for carrying electrical current and is directly related to the concentrations of ionized substances in the water. EC showed notable variations among the different samples, ranging from 2.23 to 6.04 $\mu S \cdot cm^{-1}$ suggesting differences in the ionic activity. A modest correlation was observed between EC and TN (r=0.73), indicating its presence in the soluble phase. Unfortunately, nitrogen species could not be measured for the current work to confirm this.

Some elemental concentrations fluctuated significantly, particularly in the case of Al, which ranged from 4.8 $mg \cdot L^{-1}$ to 741.1 $mg \cdot L^{-1}$, and Ca, which ranged from 97.9 $mg \cdot L^{-1}$ to 978.2 $mg \cdot L^{-1}$. These changes highlight the presence of diverse chemical compositions within the captured manure streams.

The elements Al and Fe had a significant association (r=0.95), suggesting a possible shared geological source, such as the water used for cleaning or soil particles present in the effluent. Ca had significant positive associations with S (r=0.847) and Zn (r=0.77), as well as moderate positive correlations with Mg (r=0.67), Cu (r=0.677), TS (r=0.70), TN (r=0.70), and P (r=0.75). Mg exhibited a robust link with TS (r=0.82) and moderate relationships with components such as Fe (r=0.65), S (r=0.67), P (r=0.62), Zn (r=0.62), and TN (r=0.56). Ca and Mg both contribute to water hardness, and their associations with P and TS, and Mg with N, could indicate the formation of mineralized structures or precipitates, such as struvite, calcium phosphate, or magnesium phosphate. This, aligned with an observed negative correlation (r = -0.51) between pH and TS, as the solubility of relevant mineral precipitates generally tend to increase with decreasing pH. The implications of these observations on resource recovery are further discussed below (Section 4.4).

4.4. Discussion

4.4.1. Dairy manure residues as a fertiliser source and soil amendment

When facing the global dependency on synthetic fertilizer and the consequential supply pressures, the need to explore the beneficial reuse of renewable alternatives becomes apparent (Rosa & Gabrielli, 2023). Livestock wastes, such as dairy manure residues, can potentially serve as alternative nutrient sources for soil (O'Brien & Hatfield, 2019). Even though nutrient concentrations of liquid effluent were not significantly influenced by the production type (Section 4.3.1), the residue types tend to vary somewhat across the production systems. Specifically, where manure is captured as a scrape or as a solid mixed with bedding, this would potentially aide handling and reuse. The sample size in the current study was small but still aspirational for full-scale sampling of commercial facilities. Regardless, a broader data set may have revealed significant differences in composition between different production systems. For example, nutritional differences between grazing in PB cattle and mixed rations in PMR and TMR would be expected to translate into compositional differences in captured manure.

Nonetheless, nutrient capture rate on a per-cow basis is also important for nutrient resource recovery and nutrient management. Specifically, TMR systems are able to capture more nutrients (>80%) per cow than PB systems (Section 4.3.1). PMR systems reflect an intermediate between PB and TMR, which explains why differences PMR and either of these production systems were not significant (Section 4.3.1). The higher capture rate in TMR systems can have a positive effect on nutrient recycling but also increases the localised point source of nutrients to manage. Thereby environmental concerns like nutrient imbalances in soil and water, and risk of run-off and leaching with associated pollution, also increases. In contrast, the spatial distribution of excreted nutrients in grazing systems is predominantly dictated by the deposition of grazing animals. However, the pattern of nutrient deposition can still often be heterogeneous in PB systems, with localized zones of elevated nutrient concentrations near the milking shed. The risk of nutrient loss and environmental contamination is altered by this distribution and livestock stocking rates (FAO, 2018).

Both extensive (PB) and intensive (PMR and TMR) systems usually require additional external nutrient inputs, which increases their environmental footprint and reflects their reliance on synthetic fertilizers. However, the higher nutrient capture rates in TMR systems offer an increased opportunity to reduce reliance on external nutrient inputs via efficient nutrient recycling and controlled application to grow pasture and feed. The formulation of nutrients in mobile and particulate forms is one of the critical challenges with manure residues. While mobile fractions, such as ammonium, nitrate, and potassium are easily taken up by plants, they are also susceptible to environmental losses. Particulate forms, on the other hand, may be less plant-available but also less likely to be lost via leaching, run-off and volatilisation and contribute to environmental degradation. The distribution of nutrients between particulate and fine/mobile fractions are influence by recovery approaches (Section 4.4.2).

Nutrient ratios (N:P:K) in the capture manure streams (e.g. effluent, slurry, etc.) are also important to match specific crop nutrient demands. This is an ongoing challenge with utilising dairy effluent efficiently, as the nutrient ratios do not generally match crop demands very well (Longhurst, 2017). Improper management and application can lead to nutrient imbalances in soils and eutrophication. Furthermore, nitrogen in the effluent can contribute to nitrous oxide emissions when applied to soil; albeit that this is also applicable for synthetic fertiliser, and the slow-release properties of organic N in manures supports a more gradual supply of N as compared to synthetic fertiliser (Amon et al., 2006; Chai et al., 2019; Saggar et al., 2004; Shakoor et al., 2021). Recovery strategies may also target altering of the nutrient ratios to fully capitalise on the potential benefits of dairy effluent in agriculture while reducing its environmental risks (Section 4.4.2).

4.4.2. Closed-loop Recovery Technologies and Options

As highlighted above, the dilute nature of captured dairy manure as effluent or slurry hinders their efficient and cost-effective reuse (Tait et al., 2021). Furthermore, the predominance of soluble or mobile macro-nutrients, most notably K (Section 4.3.2), but also N and P, can also hinder the ability to separate these nutrients into a more concentrated form for beneficial reuse (Meyer et al., 2007; Moller & Muller, 2012). Soluble or mobile elements are frequently problematic to recover using traditional methods, such as separation or sedimentation without the aid of chemicals (Grell et al., 2023; Meyer et al., 2007).

However, statistical correlations in the current work also demonstrated association between Ca and P which could imply together with correlations with TS that calcium phosphate ($Ca_3(PO_4)_2$) minerals (Equation 1) are present and are indicative of chemical precipitation(Carlsson et al., 1997).

$$Ca^{2+} \quad + \quad PO_4^{3-} \quad \rightarrow \quad Ca_3(PO_4)_2 \qquad (1)$$

Grell et al. (2023) demonstrated an important influence of mineral precipitation as a mechanism to convert P in dilute dairy effluent into particulates which can be filtered out and thus separated. In that study, it was possible to increase the P removal efficiency up to 90% by combining dilute dairy effluent with hydrated lime and cationic polymer flocculant. Similarly, a significant association was seen between K and Mg (Section 3.2.2), and it is noted that other minerals such as struvite could form ($MgNH_4PO_4\ 6H_2O$) (Equation 2), albeit that the dissolved concentrations of Mg, NH_4^+ and PO_4^{-3} in the effluent or slurry all need to exceed solubility limits for this mineral to form.

$$Mg^{2+} \; + \quad NH_4^+ \; + \quad PO_4^{3-} \rightarrow \quad MgNH_4PO_4\ 6H_2O \qquad (2)$$

As nitrogen and phosphorus species could not be measured in the current study, struvite formation could not be substantiated.

Chemical precipitates such as struvite and calcium phosphate can act as slow-release fertilisers, to moderate the release of nutrients and thus aide environmental protection.

Mineral nutrient forms can be separated together with organic N and P into concentrated solid manure products using solid-liquid separation. Not only may the nutritional value of the recovered product be improved by connecting methods such as solid-liquid separation with precipitation and flocculation to up-concentrate the nutrients, but transportation can then be more cost-effective and its application in agricultural settings easier to implement (Cucarella et al., 2007; Rugaika et al., 2019). Moreover, complementary methods, like the incorporation of biochar, have the potential to further mitigate N losses and nitrous oxide emissions (Shakoor et al., 2021), and also mitigate phosphorus leaching risks (Laird et al., 2010).

4.4.3. Resource recovery potential for different production system types

Given the challenges and possibilities that have been examined, there is an enormous potential for the combination of multiple manure management technologies in order to improve the nutrient recovery from dairy effluent. In accordance, the nutrient excretion rates from estimates of the method by Nennich et al. (2005) (Chapter 3) with an average milking yield is 16,5L per head indicate that the Australian dairy herd of 11,5 million cattle is excreting daily an estimated 4,514 tonnes of N, 728 tonnes of P and 2,052 tonnes of K, containing a daily value of appr. 18 billion AUSD. Currently, 9% of the available nutrients are reused in grazing systems represented in PB and PMR (Aarons et al., 2023). While in PB systems, only 9 % N, 14% P and 18% K (Table 6), can be captured in form of liquid effluent, PMR systems offer the ability, through additional feeding on concrete yards, to extend capture rates in liquid effluent to 29% N, 34% P and 24% K (Table 6). Considering a TMR production system, the capture rate of nutrient in liquid/slurry effluent can be extended to 29% N, 34% P and 63% K (Table 6). It is important to mention, that the remaining nutrients in TMR systems are predominantly contained in solid materials, such as bedding. Thus, TMR systems offers great opportunity for holistic and controlled recycling approach. The daily estimated capture rates are demonstrated in Figure 6.

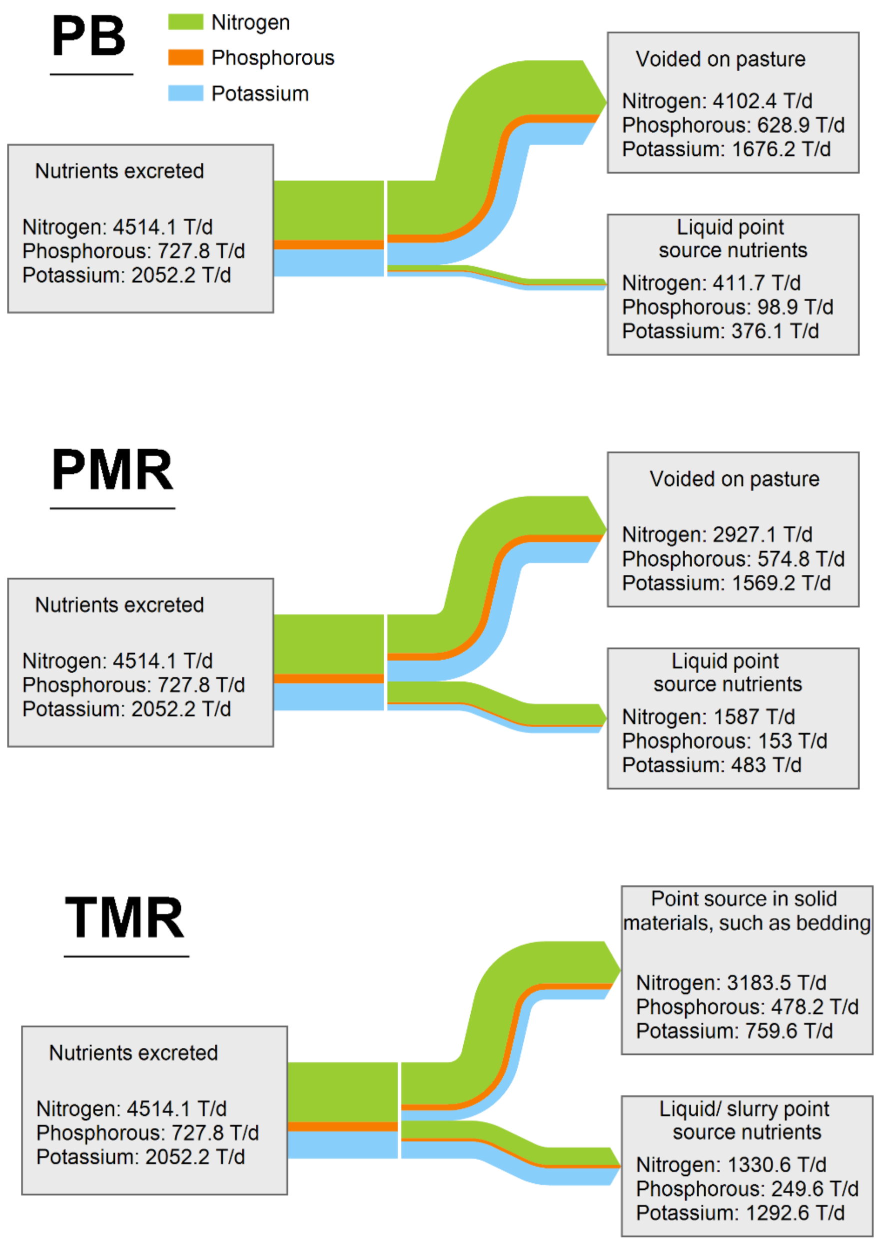

Figure 6 Nutrient source and possible sinks in different production systems (PB, PMR or TMR). The amount of nutrients excreted represents the total available across the entire Australian dairy industry, not just from TMR, PMR and PB. Note that the make-up of the industry in the future will likely be a mix of PB, PMR and TMR.

Policy levers can also play a vital role to reward nutrient recovery strategies. This might be achieved by the provision of positive economic incentives (e.g. green credits, or price premiums), or via regulation, to drive more sustainable practices, and facilitate the uptake of technological advances in nutrient recovery and beneficial reuse. This will also be important in Australia, as the dairy industry will likely transition from traditional grazing-dominant practices to more intensive systems, which as Figure 6 highlights will increase nutrient capture, and opportunities for beneficial reuse. Other countries with an intensified dairy industry, such as in Europe or the United States, might serve as an example for a successful policy for the successful implementation of circular technologies across countries like Australia traditionally dominated by pasture-based systems but progressively transitioning towards intensive production.

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CHAPTER 5: PAPER 2 – RESOURCE RECOVERY FOR ENVIRONMENTAL MANAGEMENT OF DILUTE LIVESTOCK MANURE USING A SOLID-LIQUID SEPARATION APPROACH.

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Expanding upon the outcomes of Chapter 4, which indicated no significant differences in nutrient levels among various dairy production systems but did highlight significant correlations between elements, this publication progresses logically from these findings. The investigated capacity of Phosphorous to form precipitation reactions, such as calcium phosphate or struvite were used to increase the P removal efficiency. These outcomes present interesting opportunities for the implementation of sustainable strategies in the management of manure, by providing flexibility for nutrient control on dairy farms.

Drawing upon these valuable insights, the present study aims to examine the effectiveness of mechanical solid-liquid separation as a closed-loop technique for the recovery and reuse of nutrients, carbon, and water from highly diluted dairy shed effluent (TS content of 0.52%). The study examines the implementation of a modular separation technology called Z-Filter in a PB system in WA. The study specifically investigates the effluent produced, which has flow rates ranging from 200 to 400 L min^{-1}. This research is the first comprehensive application of Z-Filter on dairy effluent at a full scale.

This study investigates the efficiency of separation and the composition of the solid fraction that is separated. Additionally, it explores the effects of chemically assisted separation using a cationic polymer flocculant, both with and without the presence of hydrated lime.

The primary goal is to enhance the efficiency of extracting vital components such as TKN, P, and VS from dilute manure effluents. The focus is to provide flexibility in the transportation and possibilities for additional processing of the solid fraction, together with the aim of minimising fugitive methane emissions from uncovered anaerobic effluent ponds.

By utilising the empirical data obtained from Chapter 4, this publication aims to offer dairy farmers improved recovery options for environmental management of diluted dairy effluent. The primary objective was to develop practical approaches that facilitate the effective utilisation of organic matter and nutrients, thus reducing environmental risks associated with dairy effluent.

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Research article

Resource recovery for environmental management of dilute livestock manure using a solid-liquid separation approach

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ABSTRACT

Mechanical solid-liquid separation is an emerging closed-loop technology to recover and recycle carbon, nutrients and water from dilute livestock manure. This closed-loop concept is tested using a modular separation technology (Z-Filter) applied at full-scale for the first time to treat effluent from a pasture-based dairy. Effluent flow rates were 200–400 L min^{-1} at a total solids (TS) content of 0.52% (pH 7.2). Separation efficiency and composition of the separated solid fraction were determined, and chemically-assisted separation with cationic polymer flocculant with/without hydrated lime was also tested. Without flocculant and lime, 25.9% of TS and 33.4% of volatile solids (VS) ended up in the solid fraction, but total Kjeldahl nitrogen (TKN), phosphorus (P) and potassium recovery was not significant, likely being in poorly separable fine particle or soluble fractions. With a 5% flow-based dosage of flocculant, most of the TS (69%) and VS (85%), and notable amounts of TKN (52–56%) and P (40%) ended up in the solid fraction. Phosphorus recovery was further increased to 91% when both flocculant and hydrated lime was added up to pH 9.2. The solid fraction was stackable with 16–20% TS, making transport more economical to enable further processing and beneficial reuse of nutrients and organic matter. Removal of VS also reduces fugitive methane emissions from uncovered anaerobic effluent ponds. Overall, the results indicated that solid-liquid separation could provide improved environmental management options for dairy farmers with dilute manure effluent to beneficially utilise organic matter and nutrients.

1. Introduction

It is becoming increasingly difficult to feed the global population whilst protecting natural environments that support global food production (Mueller et al., 2012). It will require more sustainable food systems (Odegard and van der Voet, 2014), including technology platforms to recover and recycle resources from waste (Mehta et al., 2015). This is especially important if animal product consumption increases, because animal product diets can require more resources than vegetarian diets (Odegard and van der Voet, 2014). However, waste from livestock food production, such as animal manures, can also be an increasingly important future source of non-renewable phosphorus (P) (Cordell et al., 2011) and a potential source of soil carbon (Abbott et al., 2018). Unfortunately, there is a lack of viable closed-loop technology options to enable recovery and recycling of nutrients and organic matter from dilute animal manure. Such animal manure is typically generated as an effluent at dairies with flood wash systems and piggeries with flushing systems, as are common in the Americas and Asia-Pacific region. Furthermore, with pasture-based dairy systems in Australia and New Zealand, cows spend most of their day in outdoor paddocks with only a portion of the daily manure output captured, resulting in an even more dilute effluent (Tait et al., 2021). Animal-derived waste (e.g. manure and effluent) is commonly used as a fertilizer because of its nitrogen (N), P, and potassium (K) contents, but is bulky due to its carbon and moisture content (Fyfe et al., 2016; Mehta et al., 2015). This entails low nutrient concentrations and a low value, or even a negative value, when transport costs are included (Mehta et al., 2015). Consequently, dairy effluent is often not recycled and are instead frequently stored in uncovered anaerobic ponds causing fugitive methane emissions, before being land-applied over small areas close to their source that increases the risk of detrimental nutrient run-off into surface waters or leaching into groundwater. A separation technology approach is needed that can be integrated with common farm infrastructure. This would provide farmers with options to beneficially reuse organic matter

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and nutrients in heterogeneous manure streams. The ability to control waste stream nutrient and organic matter concentrations and to produce transportable separated manures are desired features of a sustainable recycling approach. Solid-liquid separation could become increasingly important as a first processing step to up-concentrate dilute manures (Hjorth et al., 2010) to enable more economical beneficial reuse, or to prepare separated manure feedstocks for more efficient and cost-effective transport and processing into value-added products. Solid-liquid separation produces a concentrated solid fraction and a clarified liquid fraction. Anticipated benefits potentially extend to manure storage and applying manure to land. Specifically, the removal of organic matter could reduce fugitive methane emissions from subsequent storage of manure effluent (Amon et al., 2006). The removal of nutrients reduces the risk of nutrient run-off and leaching when effluent is stored, and/or land-applied because nutrient application can better match crop nutrient demands.

Research on solid-liquid separation of livestock manure slurry/effluent is global and a range of separation technologies are commercially available and widely applied (Burton, 2007; Hjorth et al., 2010). Current commercial technologies are capable of handling high flow volumes (e.g. inclined screen), or can achieve a high extent of dewatering of the solid fraction (e.g. screw press); but generally do not have a high spatial loading combined with good dewatering capabilities. This makes mechanical separation less economically attractive, especially for small to medium scale applications. There is need for a compact modular mechanical separation approach to resolve spatial loading and dewatering issues and provide options for farmers to improve on-farm environmental management. One possible technology option considered in the current work is the Z-Filter, a separation technology developed in Western Australia (WA) in 2012. The Z-Filter uses a fabric filter element like a filter press, folded and sealed into a sock-like tube to make the unit compact. The Z-Filter has been previously applied and tested at full-scale at a piggery and with use of a commercial coagulant and flocculant achieved 73%, 35% and 65% recovery of volatile solids (VS), total Kjeldahl nitrogen (TKN) and P into the solid fraction, respectively (Payne, 2014). The unit also produced a stackable solid fraction with a total solids (TS) content of 21.9% (Tait et al., 2015). No published data were found for the Z-Filter applied to dairy slurry/effluent. The physical and chemical characteristics of slurry/effluent from different animal types are distinct, and this influences solid-liquid separation (Hjorth et al., 2010).

One property of slurry/effluent that poses well-known challenges for solid-liquid separation is the particle size distribution of its organic matter and nutrient contents. Specifically, the majority of solids (expected to be mostly organic matter) and nutrients are typically found in fine particles <125 μm (Meyer et al., 2007) not easily separable by a common mechanical screen or press with a typical cut-off size of 0.5 mm (Peters et al., 2011). The addition of cationic polymer can increase the recovery of nutrients and organic matter, but appears to be ineffective at removing dissolved nutrients (Liu et al., 2016). Moreover, low charge density cationic polymers can be more effective for manure coagulation and flocculation, whilst high charge density cationic polymers can be more effective in reducing pathogen levels (Liu et al., 2016).

The efficiency of flocculants in terms of TS removal tends to decline with a reduction of TS concentration in the slurry/effluent (Rico et al., 2012). This influences treatment costs and thus feasibility, if dosage requirements and costs are proportionally higher for dilute slurry/effluent. Polymer flocculants have a relatively high cost compared to conventional coagulants (Rico et al., 2007), so polymer flocculants have often been co-dosed with coagulant to complement their function (Krumpelman et al., 2005), thereby reducing their dosage and cost. Different coagulant effects on pH may be important for end-use applications, specifically as Ca-coagulants tend to increase pH whereas aluminium (Al)- and iron (Fe)-based coagulants instead can depress pH. A lower pH would reduce ammonia volatility (Hjorth et al., 2010), but a pH closer to neutral may facilitate downstream nitrification/denitrification (Szogi et al., 2006) to further reduce nitrogen loading. In terms of P recovery, precipitation with multi-valent cations (Ca(II), Fe(III), Al(III)) could be important (Hjorth et al., 2010; Mohamed et al., 2020). However, commercially viable options for large scale disposal of alum sludge have not been well studied, perhaps due to perceived concerns regarding Al toxicity (Dassanayake et al., 2015) which may limit the use of Al-based coagulants with acidic soils. In contrast, Ca-coagulants are generally suggested to be less effective in separation than Fe and Al-based coagulants (Hjorth et al., 2010), but Ca is commonly applied in agriculture and is known for its potential to recover P as calcium mineral precipitates (Cichy et al., 2019).

The current study tests a closed-loop separation approach for dilute dairy effluent produced by a milking parlour, using the Z-Filter as separation technology option. This is the first time that test data are reported for a Z-Filter applied to dairy effluent. The study was performed at full-scale due to a general lack of scaled model systems for commercial solid-liquid separation technologies, but separation efficiency is characterised using metrics that would allow cross-comparison with other commercial technology options. The investigations aimed to fill an important data gap by clarifying solid-liquid separation options for dilute pasture-based dairy systems to recover nutrients and organic matter into useable products (liquid and solid fractions, separately). As a result, on-farm environmental management can be improved by reducing nutrient leaching/run-off risk and decreasing fugitive methane emissions from uncovered effluent ponds. This provides practical alternatives for controlled recycling using closed-loop concepts, enabling dairy farms to reduce their environmental footprint. The study also evaluated coagulation-flocculation to control carbon and nutrient capture, and thereby target economic benefits that facilitate sustainable environmental concepts.

2. Materials and methods

2.1. Materials

Industrial-grade hydrated lime (Cockburn, Kwinana, WA) containing 65–75% $Ca(OH)_2$ and 3.5–5% magnesium hydroxide, and garden lime ($CaCO_3$) (Richgro, Perth, WA), were used in the experiments. Water used in the milking parlour at the trial site (including for washing) was bore water extracted at the site and filtered with a sand filter. The quality of the bore water before filtering was moderate to poor, with a low pH (5.5–6.4) and a high iron content, close to the short-term trigger value in the Australian water quality guidelines (ANZECC, 2000). This caused iron mineral scale, so the filtered water used in the dairy was instead used as service water for the Z-Filter throughout the trial.

Cationic polymer flocculant emulsion (Drewfloc 2488) produced by Ashland Chemicals (Wilmington, US) was identified as preferred by a supplier via a preceding series of jar tests onsite. Before using it in the trial, the flocculant was diluted to 0.5% by mixing 5 L of concentrated flocculant emulsion with 1 kL of filtered water (as above) at initial high shear conditions in a centrifugal pump (DAVEY, Model CY70-90/A, 1.2 kW). From here, the diluted flocculant solution was transferred into a 1 kL tank with slow continuous mixing by a 1 kW overhead stirrer unit. This solution was freshly made for each sampling day/event.

2.2. Experimental trial

The trial site was a predominately pasture-based dairy (ryegrass) in southwest WA, milking 1,500 cows year-round, and producing approximately 11.5 million litres of milk per annum. Milking took place twice daily with cows brought in from pasture paddocks in four separate groups to be milked. The cows were herded onto a concrete yard for a short time before being guided into a rotary milking system, and then immediately released back out to the pasture paddocks. As a result, the average time that cows spent on concrete surfaces from which manure was collected was estimated to be 1.5 h per day, which determines the amount of manure collected and influences effluent properties, as below.

The cow herd was supplied with additional feed, including cut silage from the site, mainly fed at the end of summer in feed-out paddocks and grain bought in and fed year-round during milking. The site has winter-dominant rainfall (617 mm from May to August), with a mean annual rainfall of 982.3 mm (Bureau of Meteorology, 2021).

Dairy effluent at the site consisted of cow excrement, cleaning chemicals, milk residues and spilt feed, collected in wash water (Section 2.1). Approximately 110 kL of effluent was produced daily, which drained by gravity into a concrete collection sump (24 m length, 2 m width, 1.5 m depth). The site owner installed mixing/agitation in the sump to keep manure solids suspended whilst the effluent was being pumped out into one of four nearby effluent ponds (estimated total footprint = 10 ha). The mixing/agitation within the sump provided a consistent effluent for separation testing. The effluent from the holding ponds (not part of the trial) was usually irrigated over a nearby paddock of about 8 ha, not considered large enough to fully utilise nutrients.

2.3. Trial apparatus, including Z-filter

The trial apparatus is summarised in Fig. 1. A floating pump fed the mixed effluent from the collection sump (Section 2.2) into a 9 kL feed tank (D = 2.3 m). The feed tank was continuously stirred with an overhead 2 kW mechanical agitator with four 45-degree angle pitch blades (diameter 80 cm), positioned approximately 70 cm above the tank floor. The effluent (from hereon called influent) was pumped from the feed tank using a progressive cavity pump (Netzsch, Nemo, 3 kW, Selb) with variable speed drive, via a magnetic flow meter (Krohne, Optiflux2000-DN80, Wellingborough, 0–3600 (±0.5%) L min^{-1}), via 8–20 m of 100 mm pipe (length varied to vary flocculation time, see below), and into the Z-Filter.

The Z-Filter (Model Z300, patented, Z-Filter, South Africa) had a maximum influent processing capacity of 30 kL h^{-1}. Its functional operation has been described in detail elsewhere (Payne, 2014; Z-Filter, 2021). However, in short (Fig. 1), the influent is fed onto the filter mesh at the top of the Z-Filter, the filter mesh then folds into a tubular shape and seals, moving diagonally downwards with gravity drainage along its length. The filter mesh then changes direction and moves upwards through a set of rollers, and then horizontally, again through a set of two dewatering compression rollers, with applied pressure altered by adjusting an air supply pressure at 100–400 kPa. Subsequently, the filter mesh opens, discharges its solid fraction via a set of scrapers into a screw conveyer chute, and returns to the feed point past cleaning water jet sprays.

Filtrate collects at the base of the Z-Filter and flows out by gravity into a 1 kL cylindrical in-ground concrete pump sump, from where it was pumped with a submersible pump into a 50 m^3 storage tank (border of trial apparatus). Routinely, the filtrate was pumped from this storage tank via existing onsite irrigation infrastructure (not part of the trial apparatus).

The separated solid fraction was conveyed first via a screw conveyer and then a belt conveyer into an adjacent roofed storage shed (border of trial apparatus). From here, the solid fraction was semi-periodically collected and combined with other organic materials to be composted onsite (not part of the trial).

When flocculant was dosed, it was added in-line at a tapping point just after the main effluent magnetic flow meter. The flocculant was dosed with a progressive cavity pump (Netzsch, Nemo, 0.6 kW, Selb) with variable speed drive, and the dosage flow measured with a magnetic flow meter (Krohne, Wellingborough, optiflux1000, DN15, 0–125 (±0.5%) L min^{-1}). When lime was dosed (Section 2.4), it was added as a powder directly into the 9 kL feed tank, with the resulting pH measured using a pre-calibrated portable pH meter (model WP-80, TPS, Brendale).

2.4. Test procedure

The test procedure is summarised in Fig. 2. Grouped samples (influent, filtrate, and solid fraction) were collected on designated sampling days for set test conditions. For each sampling, the feed tank was filled with a semi-homogeneous batch of fresh influent to be processed. The feed tank took about 35–45 min to empty by operation of the Z-filter during which time 3 L sub-samples of influent and filtrate were collected every 5 min. An average of five influent and five filtrate sub-samples were collected in this way for each sampling. These influent sub-samples were collected from a tap on the Z-Filter feed line before the point where flocculant was dosed (when relevant). The filtrate sub-samples were collected from the end of a pipe discharging filtrate into the in-ground concrete pump sump (Section 2.3). Each set of sub-samples was combined into a 20 L aggregate of influent and a separate 20 L aggregate of filtrate. These were then stirred continuously with a steel Paint Drill Mixer (Model Universal Power Mixer, UNi-PRO, Kilsyth) before being representatively sub-sampled into bottles. After sub-sampling, pH was measured without delay using the portable pH meter above (Section 2.3).

Approximately 20 L of solid fraction was also collected during the same time period when influent and filtrate samples were being collected. The solid fraction was collected in a plastic transporter crate placed directly under the discharge chute of the screw conveyer (Section

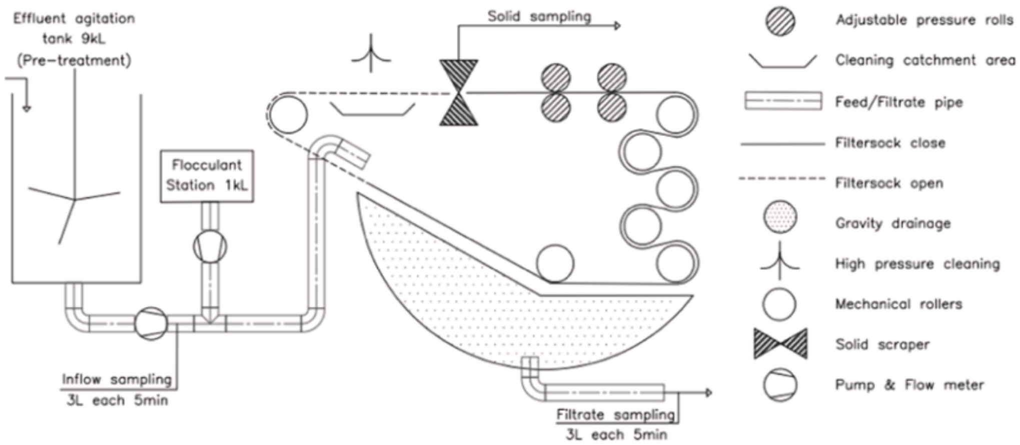

Fig. 1. Illustrated schematic of the Z-Filter with its important features.

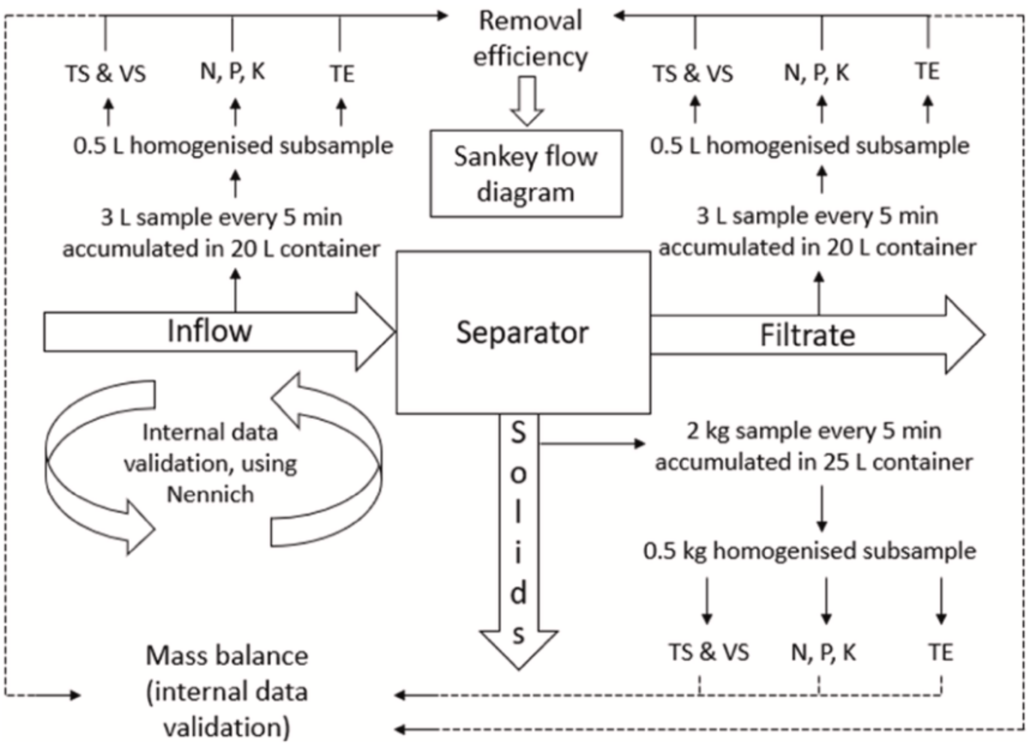

Fig. 2. Graphical summary representation of the test methodology.

2.3). An average of five grab sub-samples of solid fraction were collected in this way. These sub-samples were combined in a larger bucket and thoroughly mixed, including by hand by full inversion, and then representatively sub-sampled into a sample jar. Without delay, each sample group (influent, filtrate, and solid fraction) was cooled before being transported cold to a laboratory for analysis. All samples were analysed for TS, VS, TKN and total elements (Section 2.6).

The operational settings of the Z-Filter (i.e. influent flow rate; flocculant flow rate if dosed; compression roller pressure; speed of travel of the filter mesh) were selected to prevent overfilling and clogging of the Z-Filter and to produce a 5–10 mm thick solid fraction layer on the filter mesh as recommended by the Z-Filter supplier. For each sampling event, the operational conditions were selected, set, kept constant, and recorded.

Table 1 summarises the test conditions applied during the trial. The amount of lime added to selected tests, was as required to increase the pH of the influent to a consistent value of 9.2(±0.1). Sampling occurred over an approximate two-year period (September 2019 to July 2021). Hence, the measured results would have reliably represented seasonal variability due to feed differences, rain addition during wet periods, climate, and any other onsite changes, so that the trial results could be considered representative of "typical" pasture-based dairy farming conditions.

Table 1
Test conditions during the Z-Filter trial.

Conditions[a]	Pre-treatment	Flocculant[a]	Influent flowrate [L min^{-1}]	Sock speed [Hz]	Compression roller pressure [bar]
No lime or floc	No	None	250–400	5–10	2–3.2
5% floc	No	5%	200–350	10–25	1–2.8
5% floc & lime	Lime to pH 9.1	5%	200–350	15–25	1–1.5
3% floc & lime	Lime to pH 9.2	3%	200–350	15–20	1–1.5

[a] 5% floc & lime means an influent pre-treated with lime added to increase pH to 9.2 (±0.1), and subsequent addition of pre-diluted flocculant (section 2.1) at a flocculant flow rate equal to 5% of the influent flow rate.

2.5. Analytical procedures

TKN was analysed using the Kjeldahl standard method (APHA, 1995), with the resulting ammonia measured on a Lachat flow injection analyser as per the Lachat QuickChem Method 31-107-06-1-A. Samples were diluted 1:6 before digestion with potassium persulfate and 1:20 with Milli-Q water to bring the sample into measurement range.

Total elements, including P, Na, Al, Ca, Cu, Fe, K, Mg, S, and Zn were measured by inductively coupled plasma optical emission spectroscopy (ICP-OES) using a PerkinElmer Optima 5300DV (PerkinElmer Corp., Norwalk Ct, USA). For ICP-OES measurements on liquid samples (influent or filtrate), 4 mL of liquid sample were pre-digested with 2 mL nitric acid and 0.5 mL of H_2O_2 before being made up to 20 mL for analysis. For ICP-OES measurements of the solid fraction, the samples were dried at 70 °C and finely ground. 0.15 g dried material was accurately weighed and pre-digested with nitric acid and perchloric acid before being made up to a 10 mL solution for analysis.

Total carbon (C) and N in pre-dried (70 °C) solid fraction were measured by an Elementar Vario Macro combustion analyser (Hanau) according to the Dumas method.

TS and VS for all samples were measured according to Standard Methods procedure 2540G (APHA, 2005).

2.6. Data analysis and statistical methods

All test conditions (Table 1) were sampled with an appropriate number of replicates for statistical comparison. The data presented below are calculated mean values in replicates with 95% confidence intervals, determined using a two-tailed student t-distribution with appropriate degrees of freedom (n-1, where n is the number of samples).

To check the validity of the trial data, theoretical amounts of nutrients in dairy effluent for the site were estimated using the empirical correlations of Nennich et al. (2005) as given by Birchall et al. (2008) (Fig. 2). Assumptions for this estimation were: average milk yield = 19.2 L cow^{-1} d^{-1}; 1,400 milked cows; average cow-time on concrete = 1.5 h day^{-1} (Section 2.2). This theoretical nutrient production was compared with nutrient amounts estimated by multiplying nutrient concentrations measured in the influent by 110 kL day^{-1} (Section 3.1).

Separation performance of various solid-liquid separation devices is commonly assessed and compared using a quantitative parameter index (Birchall et al., 2008). Literature shows several approaches to determine this parameter, which depends on the operations and available setup and equipment (Birchall et al., 2008). However, the most commonly used (including for commercial devices) and probably simplest is the removal efficiency (R_E) as follows (Eq. (1)) (Hjorth et al., 2010):

$$R_E = \frac{C(x)_{influent} - C(x)_{filtrate}}{C(x)_{influent}} \tag{1}$$

where $c(x)_{influent}$ and $c(x)_{filtrate}$ are measured concentrations (g L^{-1}) of the analyte (x) of interest (e.g. TS, VS, TKN, TP, etc.) in the influent and filtrate, respectively. This parameter assumes that influent and filtrate have a much lower TS content than the solid fraction, so that the influent volume and filtrate volume are approximately equal (Birchall et al., 2008).

In the current study, the typical variance within repeat measurements for a sample was observed to be small as compared to the variance between measurements for different samples collected at the same test condition. Accordingly, individual removal efficiencies calculated for each condition by Eq. (1) were grouped and average values and standard deviations estimated for each group. Standard deviation is presented below as the measure of variance, unless otherwise stated.

A mass balance was performed to estimate the mass of TS produced as solid fraction. This determined the TS mass in the influent and in the filtrate from measured TS concentrations and 110 kL d^{-1} of influent or filtrate (see assumptions above and Fig. 2). The TS mass in the solid fraction was then calculated by difference and divided by the measured TS concentration in the solid fraction to estimate total mass of the solid fraction produced. Lastly, measured analyte concentrations were multiplied by the total mass of solid fraction to estimate the amount of each respective analyte recovered in the solid fraction. This mass balance was repeated for randomly selected samples and analytes and the obtained results were found to be internally consistent, indicating data validity. The split of nutrients, TS and VS to the filtrate and solid fraction was calculated using average influent composition and R_E values, and then presented in Sankey diagrams prepared using the software e!Sankey 5 pro (ifu Hamburg GmbH, Hamburg).

3. Results

3.1. Influent and filtrate characteristics, and recovery efficiency

Table 2 presents measured pH, TS, VS, N, P, K and Ca for the influent and corresponding filtrate samples collected during the trial. Trace element concentrations are provided in the Supplementary Material (Table S1). Fig. 3 shows calculated R_E values for each test condition. Fig. 4 presents mass flows on Sankey diagrams (Section 2.6). Based on the measured concentrations, the daily amounts of TS and nutrients collected in the 110 kL day^{-1} effluent were estimated at 0.62 tonnes TS, 24.8 kg N, 6.2 kg P and 17.6 kg K. These compared favourably with theoretical estimates (Section 2.6) of 0.68 tonnes dry matter (DM, analogous to TS), 6.1 kg P, and 17.1 kg K. The exception was N with a theoretical value of 37.5 kg N, which likely resulted from upstream volatilisation losses (see Section 4.1).

Without lime or flocculant addition, measured analytes in the influent and filtrate were not significantly different (Table 3), as also reflected in the low or zero calculated R_E values (Fig. 3). Removal of N, P, and K was also not significant ($p > 0.05$). The only exceptions were TS and VS for which R_E values appeared to be definitively greater than zero (Fig. 3A).

The addition of a 5% flocculant dosage caused a notable removal of all analytes (Table 2, Fig. 3), except for K, which is usually in mobile dissolved form not removed by mechanical separation. The removal of

Table 2
Measured characteristics of liquid samples collected during the trial, presented as average values ± 95% confidence intervals.

	pH	TS [% wet]	VS [% wet]	N [mg·L^{-1}]	P [mg·L^{-1}]	K [mg·L^{-1}]	Ca [mg·L^{-1}]
No Lime or Floc							
Replicate	n = 7*	n = 6	n = 5	n = 6	n = 6	n = 6	n = 6
Influent	7.1(±0.4)	0.59(±0.12)	0.46(±0.15)	221(±12)	62(±10)	154(±20)	98(±15)
Filtrate	7.0(±0.4)	0.44(±0.06)	0.31(±0.07)	227(±19)	60(±10)	153(±20)	93(±13)
5% Floc							
	n = 4	n = 4	n = 4	n = 4	n = 4	n = 4	n = 4
Influent	7.4(±0.3)	0.52(±0.2)	0.39(±0.14)	231(±117)	50(±12)	168(±89)	100(±56)
Filtrate	7.4(±0.3)	0.16(±0.05)	0.07(±0.06)	105(±70)	29(±7)	158(±85)	60(±27)
Typical influent before lime addition	n = 11	n = 10	n = 9	n = 10	n = 10	n = 10	n = 10
	7.2(±0.3)	0.56(±0.09)	0.43(±0.08)	225(±29)	57(±7)	160(±24)	99(±16)
5% Floc & Lime							
	n = 7	n = 7	n = 7	n = 6	n = 6	n = 6	n = 6
Influent & Lime	9.2(±0.1)	0.66(±0.24)	0.47(±0.18)	240(±67)	53(±8)	169(±44)	242(±53)
Filtrate	9.2(±0.1)	0.18(±0.04)	0.05(±0.02)	117(±41)	4.9(±2.5)	160(±47)	85(±19)
3% Floc & Lime							
	n = 6	n = 6	n = 6	n = 6	n = 6	n = 6	n = 6
Influent & Lime	9.2(±0.1)	0.61(±0.24)	0.4(±0.17)	229(±59)	45(±8)	181(±40)	190(±39)
Filtrate	9.1(±0.1)	0.23(±0.11)	0.1(±0.07)	154(±66)	13(±11)	169(±34)	93(±39)

n = number of samples for which corresponding mean values are given.

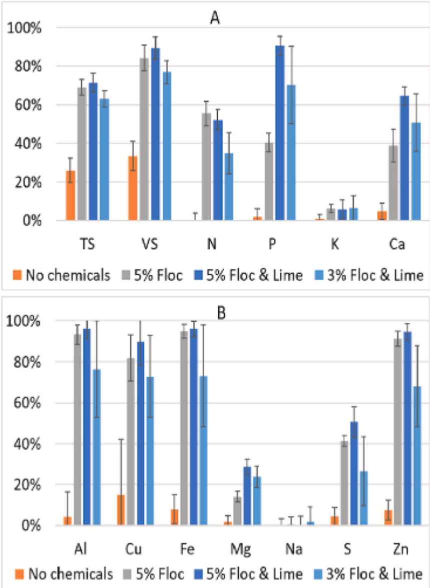

Fig. 3. Removal efficiency (R_E) (%) values (Eq. (1)) for (A) TS, VS, N, P, K, Ca, and (B) Al, Cu, Fe, Mg, S, and Zn. Values given are calculated means in replicates and the error bars are estimated standard deviations.

macro-nutrients (N and P) was accompanied by a removal of Fe, Al and trace elements such as copper (Table S1, Fig. 3B). Flocculant dosing did not alter measured influent pH.

As expected, the addition of lime to pH 9.2 resulted in much higher Ca in the influent (Table 2). However, interestingly, there was no significant difference in Ca in the filtrate between tests with and without lime pre-treatment (Table 2, Fig. 4). This indicated that most of the added Ca ended up in the solid fraction (Section 4.1). Addition of lime and flocculant considerably increased P removal compared to only dosing flocculant (Figs. 3 and 4), indicating a complementary effect of lime and flocculant. This benefit of lime was also observed at a flocculant dose of 3% (Table 2, Fig. 3) but the results were more variable (Table 2) indicating that 3% was likely the minimum dose for consistent N and P removal. The removal of N was insensitive to lime but relied on flocculant (Fig. 3A).

3.2. *Solid fraction characteristics*

Table 3 summarises measured composition of the solid fraction. TS was consistent for the tested conditions, indicating that the compression rollers achieved a consistent dewatering extent. Regardless of the tested conditions, a stackable solid fraction was produced with a much-increased concentration of all measured analytes (Table 3) as compared to the influent (Table 2). Without any flocculant or lime addition, VS/TS ratio was highest (Table 3), indicating that mostly organic manure fibres were being removed. The resulting C/N ratio was 46.2 in the solid fraction. The addition of flocculant significantly increased N and P in the solid fraction, and also decreased the C/N ratio to 12.9–25.9. The pre-treatment of influent with lime resulted in a higher Ca concentration in the solid fraction (Table 3).

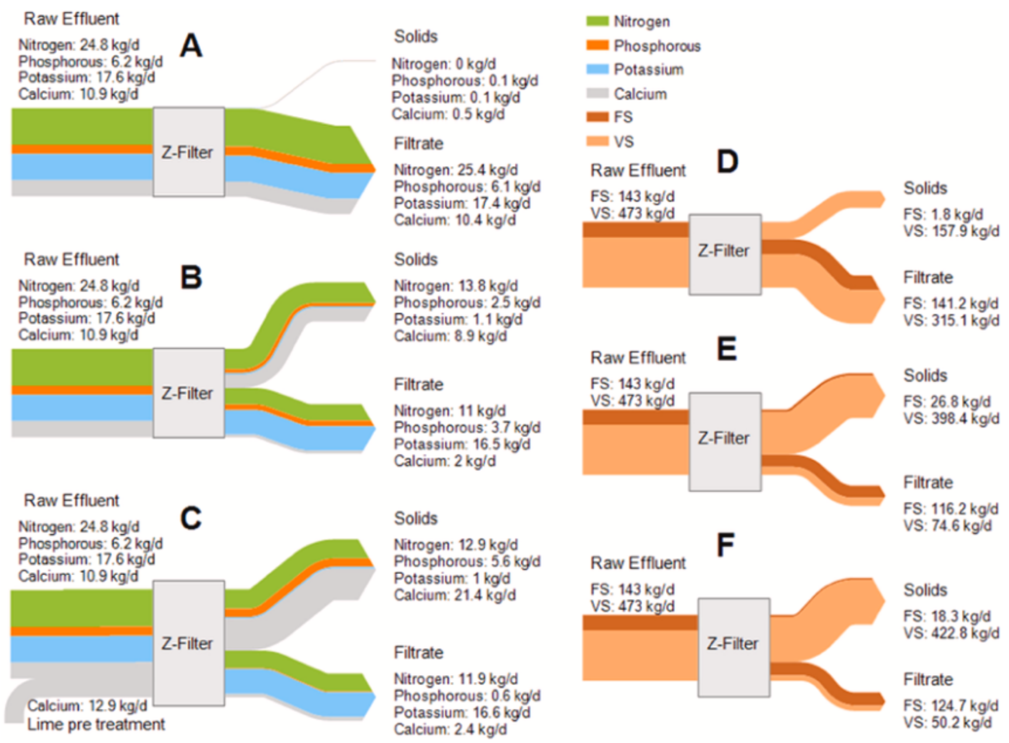

Fig. 4. Average estimated mass flows of analytes during the trial. (Fixed solids (FS) equals TS minus VS). This includes Sankey diagrams (A, D) without any lime or flocculant; (B, E) with 5% flocculant only; and (C, F) with both lime and 5% flocculant.

Table 3
Characteristics of solid fraction under the tested conditions (on a DM basis, unless otherwise stated). Values are means in replicates given with ±95% confidence intervals.

TS [% wet mass basis]	VS [% wet mass basis]	VS/TS ratio [-]	N [g·kg^{-1}]	P [g·kg^{-1}]	K [g·kg^{-1}]	Mg [g·kg^{-1}]	Ca [g·kg^{-1}]
No Lime or Floc							
n = 5*	n = 7		n = 6	n = 6	n = 6	n = 6	n = 6
17.5(±1.3)	18.5(±2.7)	1.06	11.4(±1.2)	1(±0.3)	1.1(±0.2)	1.3(±0.1)	4.7(±1.5)
5% Floc							
n = 4	n = 3		n = 4	n = 4	n = 4	n = 4	n = 4
19.6(±8.7)	12.1(±3.6)	0.62	24.8(±14.9)	3.6(±2.4)	1.4(±0.5)	1.9(±0.8)	8.2(±3.3)
5% Floc & Lime							
n = 6	n = 3		n = 5	n = 5	n = 5	n = 5	n = 5
17(±1.6)	13(±3)	0.76	26.2(±6.6)	9.8(±5.2)	1.5(±0.3)	4.7(±1.5)	27.7(±10.3)
3% Floc & Lime							
n = 5	n = 2		n = 5	n = 5	n = 5	n = 5	n = 5
16.2(±2.1)	13.8(±1.6)	0.85	23.1(±6.1)	8(±2.9)	1.7(±0.3)	4.4(±0.9)	23.8(±6.2)

* n = number of samples.

3.3. Z-filter operational observations and energy use

Despite the mixing in the effluent collection sump, manure solids in the dairy effluent tended to settle out. As a result, the TS content of the influent in the collection sump typically progressively increased (e.g. from 0.4% to 1.2%) if multiple batches of influent were processed on the same sampling day. For this reason, the Z-Filter sampling runs were performed on separate batches of influent to minimise inter-sampling variability and was conducted across multiple sampling days. The changes in TS content required changes in the Z-Filter operational settings to allow for a more consistent build-up of solid fraction and to prevent overfilling of the filter mesh. Typical solid fraction thicknesses on the filter mesh during the trial were 5–15 mm, achieved by visual inspection and adjustment of the filter mesh travel speed, influent flow rate and compression roller pressure. Unfortunately, during normal operation, this required frequent operator intervention to vary operating conditions as TS in the influent progressively increased.

The addition of flocculant tended to further complicate operations, making the solid fraction generally "sticky" and less easily dewaterable than without flocculant. For this reason, to prevent blockages and build-up of contents inside the Z-Filter with flocculant use, the influent flow rate and applied compression roller pressure typically had to be reduced, and the filter mesh travel speed usually had to be increased.

Visual observations indicated that slow mixing of influent with flocculant in the main influent progressive cavity pump and a longer length of pipe leading up to the Z-Filter (Section 2.2) promoted better flocculation and decreased particles visible in the filtrate.

Large solids (e.g. plastics, tools) were occasionally found in the influent, which caused blockages in the Z-Filter inlet port. For example, a plastic cap was found in the inlet, which significantly reduced the inlet diameter and appeared to adversely affect the visual performance of the Z-Filter. When this blockage was promptly cleared, the regular operation of the Z-Filter was restored. No data were included in the analysis nor in the results tables above (Sections 3.1 and 3.2) for such partially blocked operating conditions, as these were infrequent and were not considered representative of routine operation.

Routine maintenance included cleaning off any adhering solids from the Z-Filter, and visual inspections, typically requiring about 10 h week^{-1}. Less frequent operator intervention included cleaning of the water spray nozzles, alignment of the filter mesh and supplying grease to the internal moving parts. Under the trial conditions, the filter mesh had to be replaced about every 3–4 month due to wear and/or damage.

Energy consumption associated with the Z-Filter operation was tracked during the trial. The incremental increase in onsite electricity consumption due to operation of the Z-Filter was estimated at 4 kWe, and for ancillary equipment (e.g. mixers, pumps, air compressor and conveyer belt) an additional 18 kWe. This included electricity requirements of the mixing system in the effluent collection sump.

4. Discussion

4.1. Influent characteristics, and coagulation-flocculation effect on resource recovery

Seasonal and operational effects on dairy effluent characteristics can include the time that cows spend on concrete surfaces from which manure is collected, dilution with rain, and the quantity and quality of water used and collected as effluent. The typical variation in standard deviation for measured influent composition values in the current work were 10–20%, deemed to be reasonable compared to other field studies (Moller et al., 2007). Moreover, the mass balance estimates of TS, P and K in the influent aligned well with theoretical estimates (Section 3.1), indicating that the study results were valid and representative of dairy effluent in general. The exception was N, which appeared to be subject to substantial upstream volatilisation losses. Previous research has confirmed volatilisation losses off milking yards (Aarons et al., 2017). Volatilisation of ammonia N would further increase with lime addition, due to elevated pH increasing the proportion of free ammonia. However, the large fraction of N captured in the solid fraction with flocculant and lime use (Section 3.1) also indicated that the influent contained substantial non-volatile particulate N yet to be mineralized.

The addition of flocculant was important for TS, VS, N, and P recovery in the solid fraction (Sections 3.1 and Supplementary material). It showed that a large proportion of solids, N and P were in non-filterable fine particulate form or in colloidal or soluble fractions, consistent with the findings of others (Powers et al., 1995). The results further suggested that 3% (flow-based) was probably the minimum flocculant dosage required to achieve consistent and reliable recovery of macro-nutrients (Section 3.1).

The addition of lime complemented the function of the flocculant, greatly increasing P recovery (Section 3.1). Mass balance analysis indicated that most of the Ca added as lime ended up in the captured solid fraction (Section 3.2) and is therefore likely to be in particulate form. Lime solubility was not expected to be limiting at pH 9.2, because saturated lime solutions typically have a much higher pH > 12.0. Instead, lime dissolution likely released Ca which induced subsequent P-mineral precipitation (Monballiu et al., 2018; Rugaika et al., 2019). This interpretation was corroborated by side bench scale experiments which tested the effects of various calcium chemicals and pH increase on P removal (Supplementary material, Section S3). Elevating pH also increases the proportion of free phosphate (PO_4^{-3}) which can promote Ca minerals precipitation (Kazadi Mbamba et al., 2015).

The addition of garden lime in the bench tests showed a lower level of P removal than hydrated lime (Supplementary material, Section S3) and garden lime was found to be operationally problematic in the field trial because of poor solubility and settling out in the feed tank despite continuous mixing (Section 2.3).

The elevation of pH by hydrated lime will, however, increase ammonia volatility (Hjorth et al., 2010), so future closed-loop concepts might consider the recovery of targeted nutrients, whilst minimising the loss of other nutrients (Section 4.2). Other methods could also be explored for enhanced recovery of mobile N, including precipitation as the mineral struvite. Struvite has been identified as a slow-release fertilizer which can reduce P losses to the environment (Muys et al., 2021).

4.2. *Z-filter performance compared to other commercial technologies*

Like a belt filter press, the Z-Filter uses a combination of gravity filtration to remove the bulk filtrate volume, followed by pressure filtration to achieve effective dewatering of the solid fraction. Like an inclined screen, gravity filtration in the Z-Filter occurs on an incline to prevent build-up of a solid cake on the filter mesh via continuous action of fluid shear across its surface. The Z Filter achieves effective dewatering through mechanical compression, similar to a Screwpress (compression) or a Wendelfilter (vacuum and compression).

The lack of model versions to test commercially available separators at smaller scale is an on-going challenge. For this reason, testing in the current trial had to occur at full-scale to simulate real hydraulic, shear and compression conditions. This is also necessary because coagulation-flocculation chemicals, usually selected based on simple "bucket chemistry" via jar testing, may not translate well into full-scale application, because of complex hydraulics, shear and flocculant formation and structure interactions. To attempt to address this, the Britt dynamic drainage jar (BDDJ) tester had been previously developed to simulate a full-scale paper machine at laboratory scale, and this has been previously tested on algae recovery (Musa et al., 2020). It would be interesting and worthwhile to develop similar model test apparatus for other commercially available separation technologies.

A previous study (Payne, 2014) explored the Z-Filter for piggery effluent treatment in a TS range from 1.3% to 2.4% and used a commercial coagulant Floquat FL 2949 and a similar flocculant to the current work. That study achieved similar or moderately lower VS, TKN, and P removals of 73%, 35% and 65%, respectively, and similarly produced a stackable solid fraction with an average TS and VS content of 21.9% and 19.1%, respectively (Payne, 2014). The same study (Payne, 2014) indicated that chemical costs (coagulant and flocculant) dominated the overall economics, estimated at AUD84 per tonne TS treated. This is one reason why the current study investigated hydrated lime as a less costly coagulant-aide.

4.3. *Environmental implications and potential benefits*

Mechanical solid-liquid separation can facilitate closed-loop concepts for the recovery of organic matter and nutrients from dilute livestock wastes. The process can reduce environmental impacts and risks, such as by reducing fugitive methane emissions from effluent storage and the risk of nutrient run-off into surface water or leaching into groundwater. This is achieved by decreasing organic matter stored in uncovered effluent ponds to reduce anaerobic conversion into fugitive methane emissions (Laubach et al., 2015), and by reducing and/or controlling nutrients being land-applied as effluent/filtrate.

The use of coagulation-flocculation chemicals offers control over the recovery of nutrients and carbon. The current study showed that the addition of hydrated lime was cost-effective to complement the function of the flocculant and increase P recovery. Lime is commonly applied to agricultural soils. It can increase soil pH, which could be beneficial for acidic soils. This is because Fe and Al can decrease P availability in acidic soils, either as free Fe and Al cations in soil solution, or as exchangeable Fe and Al cations occupying available exchangeable sites of soil colloids, or as mineral oxide clay-sized colloids adsorbing P (Antoniadis et al., 2015). These acidic Fe and Al cations react with P species, reducing their activity (Weil and Brady, 2017). Increasing soil pH can impair the effects of Fe and Al on P, thereby increasing P availability (Antoniadis et al., 2015). Accordingly, the best practice in acidic low-P soils, is to add P and lime concurrently (Antoniadis et al., 2015).

The elevated solid content of a recovered solid fraction makes it more readily transportable to be further processed (e.g. into biogas energy and/or compost) and/or land-applied. Moreover, nutrient run-off risk is directly related to soil moisture status and the amount of nutrients being land-applied; so, by allowing the controlled storage of nutrients and controlled land-application of a reduced amount of nutrients at times when most needed by crops, the risk of surface run-off and groundwater impacts can be minimised.

Mechanical solid-liquid separation recovers effluent nutrients and carbon to be beneficially and safely recycled to soils, including to displace synthetic fertilisers. The latter is important, considering for example that Australia is a net importer of fertilizer nutrients (Mehta et al., 2016), and the global demand and associated costs of fertilisers continue to be erratic and will likely increase over time (Fertilizer Australia, 2021). The possibility to recycle effluent/nutrients by solid-liquid separation could help decrease the global dependency on non-renewable fertilizers. However, short and long-term agronomical benefits should be investigated in future work.

In the present work, calculations were performed to assess the high-level cost feasibility of the separation concept in dairies, including the cost of the Z-Filter, energy costs (Section 3.3), lime and flocculant costs (estimated at AUD68 per tonne TS treated) and operator labour costs (Table S2, Supplementary Material). The current analysis indicated that if an operator was continuously required to alter Z-Filter operating conditions (Section 3.3) or quickly shut the filter down to protect the equipment from damage by infrequent internal blockage, a water supply interruption, or another malfunction, the labour costs would dominate and this would result in a negative cashflow (Table S2). It may be possible to minimise the requirements for operator input (and labour costs) to a more practical level by implementing an appropriate level of automation. The analysis suggested that if only a minimal amount of weekly maintenance of $\sim$10 h week^{-1} is required (Section 3.3), estimated payback period could be as short as 3–4 years. The financial benefit here mostly originated from the substantial value of the solid fraction used instead of commercial compost to maintain on-farm soil productivity. In fact, when no flocculant was used, the temperature of the piled solid fraction seems to have a near-optimum C/N ratio for composting (Section 3.2) and was found to generate heat during storage up to a measured temperature of 50+°C at approximately one week after collection. This suggests the potential value and applications of organic matter in the captured solid fraction.

4.4. *Regional environmental sustainability context and implications*

The broader environmental sustainability context originated from efforts by government agencies and other relevant stakeholders to improve conditions of estuaries in the South-Western WA region in support of urbanisation, tourism, and recreation. Actions taken in response included a program with a range of strategies to facilitate dairy effluent management system upgrades and environmental technology adoption (Department of Water and Environmental Regulation, 2019). This study, testing the effectiveness and efficiency of a closed-loop dairy effluent management option, formed part of this program. The study tested a complementary approach to the current practice of storing effluent in uncovered effluent ponds with associated fugitive methane emissions. The aim was to identify complementary strategies that could promote cost-feasible and practical beneficial reuse of effluent, to protect on-farm and off-farm environments. Into the future, governments may play a similar crucial role in facilitating sustainable solutions for environmental management and protection in agriculture, by supporting innovations of environmental-related technologies for achieving sustainable growth and environments (Khan et al., 2022).

5. Conclusions

The current work tested a closed-loop separation approach for dilute dairy effluent from pasture-based dairies, using a commercial separation technology (Z-Filter) applied at full-scale. This was to provide options for on-farm environmental management aiming to minimise fugitive greenhouse gas emissions and reduce nutrient leaching and run-off risks. Separation without cationic polymer flocculant and hydrated lime recovered 25.9% TS and 33.4% VS into the solid fraction, but achieved no notable N, P and K recovery, indicating these macro-nutrients were predominantly in poorly separable fine particle or soluble fractions. The addition of a 5% flow-based dose of cationic polymer flocculant recovered the majority of TS (69%) and VS (85%), and also notable amounts of N (52–56%) and P (40%). Flocculant together with hydrated lime (added up to pH 9.2) greatly increased P recovery (91%). This is important to provide farmers with the ability to control nutrients and organic matter in separated liquid and solid fractions for preferred beneficial reuse options. Moreover, the stackable solid fraction from separation (in this case 16–20% TS) would be more readily transportable, thereby facilitating further processing (e.g. into compost or biogas) and beneficial reuse. Overall, the results indicated that solid-liquid separation could enable closed-loop nutrient and carbon resource recovery for better environmental management. However, the economics and practicality of operation could be further improved to facilitate widespread application (e.g. by increasing the level of automation). Other separation technologies should also be tested to determine suitability. Future research should also assess the agronomic value of land application of the separated fractions, including before and after further processing. This should include short- and long-term agronomical benefits, which may include composting of the solid fraction prior to land application. This should also consider the separate environmental implications of flocculant, including potential effects of flocculant and its degradation products on short- and long-term soil health. Solid-liquid separation performance may also differ for digestate, effluent pond sludge, or pure manure, because these contain different manure fibre concentrations, and this could be tested in future work. In conclusion, solid-liquid separation is seen as a key technology step to facilitate more sustainable agriculture that protects the environment. This is achieved by recovering and diverting manure organic matter away from uncovered effluent ponds, thereby reducing fugitive methane emissions, and providing options for improved beneficial reuse of nutrients and organic matter as valuable natural resources.

Credit author statement

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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.jenvman.2022.116254.

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CHAPTER 6: PAPER 3 – BIOCHEMICAL METHANE POTENTIAL OF DAIRY MANURE RESIDUES AND SEPARATED FRACTIONS: AN AUSTRALIAN-WIDE STUDY OF THE IMPACT OF PRODUCTION AND CLEANING SYSTEMS

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This Chapter builds on the precious studies for a sustainable manure management across different production systems. Nutrient concentrations were not significantly different, but notable relationships did occur. For example, Chapter 4 highlights the potential precipitation of calcium phosphate, which was used to increase separation efficiencies and facilitate resource recovery (Chapter 5). This reduces the organic loading rate into uncovered lagoons, resulting in less fugitive methane emissions.

This study focuses on the investigation of biochemical methane potential (B_0) values, which play an important role in accurate emissions estimations, biogas evaluations, and precise decision-making within the industry. The detailed investigation of 12 commercial farms, including 29 different sample types, presents for the first time B_0 values for PB dairy products, which are common in Australia and New Zealand. The TS concentration of effluent was influenced by cleaning methods, but their effect on B_0 values was minimal. Furthermore, the influence of solid liquid separation on methane yields was addressed This might enable the preservation of methane yields for biogas capture and renewable energy generation.

The results of this study offer opportunities to minimise the carbon footprint of dairy farms and make a valuable contribution towards sustainable manure management in livestock agriculture, to achieve a circular economy.

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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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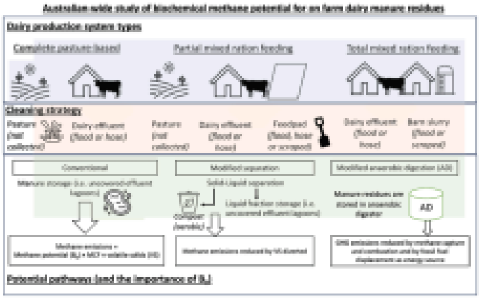
HIGHLIGHTS

- 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

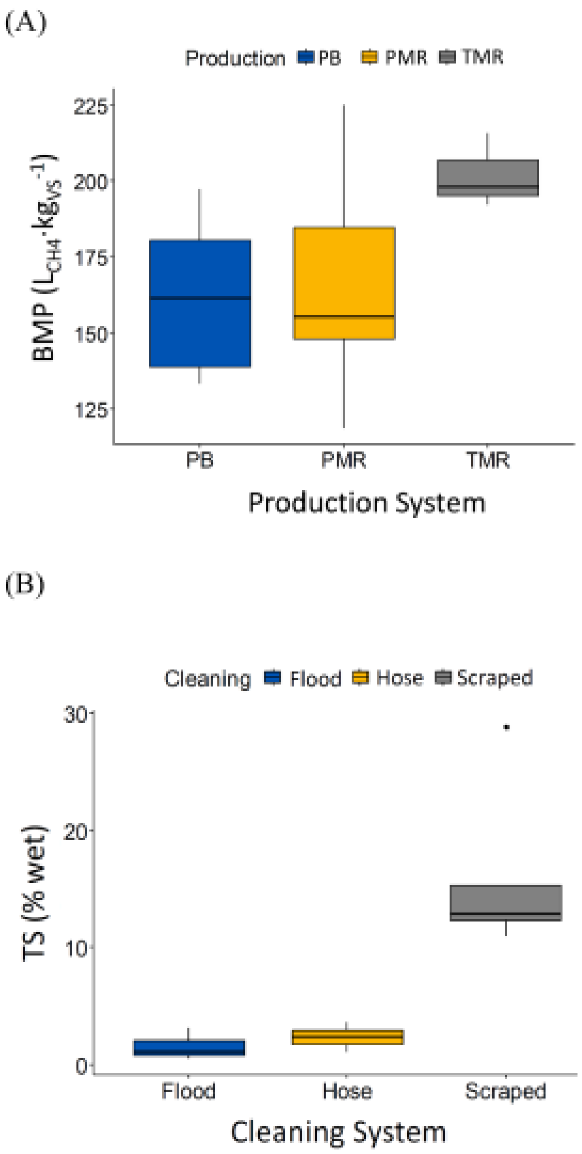

Fig. 1. Summary statistics as box-and-whisker plots, showing (A) TS in manure/effluent as affected by cleaning method, and (B) B_0 of dairy effluent as affected by production type. Outliers are also shown as single data points in the case of TS, which were excluded from the statistical analysis.

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.

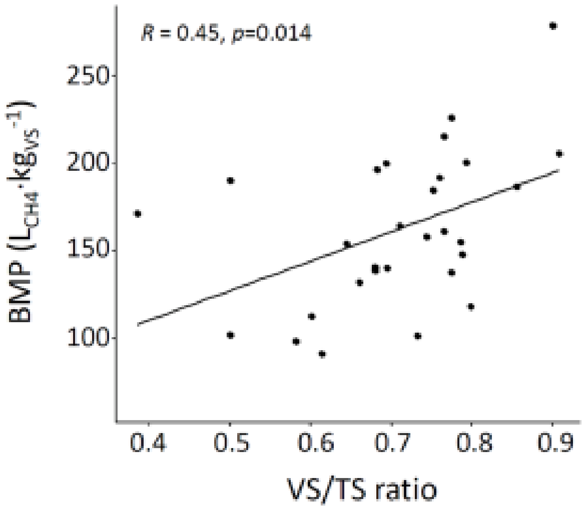

Fig. 2. Correlation plot between B_0 and VS/TS ratio (for all samples) tested in the current work. Note that measured VS was used in this case, without adding measured VFAs.

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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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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Supplementary Material

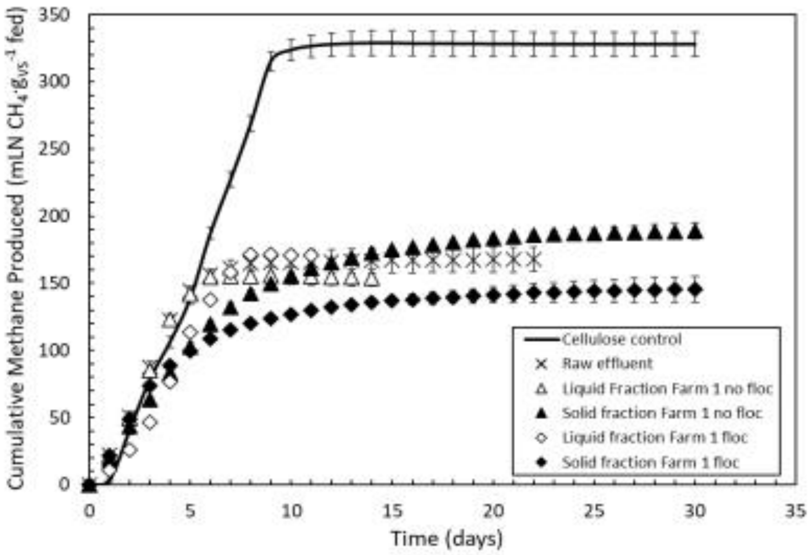

Fig. S1. Example raw data from the biochemical methane potential tests, specifically in this case showing data for Farm 1, including results for the raw effluent (prior to any separation) and liquid and solid fractions from separation without (Sample 1a) and with (Sample 1b) the aid of flocculation chemicals. Error bars show standard deviation for triplicates.

Table S1 Summary Statistics for B_0 by Production system, including descriptive statistics, Shapiro-Wilk Normality Test, Levene's Test for Homogeneity of Variance, Type III Wald Chi-Square Tests (**with** cleaning as a random factor), and the Pairwise Comparisons (Kenward-Roger Method)

Production	n	Mean	SD				
PB	7	161.0	26.9				
PMR	5	166.0	40.4				
TMR	3	202.0	12.3				
Shapiro-Wilk Normality Test							
Variable	Statistic	p-value					
B_0	0.954	0.584					
Levene's Test for Homogeneity of Variance							
df1	df2	Statistic	p-value				
2	12	1.10	0.364				
Analysis of Deviance Table (Type III Wald Chi-Square Tests) with random factor cleaning							
Response	Chi-Square	Df	p-value				
Intercept	124.7884	1	< 2e-16				
Production	5.7824	2	0.05551				
Pairwise Comparisons (Kenward-Roger Method)							
Comparison	Estimate	Std. Error	df	t-value	Lower	Upper	p-value
ProductionPB - ProductionPMR	-7.9537	17.9314	10.9	-0.443	-47.466	31.5594	0.66604
ProductionPB - ProductionTMR	-51.0484	26.7021	11.9	-1.911	-109.28	7.1930	0.08031
ProductionPMR - ProductionTMR	-43.0947	25.7173	12.0	-1.675	-99.13	12.9484	0.11967

Table S2 Summary Statistics for B_0 by Production system, including descriptive statistics, Shapiro-Wilk Normality Test, Levene's Test for Homogeneity of Variance, Type III Wald Chi-Square Tests (**without** cleaning as a random factor), and the Pairwise Comparisons using emmeans

Production	n	mean	sd		
PB	7	161.0	26.9		
PMR	5	166.0	40.4		
TMR	3	202.0	12.3		
Shapiro-Wilk Normality Test					
variable	statistic	p			
B_0	0.954	0.584			
Levene's Test for Homogeneity of Variance					
df1	df2	statistic	p		
2	12	1.10	0.364		
Type III ANOVA					
Sum Sq	Df	F value	Pr(>F)		
(Intercept)	182340	1	196.0492	8.52e-09	
Production	3620	2	1.9461	0.1853	
Residuals	11161	12			
Shapiro-Wilk Test for Residual Normality					
W	p-value				
0.97097	0.8721				
Pairwise Comparisons Using emmeans					
contrast	estimate	SE	df	t.ratio	p.value
PB - PMR	-4.86	17.9	12	-0.272	0.9602
PB - TMR	-40.49	21.0	12	-1.924	0.1743
PMR - TMR	-35.63	22.3	12	-1.600	0.2832

Table S3 Summary Statistics for TS by cleaning type, including descriptive statistics, Shapiro-Wilk Normality Test, Levene's Test for Homogeneity of Variance, Type III Wald Chi-Square Tests (**with** Production as a random factor), and the Pairwise Comparisons using emmeans

Cleaning	n	Mean	SD				
Flood	7	1.46	1.04				
Hose	8	2.32	0.855				
Scraped	5	16.0	7.35				
Identified Outliers							
Cleaning	Farm	Production	Recycling	TS	B_0	is.outlier	is.extreme
Scraped	5b	PMR	No	28.8	119	TRUE	TRUE
Shapiro-Wilk Normality Test for TS							
Variable	Statistic	p-value					
TS	0.679	0.0000220					
Shapiro-Wilk Normality Test for Log-transformed TS							
Variable	Statistic	p-value					
TS.log	0.881	0.0186					
Levene's Test for Homogeneity of Variance							
df1	df2	Statistic	p-value				
2	17	0.149	0.862				
Analysis of Deviance Table (Type III Wald Chi-square tests)							
Response	Chisq	Df	p-value				
Intercept	28.681	1	8.533e-08				
Cleaning	120.412	2	< 2.2e-16				
Shapiro-Wilk Normality Test for Residuals							
W	p-value						
0.91807	0.09096						
Pairwise Comparisons (Kenward-Roger Method)							
Comparison	Estimate	Std. Error	df	t-value	Lower	Upper	p-value
Flood - Hose	-0.3831	0.175035	16.6	-2.188	-0.7531	-0.01309	0.04324
Flood- Scraped	-1.8723	0.178724	15.4	-10.47	-2.252	-1.49221	2.094e-08
Hose - Scraped	-1.4891	0.189616	16.4	-7.853	-1.8904	-1.08791	6.091e-07

Table S4 Summary Statistics for TS by cleaning type, including descriptive statistics, Shapiro-Wilk Normality Test, Levene's Test for Homogeneity of Variance, Type III Wald Chi-Square Tests (**without** Production as a random factor), and the Pairwise Comparisons using emmeans

Cleaning	variable	n	mean	sd			
Flood	TS	7	1.46	1.04			
Hose	TS	8	2.32	0.855			
Scraped	TS	5	16.0	7.35			
Outliers for TS by Cleaning							
Cleaning	Farm	Production	Recycling	TS	B_0	is.outlier	is.extreme
Scraped	5b	PMR	No	28.8	119	TRUE	TRUE
Shapiro-Wilk Normality Test for TS							
variable	statistic	p					
TS	0.679	0.0000220					
Shapiro-Wilk Normality Test for Log-transformed TS (TS.log)							
variable	statistic	p					
TS.log	0.881	0.0186					
Levene's Test for Homogeneity of Variance (Log-transformed TS)							
df1	df2	statistic	p				
2	17	0.149	0.862				
Type III ANOVA for Cleaning (Response: TS.log)							
	Sum Sq	Df	F value	Pr(>F)			
(Intercept)	4.8351	1	41.744	5.865e-06 ***			
Cleaning	12.0683	2	52.096	5.614e-08 ***			
Residuals	1.9691	17					
Shapiro-Wilk Normality Test for Residuals (lm)							
W	p-value						
0.92844	0.1441						
Pairwise Comparisons for Cleaning (Tukey Method)							
Contrast	Estimate	SE	df	t.ratio		p.value	
Flood - Hose	-0.337	0.176	17	-1.912		0.1655	
Flood - Scraped	-1.942	0.199	17	-9.744		<.0001	
Hose - Scraped	-1.605	0.194	17	-8.272		<.0001	

CHAPTER 7: DISCUSSION AND CONCLUSIONS

7.1. Research contribution and implementation

The pressure to develop sustainable manure management is growing, and research activities that translate theoretical knowledge into practical application become more important. Typically, research activities are not only judged by their contributions to science but also by their broader implications. This might include the establishment of policy, on-farm practices, or future research paths. This section discusses and explains the major contributions of this thesis to the current state of knowledge, as well as its wider implications for practical implementation and research opportunities.

7.1.1. Effluent characteristics and nutrient concentration of different production systems

<u>Research Contributions</u>

The first objective of this thesis was "to understand the variation in physico-chemical characteristics of effluent generated from different dairy production systems". This objective was addressed by the investigations in Chapter 4 of the thesis. The aim was to obtain valuable insights that could contribute towards the implementation of sustainable manure management practices and resource recovery.

A key finding was the lack of significant differences in nutrient concentrations in dilute effluent across various commercial dairy production systems. Differences in mean values were present but did not vary between production systems above typical variability in the dataset ($p>0.05$). This implies that effluent characteristics and nutrient concentrations are similar regardless of the production system.

This is important because the similarity in effluent characteristics might suggest that recovery approaches can be generalised and efficiently implemented across the industry.

While effluent concentrations did not significantly differ across different production systems, total capture rates of nutrients (N, P, and K) were notably and significantly higher in TMR-fed indoor systems than in pure PB systems ($p<0.1$). This was expected because cows spend more time on concrete, from which manure residues are collected. This is important for resource recovery strategies and nutrient management on farm. The different amounts of point-source nutrients from the different production systems need to be included into a sustainable nutrient management on farms.

Lastly, the work described in Chapter 4 identified potential relationships between nutrients and other waste characteristics, such as TS, pH, and EC. In total, 73 significant correlations were identified. This enables a detailed understanding, such as determining if nutrients are predominantly in particulate or mobile form. Furthermore, it might indicate promising relations between nutrients to form mineral precipitates, which are relevant for nutrient recovery (Chapter 5). Understanding these relationships can facilitate targeted recovery methods that increase separation efficiency and effectiveness. This was demonstrated by the example of calcium phosphate precipitation that can enhance P recovery (Chapter 5).

<u>Implementation</u>

The detailed nutrient composition can be used for future research, industrial practises, and policy decision-making for sustainable manure and soil management. The implementation of circular technologies might be able to convert dairy residues into reusable fertilisers and organic soil amendments. Thereby reducing environmental risks and reliance on synthetic fertilisers, and ultimately contributing to improved soil health as well as crop growth.

Environmental nutrient point sources and potential risks can be evaluated by lifecycle assessments, combining the effluent characteristics and capture rates from different production systems across Australia. This can then help to identify opportunities for circular technology and its environmental implications.

It might be highly beneficial for agriculture in general to combine the optimisation of separation efficiency with fertiliser applications. This can be achieved by the use of organic fertilisers, produced from manure residues, such as slow-release fertilisers containing struvite or calcium phosphate.

7.1.2. Full-scale implementation of chemically enhanced solid-liquid separation of dilute dairy effluent

Research Contributions

The second objective of the thesis was "to optimize carbon and nutrient recovery from dilute dairy effluent through chemically enhanced solid-liquid separation techniques." This was addressed by investigations outlined in Chapter 5 of the thesis.

Applied scientific research at full scale was a major novelty of this thesis. Prior to the thesis investigations in Chapter 5, previous published research in the field of chemically enhanced solid-liquid separation of manures was mostly limited to laboratory experiments (Ellison & Horwath, 2021; Sherman et al., 2000; Wang et al., 2020). The importance of past research to general implementation could thereby have been somewhat limited by scale because results were not always confirmed in real-world situations. For example, Rico et al. (2007) suggested full-scale trials would be needed to confirm laboratory results. By implementing and researching the Z-Filter technology in a commercial dairy farm setting, this limitation was addressed, and its effectiveness confirmed.

Detailed data on the mixing ratios and composition of the effluent inflows and outflows around the separation technology were provided. This gave insights into the actual performance and efficiency of the specific technology on dilute dairy effluent, for which data were obtained for the first time. However, in addition, a unique combination of cationic polymer flocculant with lime resulted in a surprising and significant improvement in P recovery to the solid fraction of up to 90%. This, together with the discovered correlations between nutrients and calcium in Chapter 4, could indicate an important role of calcium-phosphate precipitation in determining the recovery efficiency of P from dilute dairy effluent. These findings may be useful in future research for the potential formulation of novel residue-derived fertilisers, including for reuse in organic soil amendments. This could also successfully address the regional objectives of the water quality improvement plan from the Department of Water and Environmental Regulation (the environmental authority in the Australian state where the research in Chapter 4 was conducted) by helping to protect local waterways (White, 2012), specifically by targeting nutrient recovery and reduction from dairy effluent in the region.

Implementation

The first full-scale application of a Z-filter on a commercial PB dairy farm was one of the major achievements of this thesis, tackling the well-known challenge of gaining control over nutrient use with heavily diluted effluent. Manure fibre removal facilitates the conveyance of particle-free filtrate to be used cost-effectively via conventional farm equipment such as pipes, pumps, and irrigation systems. Moreover, dewatered solids can be cost-effective to transport for further processing or soil application where most needed, rather than being locally applied across small areas close to the dairy shed or lagoon with a nutrient surplus.

Despite the success of the current demonstrated application of solid-liquid separation at the large-scale trial dairy operation (1,400 herd), the average dairy herd size in Australia is only 300, generally with limited labor and financial capacity. Moreover, the operational complexity and investment costs of commercially available mechanical separators (such as implemented in the current work) will likely exceed the available capacities, limiting their applicability. Therefore, recovery strategies are needed for smaller farms, balancing removal efficiencies, operational complexity, and associated costs.

Chemically enhanced separation with the addition of lime and flocculant showed surprisingly good P removal for dilute dairy effluent, and it may be possible to build on these successes to explore application in simpler, more cost-effective separation systems for smaller farms. More research is also needed to achieve or optimise the recovery of other nutrients, such as N or K.

The ability to influence effluent characteristics via controlled separation could be useful to achieve desired concentrations in the liquid and solid fractions from separation, thereby better matching applications to specific crops or soils. This is an interesting field, especially in combination with potential further stabilisation treatments such as composting or anaerobic digestion to stabilise carbon content, respectively addressed in Chapters 5 and 6 of the thesis. This may also be important to address concerns related to the potential toxicity in soils of chemicals used for enhanced separations, such as polymer flocculants.

7.1.3. Biochemical methane potential of the Australian dairy sector

<u>Research Contributions</u>

Objective 3 of this thesis was "to evaluate the biogas production potential of manure residues from diverse dairy production systems in Australia by measuring their biochemical methane potential." This is addressed by the investigations outlined in Chapter 6 of the thesis.

This part of the thesis contributes to the scientific community by filling key gaps in existing literature and providing the first comprehensive dataset of B_0 values for manure residues in the Australian dairy sector. There were no published studies available prior to the thesis investigations that measured B_0 for on-farm dairy manure effluent in

Australia. Accordingly, Australia's National Inventory (NGER) has to date been using the IPCC default value for B_0 of 240 $L_{CH4}\cdot kg_{VS}^{-1}$ for dairy cattle manure (Commonwealth of Australia, 2022). Australian industry guidelines listed B_0 of 180-250 $L_{CH4}\cdot kg_{VS}^{-1}$ (Birchall et al., 2008) based on international literature sources. This is despite the IPCC (2006) suggesting that country-specific values for B_0 be measured and applied, especially for livestock because of varying diet profiles. The default B_0 value of 240 $L_{CH4}\cdot kg_{VS}^{-1}$ (IPCC (2006)) appeared to be significantly higher than the mean of 161.1(±43.6) $L_{CH4}\cdot kg_{VS}^{-1}$ found in the current work, which would lead to higher carbon emissions predictions from manure management in the Australian dairy sector. The values obtained from the thesis investigations should be considered for updating Australian industry guidelines (Birchall et al. (2008) and for incorporation into national dairy sector emissions estimates.

Measurements indicated B_0 of pure PB manure being 161(±26.9) $L_{CH4}\cdot kg_{VS}^{-1}$ was comparable (within experimental error) to that of TMR-fed cattle being 202.0(±12.3) $L_{CH4}\cdot kg_{VS}^{-1}$, with the effect of production system type being not significant relative to statistical variability in the dataset. However, potential differences in B_0 could be caused by different dietary intakes. The results showed that manure capture rates were much higher for TMR and PMR as compared to PB. This clearly demonstrates that a generalised approach would be ineffective for the entire dairy sector in terms of assessing emissions profiles and/or energy recovery potentials. Solutions for different production systems and herd sizes need to be further investigated, implemented, and scientifically tested, including at full scale.

The investigations in Chapter 6 of this thesis additionally showed an impact of cleaning strategy (floodwash vs. hosing vs. scraping) on TS concentrations of collected manure residues. Flood wash systems produced the most dilute effluent, followed by high-pressure hosing, with a mean TS of 1.46(±1.04) % and 2.32(±0.86) %, respectively. Scrape cleaning predominantly results in a slurry, with a TS concentration of

16.0± (7.35) %. This is important for recovery platforms because observed TS concentration changes are directly related to their performance and suitability for nutrient and organic matter recovery. Heavily diluted effluent can cause problems for efficient nutrient and organic matter recovery (Chapters 5 and 6). Furthermore, these differences also have implications for the selection of AD technologies. This is because systems producing predominantly liquid effluent may be better suited for CEPs, whereas systems producing more concentrated manure residues (slurry) may benefit from CSTRs, and the higher TS may overcome the associated hydraulic limitations of AD (Chapter 2).

Mechanical solid-liquid separation can, however, also facilitate closed-loop concepts for the recovery of organic matter and nutrients from dilute livestock wastes (Chapter 5). The separation process can reduce environmental risks by reducing VS loading rates in uncovered effluent lagoons to reduce anaerobic conversion into fugitive CH_4 (Laubach et al., 2015). Furthermore, solid-liquid separation controls nutrients in the liquid and solid fractions for flexible processing options such as AD or composting and subsequent land application as effluent or filtrate. The thesis investigations in Chapter 7 importantly showed that mechanical separation preserved B_0 values in the filtrates, especially notable for PB systems. In contrast, passive separation methods, such as trafficable solids traps with weeping walls, indicated a reduced methane yield of the resulting filtrates compared to the inflows. Furthermore, maintenance of those systems may influence energy recovery.

Daily cleaned solid traps might be able to preserve methane yields, but this would be difficult to achieve in practice because cleaning out of solids is labour-intensive and therefore more often conducted on a weekly, fortnightly, or even monthly basis.

Implementation

The thesis study fills gaps in existing studies, confirming the importance of country specific B_0 values and providing such values for emission predictions in the Australian dairy sector. In addition, reliable B_0 values are essential to quantify biogas recovery potentials for the efficient implementation of AD technology in dairy farms. In this regard, it is noted that currently there are very few AD installations at dairies in Australia (Chapter 2). Lastly, B_0 values are also important for identifying strategies for GHG reduction in the dairy industry through optimised manure management. Collecting more data across different types of dairy operations across Australia would, however, further assist in understanding and revealing significant differences in methane yields obtained from TMR vs. PMR vs. PB. A broader dataset might enable us to observe significance above the typical high variability in full-scale sampling. To further assess potential differences between B_0 in TMR and PB systems, B_0 and microbial analysis of pure manure could be conducted, which reduces the influences of commercial farm operations, such as impacting dilution extent and cross-contamination. This could then further explore the importance of microbial communities in manure management systems.

There is a need for a deep investigation into how ways of managing practices, including feeding regimes and waste processing systems, alter both the composition of manure and its biogas potential. The potential influence of forage types on the methane yields from manure residues is exciting because it could either reduce emissions from manure storage or beneficially boost yields when energy recovery platforms are in place. Further research in this aspect is important because understanding the synergies between methane yields and feed intake could lead to optimizations in biogas production and the development of more sustainable livestock management practices.

Finally, further research is needed into the broader environmental and economic implications of dairy intensification, including, but not limited to, life-cycle assessments (using the revised B_0 values) and the economic feasibility of VS and biogas recovery technologies.

7.2 Resource recovery implications

The extensive sampling of commercial farms and full scale research implementation, emphasizes that it is crucial to understand the individual farm infrastructure and effluent characteristics before implementing a recovery technology. There is no one-size-fits-all solution due to significant differences, as explained in Chapter 5 and further explored in Chapters 4 and 7. This is even though multiple recovery technologies are commercially available. The implementation of technology for circular manure management is important for sustainable agricultural in the future (Chapter 1). It is fundamentally crucial to highlight the need for specifically designed closed-loop systems to suit the individual production systems in place, as well as the existing infrastructure and operations at the dairy farm. A one fit all solution will never account for all occurring variances across different farm operations. However, modified implementations can allow for more efficient resource recovery by accommodating site-specific factors into the design and technology selection. This section discusses two circular technologies explored in the thesis investigations, namely solid-liquid separation, and anaerobic digestion.

7.2.1. Solid liquid separation technologies

Separation technologies in manure management are used to provide flexibility to control and transport nutrients as well as organic matter (carbon). As highlighted in Chapters 5, these technologies need to meet a variety of objectives and face several limitations. The selection of a suitable technology depends on specific operational needs. This includes the TS concentrations of the effluent, the desired quality of recovered solids, sludge, and filtrate required, as well as energy and investment considerations. The most practicable separation technologies for livestock operations are explained below:

Trafficable Solid Trap (or weeping walls)

This is a low-tech system that catches solid waste in a trap by sedimentation. No energy input and only minimal investment is required for its implementation. However, it is sensitive to sludge quality and has high maintenance needs to achieve functionality. Preserving methane yields can only be achieved by daily cleaning of the solids trap, but this would be labour-intensive and is highly unlikely in practice. However, trafficable solid traps show best performance when effluent streams are consistent over time. This makes them ideal candidates for PB or PMR systems with dilute effluent, originating from hose or flood wash systems.

Screwpress

This is a reliable separation technology, which uses a screw mechanism to put pressure on sludge against a cylindrical mesh. It is relatively energy-efficient while producing high-quality sludge and removing organic matter from the influent. Moreover, it is relatively sensitive to sludge quality and requires relatively little maintenance, which can be done by farmers or service providers. However, this technology is not suitable for dilute effluent because a TS concentration above 2% is required for efficient separation (Hjorth et al., 2010).

Therefore, this technology can be implemented for slurry from TMR systems but not for dilute effluent. Additionally, this technology could be highly effective for digestate separation after AD in a CSTR and is broadly used in Europe for manure separation.

Inclined Screen

This system uses a sloped screen to separate solids from liquids by using gravity. It can be particularly sensitive to TS and deliver low-quality sludge and TS removal, but with relatively few energy inputs and minimal cost requirements. From observations in the thesis, the application seems to be more suitable for PMR and TMR systems because two investigated PB dairy operations had functionality difficulties with this system in operation. Neither of the inclined screens in PB systems were operating, while one at a TMR system demonstrated robust functionality for dairy effluent accumulated by floodwash. This could indicate suitability constraints for this particular technology.

Decanter Centrifuge

Separates sludge components using centrifugal force in a spinning drum. This relatively high-energy, high-investment system produces high-quality sludge and filtrate while also effectively removing TS and particulate nutrients of a range of sizes. This high-quality separation might only be economically viable when the effluent is converted into commercial fertilizer at a relevant scale.

Z-Filter

As explained in detail in Chapter 5 of this thesis, this system presses sludge against a filter sock, producing high-quality solids and filtrate, and is less susceptible to sludge and effluent fluctuations. However, operation requires significant know-how and maintenance to operate functionally and would benefit from a higher degree of automation.

This technology appeared to be most suitable for PB and PMR operations with floodwash, where vast amounts of effluent are produced in a short timeframe. This is because of the high-volume flow up to $400 L \cdot min^{-1}$, which is a unique characteristic compared to other separators.

Belt Filter Press

Separates sludge by applying pressure to a cloth or metal belt. This energy- and investment-intensive system produces high-quality sludge and filtrate and provides good TS and particulate nutrient removal, but as for the decanter centrifuge, it is generally most suitable for larger scales.

Wendel Filter

In this technology, a rotating drum with small holes immersed in sludge is used to pull liquid through the drum via a vacuum. This leaves the solids on the surface, which are then transported to the top and then finally disposed. This relatively low-energy and low-cost system produces medium- to high-quality filtrates. This simple technology requires minimal maintenance and is known for robust operation. This technology was developed especially for the treatment of dilute effluent, focusing on particle-free filtrate and dewatered solids. Though it runs reliably, it is not suitable for TS concentrations above 3%. Therefore, the Wendel filter could be implemented well in both PB and PMR systems to treat dilute effluent from floods or hose washes, especially in smaller dairies, due to its lower investment cost compared to other mechanical separation systems. However, it should be noted that the processing capacities do not exceed 1 $m^3 \cdot h^{-1}$, which might limit the implementation on large scale operations.

Table 9 provides a comprehensive overview of commercially available separation technologies and their important performance characteristics. This might be valuable for the decision-making processes of farmers and researchers for final implementation of circular technology. The matrix was created by the author primarily to compare the Z-filter to other available recovery technologies.

Table 7 Solid liquid separation technology matrix

	Traffic-able solid trap	**Screw-press**	**inclined screen**	**Decanter centri-fuge**	**Z-Filter**	**Belt filter press**	**Wendel-filter**
Reference	(Birchall et al., 2008; Dairy Australia, 2021b)	(Cielejewski, 2019; Hjorth et al., 2010)	(Birchall et al., 2008; Varma et al., 2021)	(Hjorth et al., 2010; Varma et al., 2021)	(Grell et al., 2023; Payne, 2014)	(Cielejewski, 2019; Hjorth et al., 2010; Varma et al., 2021)	(Cielejewski, 2019)
Energy	-	low	minimal	high	medium	high	low
invest	minimal	low	low	high	high	high	low
TS of Influent	< 2 %	> 2 %	low	low	low	low	< 3 %
Sensitive to sludge quality	high	medium	high	high	low	medium	medium
Mainte-nance	high	low	medium	medium	high	high	medium
Com-plexity	low	medium	low	high	high	high	medium
Sludge quality	low	high	low	medium	high	high	high
Filtrate quality	low	medium	medium	high	high	high	medium/ high
TS removal	low	high	low	high	high	high	high
Solid moisture	An-aerobe	medium	high	high	low	low	low
nutrient removal	low	medium	low	high	high	high	high
Flow rate by under 1% TS	medium	low	medium	medium	high	medium	medium
Reliability	medium	high	medium	medium	low	high	high
Contained com-ponents	low	low	low	high	high	low	low
Chemical treatment	not possible	possible	not possible	required	possible	possible	possible
Fugitive methane emissions	high	low	low	low	low	low	low
Potential use in	PB	PMR & TMR	PB, PMR & TMR	PB, PMR & TMR	PB & PMR	PB & PMR & TMR	PB & PMR

7.2.2. *Anaerobic digestion options*

Anaerobic digestion should continue to be a key technology in future manure management. This is because AD not only provides a controlled waste treatment option but also energy recovery in the form of biogas (Chapter 2). Furthermore, AD is known as the most efficient technology to reduce carbon emissions (Belflower et al., 2012). This dispatchable renewable energy source can be potentially used when limited wind and sun are available or simply to power the baseload of the dairy farm. However, the efficacy of the process can vary based on several factors, as highlighted in the current work. The most important factor is the dairy production system in place and the cleaning method being applied. Due to the increased capture rates, TMR systems may produce more concentrated manure slurry than the dilute dairy effluent from PB systems. The thesis investigations could not directly confirm this (Chapter 7), but they did show a random variable effect of production system type on TS. This indicates that different AD options could be more suitable for manure residues from different production types. Furthermore, the cleaning method—floodwash vs. hose cleaning—influences the TS content in the effluent. For example, higher TS concentrations often require higher investment costs because of the larger volume of the digester (Chapter 2). The following section lists the potential AD technologies, including their practical implementation based on the findings of this thesis.

<u>Covered Effluent Pond</u>

CEPs are a cost-effective AD technology to capture fugitive methane from manure residues for combustion, particularly for PB and PMR systems with highly diluted effluent. This is because CEPs are most efficient when dealing with liquid manure containing less than 2% TS (Angelidaki et al., 2018). This is especially the case for PB and PMR systems investigated in this thesis with hose or floodwash cleaning methods.

CEPs are relatively simple to implement, by only covering existing lagoons with an impermeable cover. This then collects the biogas occurring from the AD process.

Despite their low cost and effectiveness in odour removal, they require a large amount of land space and offer limited and slow process control because of their large size and volume. However, these limitations could be addressed through process modifications, such as internal heating and even mixing. Furthermore, CEPs may be well-suited for treating the liquid fraction of mechanical solid-liquid separation, a process that efficiently eliminates manure fibre while preserving methane yields prior to a CEP (Chapter 6). This is especially interesting because this thesis demonstrated the quicker biogas production from filtrates compared to the effluents, which ultimately results in smaller digester sizes and investment costs.

Plug-Flow (PF) Digestion technology

PF digestion technology presents a viable option for manure management, especially when dealing with slurry that has higher total solids (TS) concentrations in the range of 10-14%. This technology might be used for TMR systems and scraped feedpads from PMR systems. In PF, manure is channelled through a lengthy, often tunnel-like concrete chamber, which is sealed with an airtight membrane. The manure first enters a mixing pit for solids concentration regulation through water addition (dilution can be also achieved by using the diluted effluent from the milking operation). The system also offers the flexibility of thermal control, maintaining either mesophilic or thermophilic conditions. With a solids retention time of 15 to 30 days, these systems offer a specific set of advantages and restrictions. The longer retention time may impact throughput, but it ensures effective biogas production and nutrient breakdown.

Continuous Stirred Tank Reactor

CSTRs are extremely effective for treating manure, particularly scraped manure (slurry) from TMR systems, with TS concentrations already being near suitable, ranging from 9% to 11.5%. CSTRs are designed for robust agitation and can support a wide TS spectrum of 5-14% while maintaining a solids retention time of 10–30 days (Fantozzi & Buratti, 2009).

Their continual mixing and temperature control allow faster anaerobic digestion rates compared to low-rate digesters such as CEPs. Furthermore, their scalability, with sizes ranging from a few hundred to a few thousand cubic meters, allows them to suit a variety of industrial applications. Co-digestion is worthwhile to mention in this context, especially with dairy manure as a substrate. However, in the context of TMR systems, the design of CSTR`s allows for the beneficial reuse of the diluted effluent from milking operations to dilute the scraped slurry. This improves both, pumping and digester mixing efficiency (Qi et al., 2020). Despite a wide operating flexibility, this technology provides strong performance at TS concentrations around 8% -9% (Angelidaki et al., 2018). Therefore, CSTR is a suitable technology various manure residue streams, also including scraped manure from feedpads of PMR systems.

Upflow Anaerobic Sludge Blanket (UASB)

UASB technology demonstrates efficient performance in managing low concentrations of TS below 3%. Consequently, these systems offer efficient treatment options, especially for the liquid fraction (Filtrate) from solid liquid separation, which can form granules without a pre-fermentation step (Rico et al., 2015). UASB systems are known for their high processing rates and improved biogas generation (de Mendonça et al., 2017). These characteristics demonstrate efficacy in the treatment of tiny particles due to their increased surface area, which accelerates the digestion process (Monballiu et al., 2018).

In addition, mechanical separation techniques are well-suited for these systems and effectively preserve the B_0 value during separation (Garcia et al., 2008). Moreover, the possibility of improving biogas generation via chemically improved separation techniques presents an attractive case for the UASB's adaptability and effectiveness in particular waste management contexts (Chapter 5 and 6).

7.3 The Australian dairy industry and its BMP potential

Australian dairy farming relies primarily on PB systems, though intensive feeding systems are emerging and account for about 26% of the industry (Australia, 2017; Watson & Watson, 2015). Therefore, dairy manure residues are mostly particulate, in the form of liquid effluent (Birchall et al., 2008). The effluent is dilute with a low solid content and consists mainly of wash water, cattle excreta, and cleaning agents (Tait et al., 2021a). The total volume of accessible manure waste has been estimated by using methods from the Australian National Greenhouse Accounts (Commonwealth of Australia, 2018). This assumed an average milk yield of 16.5 kg per cow per day, a liveweight gain of 0.016 kg per day, and a milking cow weight of 550 kg. Accordingly, the daily manure output was estimated to be 4.5 kg of VS per head. This daily output multiplied by 1.44 million cows (the total cow population in Australia (Section 1.1)) and the manure proportion not voided on pastures (=1-0.2) (Christie et al., 2018) showed an accessible amount of 475 kilotons VS·annum^{-1} (Tait et al. (2021a). The current thesis investigations showed the influence of different production systems on manure capture rates (Chapter 4 and 6) not included in the estimates of Tait et al. (2021a). The same work also used a theoretical CH_4 yield of 200 $L_{CH4}\cdot kg_{VS}^{-1}$., which seems reasonable considering the range of values found in Chapter 7 of the thesis. Based on this, a total biochemical methane potential of approximately 94,951,000 $m^3{}_N$ methane per annum was estimated by , equivalent to a theoretical energy potential of 931 GWh·a^{-1}.

To assess translation into energy used at dairies, specific energy efficiencies would need to be applied, which range from about 35% for electricity generation to about 80% for thermal energy generation (Gur, 2016).

Based on the data from the current thesis investigations, future manure capture could range from 475 kilotons VS·annum^{-1} for predominantly PB, to 815 kilotons VS·annum^{-1} for predominantly PMR, up to 1,688 kilotons VS·annum^{-1} for predominantly TMR. This could then increase CH_4 and energy yield for PMR or TMR to a hypothetical CH_4 yield of 163,060,305 and 337,528,680 m^3_N methane per annum, respectively. It is important to note that for these calculations, generalised B_0 of 200 $LCH_4 \cdot kgVS^{-1}$ and VS excretion of 4.5 kg of VS per head were used across all the production systems, and only the proportion of VS captured was altered. This was done to allow a coherent comparison with the theoretical estimate by Tait et al. (2021a). Under consideration of differences in specific milk yields for manure excretion, the range is expected to further increase. Importantly, these differences in CH_4 yield could also translate into increased fugitive methane emissions from manure management in TMR systems if robust recovery technologies are not implemented.

7.3.1. Impact of production system with conventional manure management on fugitive methane emissions and recovery potential

This section provides a scenario, based on an average-sized Australian dairy herd with conventional manure management, such as manure storage in uncovered anaerobic lagoons. This will provide a comprehensive understanding of the impacts of production systems on residue excretion, and capture rates for final methane emissions/recovery potential. The daily manure excretion of 300 cattle was determined by the simplified approach of Nennich et al. (2005), with milking yields of 16.5 kg, 22.5 kg, and 30 kg for PB, PMR, and TMR, respectively.

The corresponding VS amount was calculated by using the averaged VS to TS ratio of 0.726 from Chapter 7. This is slightly lower than reported by Birchall et al. (2008) with 0.8. Manure capture rates for PB, PMR, and TMR systems were 15.5%, 34.3% and 71%, respectively (Chapter 4) and comparable with those stated by Dairy Australia and Agriculture Victoria (2023). Production-specific B_0 values from Chapter 6 were used to determine the resulting methane yields. Under consideration of the MCF (Chapter 2.4.1. Table 2), fugitive methane emissions were calculated, and the entire process was graphically demonstrated in a Sankey diagram (Figure 8).

VS voided on pastures was observed to have a minimal effect on total methane emissions PB and PMR systems, accounting for 25.6% to 10.7%, respectively. For TMR systems, the VS that was not captured was assumed to be included in solid bedding material, suitable for solid storage, and only responsible for an estimated 2.1% of total excretion. The amount of VS captured in uncovered holding ponds is the primary source of fugitive methane emissions in dairy production systems, and these emissions tend to increase with system intensification. Under consideration of the same herd in different production systems, PB releases 5 times and PMR 2.5 times less methane into the atmosphere than highly intensified TMR systems. This is important because, with the increased emissions, the energy recovery potential also increases, highlighting the importance of effective recovery technologies such as solid-liquid separation and AD. This is especially important when the dairy sector intensifies.

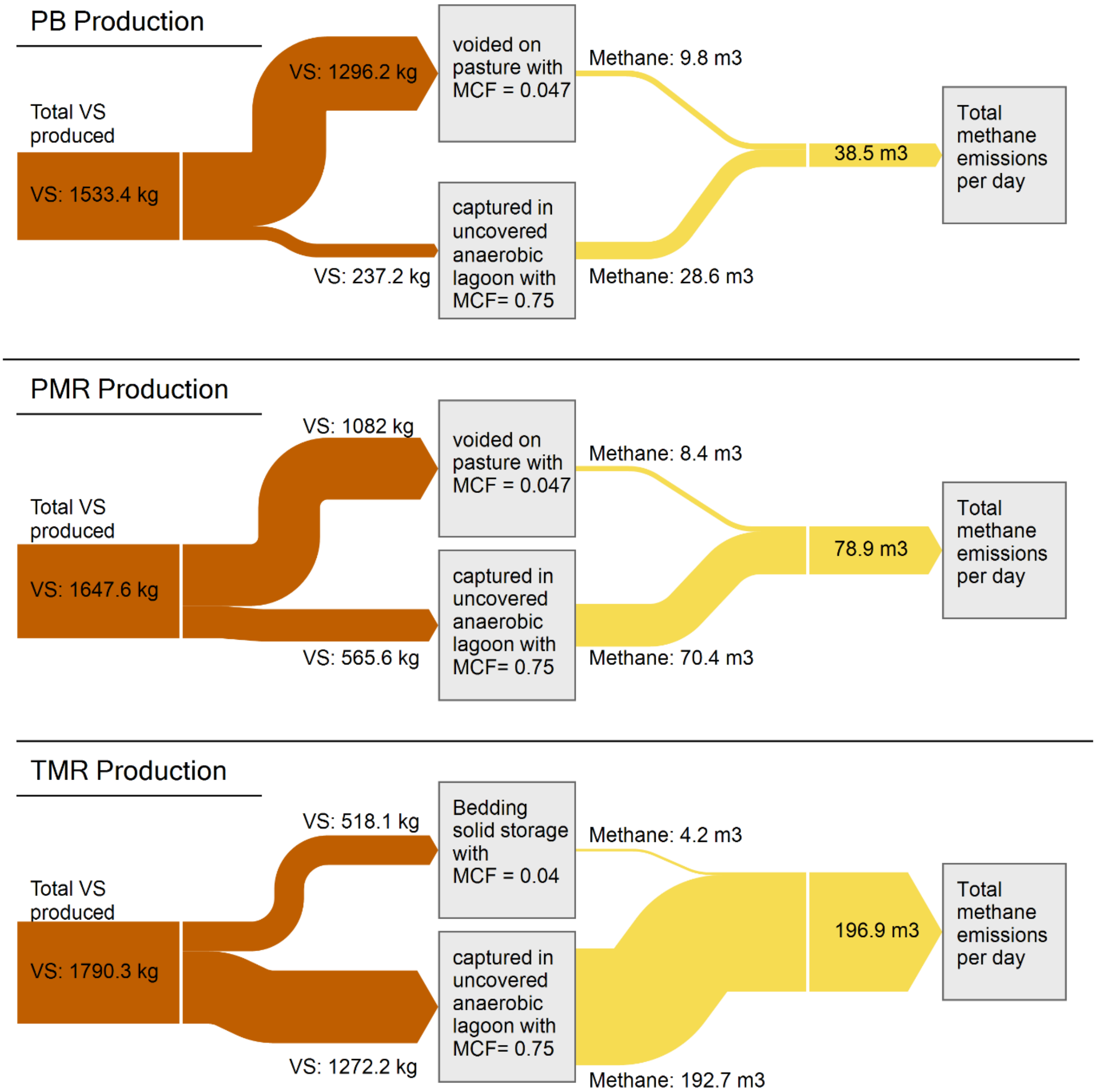

Figure 7 Sankey diagram of fugitive methane emissions and energy potential from different production systems on a daily basis

7.3.2. Solid liquid separation for heavily diluted effluent and associated fugitive methane reduction

The implementation of solid liquid separation in manure management not only facilitates transportation and flexibility of nutrient application but also reduces fugitive methane emissions from uncovered anaerobic lagoons. In order to demonstrate the impact of the Z-filter on fugitive methane emissions in heavily diluted effluent from PB and PMR systems, Figure 9 shows the VS and methane flow in a Sankey diagram. Therefore, the VS removal efficiency of the Z-Filter (33.4 %) from Chapter 5 was applied to both systems. Chapter 4 highlighted the insignificant differences in effluent characteristics of diluted effluent across different production systems, suggesting its suitable implementation across different production systems. For the calculation, it was assumed that the specific B_0 values are identical for the solid and liquid fractions of the separation process. This assumption seems reasonable, considering the more detailed experience at Farm 1 from Chapter 5 and Chapter 6.

Solid liquid separation reduces fugitive methane emissions in PB systems by 24% compared to conventional manure management, as demonstrated in Figure 9. Due to the differences in specific methane yields (161 $L_{CH4} \cdot kg_{VS}^{-1}$ for PB and 166 $L_{CH4} \cdot kg_{VS}^{-1}$ for PMR), the methane reduction for PMR systems can be even further reduced by 29 % when a separator is installed. The chemically enhanced separation with the use of flocculant increases the VS removal to 85%, therefore reducing fugitive methane emissions to approximately 70% to 74% from uncovered anaerobic lagoons for PB and PMR systems, respectively (Figure 10).

A further advantage of effluent separation is the facilitated transportability of the solids due to the reduced water concentration in the solids. The relatively small mass (Chapter 5) contains an elevated VS content and therefore methane yield, making the solid fraction an ideal candidate for biomass transportation to a centralised co-digestion facility (Wang et al., 2012). The liquid fraction might be further converted into energy using an USAB.

TMR systems were not included because the heavily diluted effluent from the milking operation is predominantly used to dilute the barn slurry to facilitate transportation by pump and pipe equipment. Therefore, making the Z-filter for TMR systems redundant

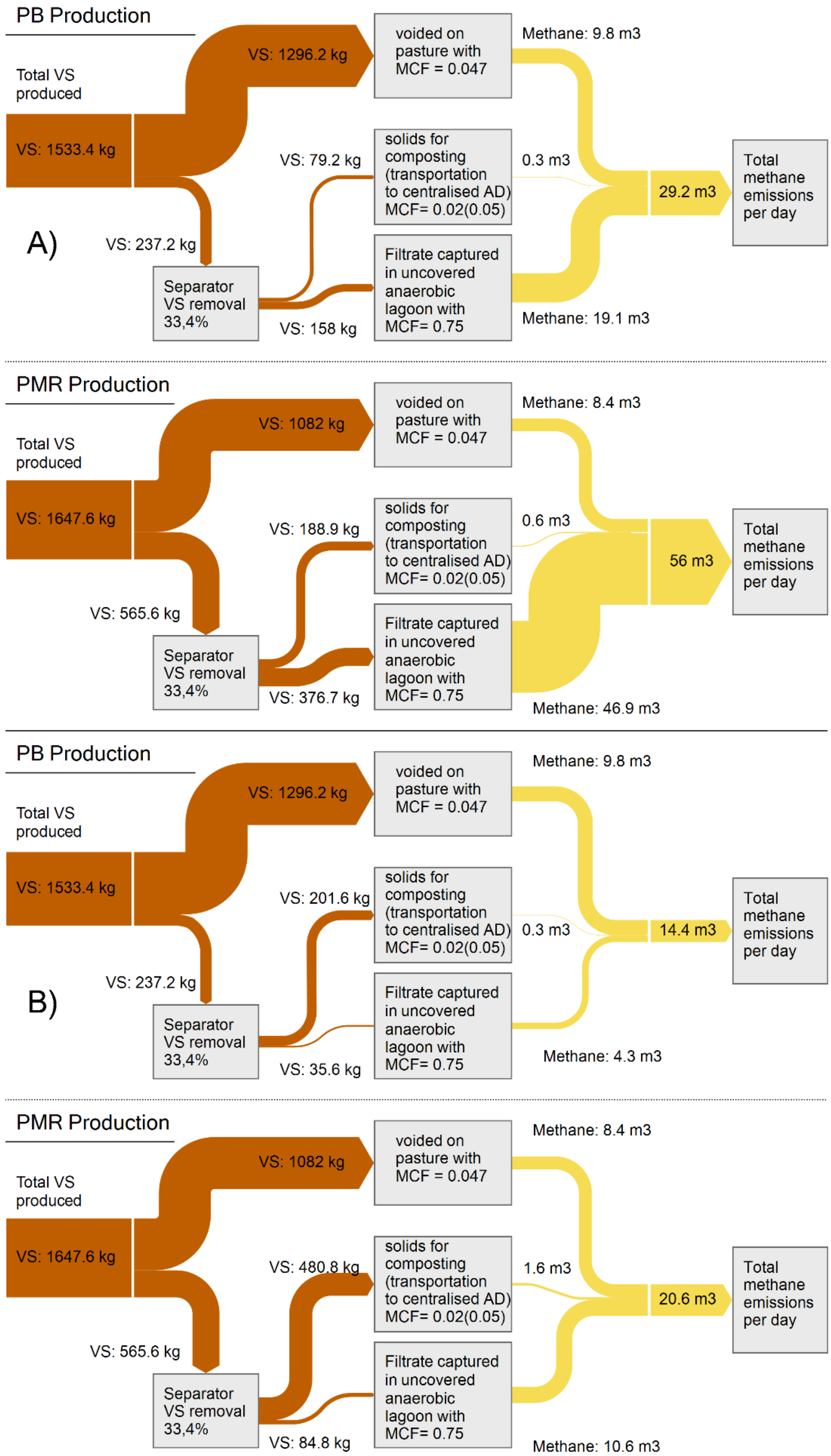

Figure 8 Sankey diagram with the implementation of solid liquid separation A) without and B) with flocculant for heavily diluted effluent on a daily basis

7.3.3. Anaerobic digestion implementation for energy recovery

The integration of AD technology into manure management is one of the most efficient technologies to reduce the carbon footprint of dairy manure management (Belflower et al., 2012). Additionally, AD is a key component in achieving a closed-loop system on dairy farms through energy recovery. However, it is important to consider effluent characteristic to identify suitable technology. Furthermore, the VS capture rates and resulting methane yields determine whether methane can be captured and converted into heat or electricity. Therefore, it is important to understand that efficiencies for only heat production are significantly higher than for combined heat (80%) and power stations (35% for heat and 35% for electricity)(Gur, 2016). Utilizing biogas for water heating or electrical power production would not only reduce dependency on fossil fuels but also contribute to direct and indirect carbon emission reductions. This would enable dairy farmers to earn ACCUs, which can provide an additional financial income stream. In order to understand suitable technology implementation, their energy recovery potential, and carbon abatement, Figure 10 shows the impact of practical AD implementations on energy production and fugitive methane emission reduction.

In all investigated PB farms, the total amount of captured methane would not justify the investment into a combined heat and power station. However, captured methane with CEP can be combusted for thermal energy production. In the case of a 300-head herd in a PB system, the captured methane could be converted into 285 $kWh \cdot d^{-1}$ of thermal energy, which is equivalent to heating 3 kL of water up to a temperature difference of 60 Kelvin. Hot water is required in every dairy for intensive cleaning and to substitute fossil fuels, which are used for thermal energy generation. Utilizing biogas for water heating would not only reduce dependency on fossil fuels but also contribute to direct and indirect carbon emission reductions.

A covered anaerobic lagoon can reduce daily methane emissions in PB systems by 70% from 38.5 m^3 (Figure 8) to 11.72 m^3 (Figure 10).

PMR systems are more diverse than the other production systems because the time of cattle on feedpads varies as well as the cleaning methods used. There are substantial changes in technology selection based on effluent characteristics resulting from different cleaning strategies (scraped, hose or floodwash). However, in this thesis, the focus was predominantly on dilute effluent, and therefore a feedpad with water cleaning was assumed for the calculations. CEP for these PMR systems is able to capture 89 m^3 of methane, which can be converted into 306 kWh per day of thermal and electrical energy. This is equivalent to a constant power output of 13 kW, which needs a side-specific assessment to determine if the CHPS investment can be justified. Alternatively, the captured methane might be destroyed or converted into thermal energy, as suggested for PB systems. The potential emission reduction is 83% even more significant than for PB systems, highlighting the increased recovery efficiency of intensified production systems.

Finally, TMR systems emit the most methane into the atmosphere when AD technology is not implemented (Figure 8). This is because of a higher specific methane yield as well as increased manure capture. However, this is also the reason why AD integration into those systems is most beneficial for energy recovery (Figure 10). For the simulated 300 cattle herd, a continuous power output of 35 kW was predicted for both electrical and thermal energy generation. Consequently, methane emissions can be reduced by 91% through the implementation of AD. Interestingly, the daily methane emissions from TMR systems are only 31% higher than for PB systems, assuming energy recovery by AD. Nevertheless, the energy recovery is 1,676 $kWh \cdot d^{-1}$, approximately 13.5 times higher than for PB systems. Understanding these environmental impacts and recovery potentials might suggest mandatory integration of circular technology into highly intensified dairy production systems.

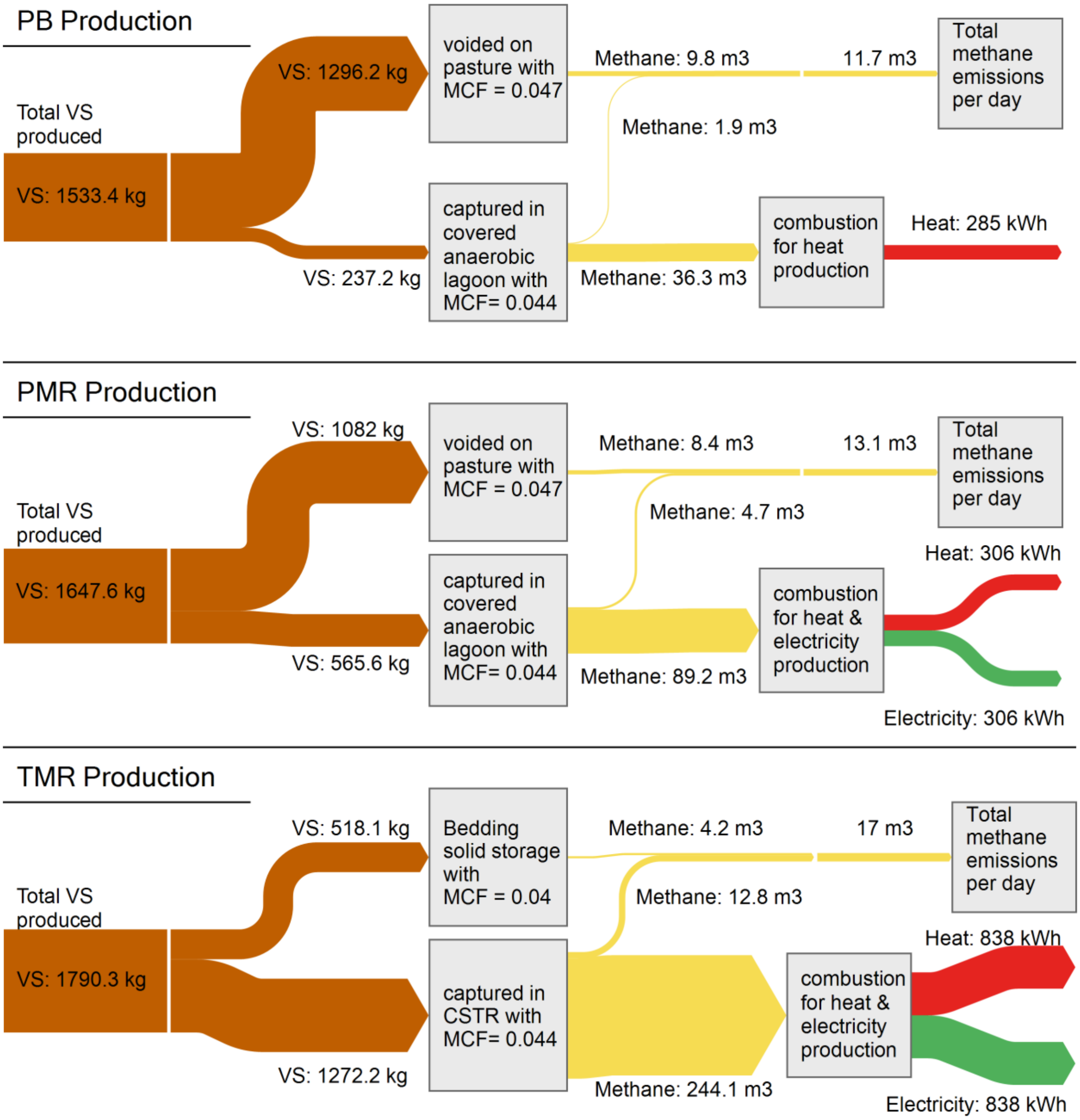

Figure 9 Sankey diagram of AD implementation for daily energy recovery

7.4. Research limitations

Understanding research limitations is important, to maintain the integrity of findings and informing future research directions. Therefore, the identified limitations are listed below.

Sample Variation: Commercial operations contain a level of uncertainty because sample collection was conducted during operation and a broader variance than under laboratory conditions was expected.

However, cross comparison with predicted nutrient and DM concentrations confirmed a representative sampling.

Full-Scale Implementation: The Z-Filter technology was tested in a single commercial context, which may not reflect performance across different commercial operations and production systems. However, the detailed investigations of nutrient concentrations from different production systems indicate its suitability.

Statistical significance of correlations: Correlations indicate a relationship between two variables but do not prove that one variable causes the other to occur. This indicates a need for confirmation by further research.

B_0 Values: The B_0 values are specific to Australian conditions, limiting their applicability in different climatic or operational conditions. While in this thesis no significant differences between production systems were observed, a broader data set might provide more clarity.

Constraints in Methodology: This thesis uses predominantly numerical data (quantitative analysis) for its conclusion. The inclusion of non-numerical data, such as interviews with farmers or observations (qualitative methods), could provide a more detailed understanding for its implementation.

Environmental Variables: Seasonal fluctuations in nutrient concentrations and methane potentials were not addressed in detail. Although samples were collected over a time of 2 years, seasonal fluctuations, might be addressed in future work.

Policy Context: This thesis was primary written, to clarify circular economy potential and option for the dairy industry. However, policy frameworks are superficially addressed but policy suggestions can be sensitive, and this thesis was conducted to inform policymakers about the benefits of on farm circular economy. However, a thorough investigation of the existing frameworks could provide greater clarity regarding which regulations should be implemented or stimulus provided.

7.5 Conclusion

The thesis contributions are multidimensional, but overall addressing current challenges in dairy manure management for an on-farm circular economy. The first part provided a detailed understanding of effluent characteristics from different dairy production systems present in Australia. The lack of significant differences in nutrient concentrations of dilute effluent, might suggests that nutrient recovery strategies can be efficient in different production systems (assuming TS concentration are suitable for recovery technology). However, total nutrient capture rates in TMR systems were much higher than in pure PB systems.

The second achievement was the first Z-Filter technology implementation on a large-scale commercial dairy operation for dilute effluent treatment. Thus, overcoming the limitations of earlier lab-scale investigations. This study discovered that the use of flocculant increased removal efficiencies and the addition of lime enhanced P recovery up to 90% through calcium phosphate precipitation. This is a promising option to reduce the potential of Phosphorous entering local waterways.

Finally, the biochemical methane potential of manure residues across the Australian dairy farming industry was investigated for the first time. This provides the first comprehensive understanding of B_0 values of grazing cattle and demonstrates that existing default values are likely overestimating carbon emissions. Consequently, it was highlighted that a generalised approach to predict energy recovery using AD technology might not be applicable across the entire industry. Therefore, highlighting the need for individualised solutions and accounting for diverse production systems including herd sizes, as demonstrated by Sankey diagrams.

This thesis addressed key challenges for a circular economy on dairy farms and highlights opportunities for potential recovery strategies. This can help policymakers and farmers to implement more sustainable manure management strategies for final nutrient and energy recovery, in a circular economy approach.

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APPENDIX A

Supplementary material from Paper 2/ Chapter 5

Nutrients and organic matter recovery from dilute livestock manure using novel modular solid-liquid separation

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Supplementary Material

Table S1 Measured characteristics of liquid samples collected during the trial, presented as average values ± 95% confidence intervals. Values shown are for total elemental concentrations.

Samples	Al [$mg \cdot L^{-1}$]	Cu [$mg \cdot L^{-1}$]	Fe [$mg \cdot L^{-1}$]	Mg [$mg \cdot L^{-1}$]	Na [$mg \cdot L^{-1}$]	S [$mg \cdot L^{-1}$]	Zn [$mg \cdot L^{-1}$]
Typical influent (before lime addition)	n=10 5.6 ± 1.8	n=10 0.2 ± 0.1	n=10 11.3 ± 2.5	n=10 75.7 ± 9.6	n=9 133.5 ± 15.9	n=10 25 ± 4.1	n=10 1.0 ± 0.2
No Treatment							
	n=6	n=6	n=6	n=6	n=6	n=6	n=6
Influent	4.8 ± 1.4	0.2 ± 0.1	10.9 ± 2.8	76.2 ± 11.8	136.2 ± 23.4	25.3 ± 4.7	1.0 ± 0.2
Filtrate	4.5 ± 1.3	0.2 ± 0	10.1 ± 2.5	74.9 ± 11	136 ± 23.4	24.2 ± 4.6	0.9 ± 0.2
5% Floc							
	n=4	n=4	n=4	n=4	n=3	n=4	n=4
Influent	6.9 ± 5.7	0.3 ± 0.2	11.8 ± 7.9	75 ± 29.1	128.1 ± 50.3	24.5 ± 12.6	1.1 ± 0.6
Filtrate	0.3 ± 0.2	0.1 ± 0.1	0.5 ± 0.3	64.5 ± 26	127.1 ± 47.3	14.2 ± 6.8	0.1 ± 0.1
5% Floc & Lime							
	n=6	n=6	n=6	n=6	n=3	n=6	n=6
Influent (after lime addition)	7.4 ± 3.5	0.3 ± 0.2	12.8 ± 4.7	83.8 ± 15.3	127.1 ± 31.9	28.9 ± 8.1	1.1 ± 0.3
Filtrate	0.3 ± 0.4	0 ± 0	0.5 ± 0.6	60 ± 13.2	129.1 ± 43.7	13.9 ± 3.2	0.1 ± 0.1
3% Floc & Lime							
	n=6	n=6	n=6	n=6	n=3	n=6	n=6
Influent (after lime addition)	6.3 ± 2.6	0.2 ± 0	10.6 ± 4.2	76.7 ± 13.7	116.7 ± 25.9	27.5 ± 7.1	0.9 ± 0.3
Filtrate	1.4 ± 1.5	0 ± 0	2.4 ± 2.6	58.6 ± 13.6	114 ± 9.9	19.4 ± 3.7	0.3 ± 0.2

n = number of samples for which corresponding mean values are given.

Cost feasibility

To identify the cost feasibility of using the Z-Filter for solid-liquid separation of dairy effluent, a simple payback period was calculated (i.e. Investment divided by net undiscounted financial benefit). No allowance was made for depreciation-related tax benefit. The methods and results are summarised in Table S2.

The initial purchase and installation costs were an estimated AUD200,000. Operational costs were based on 300 L min^{-1} influent flow to the Z-Filter (2,231 hours per annum of Z-Filter operation for 110 kL d^{-1} of influent), and an electricity cost of 16.84c kWh^{-1} (current pending rate).

The estimated costs of both flocculant and lime used to maximise P capture and thereby minimise P in stored effluent was estimated based on use of these chemicals only during the four wettest winter months when it is likely to be less appropriate to land-apply effluent. During the remainder months of the year, it was assumed that no flocculant or lime was being used, and that filtrate was simply land-applied after manure fibre removal by the Z-Filter. Nominal replacement costs were included for the filter mesh and other internal parts (based on trial observations).

The economic benefits of the separation included:

1. enabling the reuse of manure nutrients being land-applied as filtrate to offset imported synthetic fertilizer use. This was valued at nominal fertiliser costs(Fertilizer Australia, 2021; Tait et al., 2020; Trading Economics, 2021). It is noted that fertilizer prices are volatile, with the price of nitrogen fertiliser approximately doubling during the investigation period. However, for a more conservative estimate here, reported prices at the start and end of the trial were averaged and this average value was used.
2. the value of composted solids fraction being used instead of and thereby displacing commercial compost that had previously been purchased from offsite to maintain soil productivity at the site. For this, historic purchase costs for commercial compost provided by the trial site owner were used. These costs included AUD90,500 $annum^{-1}$ supply charge and AUD54,750 $annum^{-1}$ of transport costs. Because the farm was in a remote location, we opted to not include transportation cost for compost delivery as a financial benefit, and instead assumed that these are largely offset the cost associated with composting the solids fraction on farm. The nutrient value in the solid fraction was not counted in the financial benefits, because it was assumed that

commercial compost previously purchased and imported on to the site would have had a similar nutrient content.

To assess labour costs, a nominal cost of AUD45 h^{-1} was applied, reflective of specialist labour costs in Australia. The grade of automation of the Z-Filter would heavily influence labour requirements for maintenance and operator intervention, with an increasing level of automation able to reduce labour requirements. To evaluate the effect of this on high-level economic feasibility, two hypothetical scenarios were compared. *Hypothetical Scenario A* considered a need for continuous operator intervention, if little to no additional automation was added to the Z-Filter, and if it would not be possible for a dedicated Z-Filter operator to also perform other parallel on-farm duties to optimise overall labour benefits. This therefore represents a hypothetical worst-case scenario, primarily driven by the perceived need for a high level of operator presence to ensure a functional and safe separation. Continuous operator intervention would amount to an estimated 2,200 hours $annum^{-1}$ at the above operational throughput of the Z-Filter. *Hypothetical Scenario B* considered the opportunity to significantly reduce operator intervention, by instead implementing significant additional automation. For this, the assumption was that an operator would still be required for approximately 10 hours per week (520 h $annum^{-1}$) to provide essential cleaning and maintenance. A nominal cost allowance was included for the additional automation required for this scenario, estimated at AUD20,000. This would represent the best-case scenario.

Results and Discussion

For hypothetical Scenario A – Continuous operator intervention - labour costs amounted to an estimated AUD99,000 $annum^{-1}$, which together with the additional operating expenses and savings/benefits above, would have resulted in a negative cashflow of ~-AUD11,547 $annum^{-1}$. This indicated that for this scenario, the Z-Filter operation would not be economically attractive. For hypothetical Scenario B – Minimum required operator intervention - labour costs amounted to an estimated AUD23,400 $annum^{-1}$, which together with the additional operating expenses and savings/benefits above, gave an estimated simple payback period of approximately 3.5 years.

This indicated that the Z-Filter operation could be financially attractive if the level of operator input required can be minimised.

Table S2 Parameters and results of the preliminary financial benefit analysis

Costs	**AUD**
Initial investment	200,000
Total operational expenses - Scenario A - Estimated additional cost for added automation	20,000
Operational expenses	**AUD annum^{-1}**
Flocculant (@ \$8.4 kg^{-1})	27,200
Lime	700
Filter mesh replacement (2 x AUD3000 each)	6,000
Other parts replacements	1,000
Labour costs Scenario A (2,200 h annum^{-1} @nominal AUD45 h^{-1})	99,000
Labour cost Scenario B (520 h annum^{-1} @nominal AUD45 h^{-1})	23,400
Electricity (22kWe; 2,200 h annum^{-1})	8,300
Total operational expenses - Scenario A	142,200
Total operational expenses - Scenario B	66,600
Return/Savings	**AUD annum^{-1}**
Nutrients value in filtrate	
Summer (240 d)	
N (6 tonnes annum^{-1} @ AUD2.32 kg^{-1})	13,887
P (1.6 tonnes annum^{-1} @ AUD5.33 kg^{-1})	8,479
K (4 tonnes annum^{-1} @ AUD1.75 kg^{-1})	7,051
Winter (120 d)	
N (2.3 tonnes annum^{-1} @ AUD2.32 kg^{-1})	5,276
P (0.2 tonnes annum^{-1} @ AUD5.33 kg^{-1})	1,052
K (2.5 tonnes annum^{-1} @ AUD1.75 kg^{-1})	4,395
Total nutrients value in filtrate	40,139
Solids fraction displacing compost	
Summer (240 d, 221 tonnes solids fraction @ AUD153.62 tonne^{-1})[1]	33,953
Winter (120 d, 368 tonnes solids fraction @ AUD153.62 tonne^{-1})[2]	56,563
Total compost and nutrients value	130,655
Simple net cash flow: Savings/benefit minus costs:	**AUD annum^{-1}**
Scenario A) with continuous operator intervention	-11,547
Scenario B) fully automated and with minimum operator intervention	64,053
Simple payback period	
Payback period Scenario A	not applicable
Payback period Scenario B	3.4

[1] Solids fraction without flocculant

[2] Solids fraction with flocculant and lime

Bench-scale coagulation-flocculation testing

Methodology

A smaller bench-scale test set was performed to provide a deeper investigation of the effects of pH and lime pre-treatment on P-removal. For this, 100 mL of influent collected from the trial site was mixed with lime, CaCO3, CaCl2 or KOH, in pre-determined amounts in a beaker over a magnetic stirrer plate for 10 minutes at 500 rpm. After this initial mixing, 0.5 mL of pre-diluted flocculant (Section 2.1) was added with a syringe whilst the mixture continued to be stirred at 500 rpm for an additional 2 minutes. The mixing was stopped, the resulting mixture passed through a double-folded cut-out piece of the Z-Filter filter mesh, and the solids fraction gently squeezed to encourage further filtrate to drain through the mesh. The filtrate produced in this way was analysed for total elements (Section 2.6).

Zeta potential tests were performed to investigate the influence of Ca on the electronical surface charge of colloids in the influent. For this, samples were prepared by passing influent through a 500 µm sieve to remove fibre components, and then measuring Zeta potential on this screened influent as-is (condition 1), or after adding lime to a pH of 9.2 (condition 2), or after adding CaCl2 without any pH adjustment (condition 3), with the amount of Ca added here matching the amount of Ca amount added as lime in test condition 2, or after adding concentrated sodium hydroxide drop-wise to increase pH to 9.2 without any Ca chemicals (condition 4).

Results

The bench-scale test results were viewed as indicative because these tests had not been replicated like the full-scale trial. The bench-scale tests showed a similar high extent of P removal (98%) with lime and flocculant addition. The addition of CaCl2 and KOH, followed by flocculant, showed slightly less P-removal at 83% and 72%, respectively. The addition of CaCO3, followed by flocculant, showed a relatively low P-removal at 30%, which was only marginally higher than when only flocculant was dosed (26%).

The zeta potential measurements showed a consistent negative surface charge of colloids in the effluent being analysed, averaging a value of -20.5 mV. Lime addition, NaOH addition to pH 9.2, and Ca addition as CaCl2, did not greatly change the measured zeta potential, specifically being -19.1, -22.6 and -21.0 mV, respectively.

The zeta potential measurements indicated that any dissolved Ca had minimal effects on screening of the negative surface charge of colloids in the influent (Section 3.2); albeit that zeta potential generally tends to become more negative with an increasing pH, so that the dissolution and screening of surface charge by Ca via lime could have been counteracted by the effect of increasing pH. A plausible mechanism for the increased P removal with lime and flocculant addition was thought to be the precipitation of calcium minerals (Monballiu et al., 2018; Rugaika et al., 2019) catalysed by added Ca, and by elevating pH which increases the proportion of free phosphate (PO4-3) (Mbamba et al., 2015). This appeared to agree with the limited bench-scale tests (Section 3.2) where increasing pH via KOH followed by flocculant addition (no Ca addition) still showed moderate P removal, potentially due to background soluble Ca precipitating with P at the elevated pH.

The addition of garden lime in the bench tests showed a lower level of P removal than lime (Section 3.2) and in the field trial was found to be operationally problematic, because of poor solubility and settling out of the garden lime in the Z-Filter feed tank despite the continuous overhead mixing (Section 2.3)

APPENDIX B - CARBON EMISSIONS FROM SEPARATED DAIRY SOLIDS AND THE EFFECTS OF C-PAM

This chapter relates to the experiments used to investigate the greenhouse gas emissions from composted solid fractions, that were obtained with the device described in Chapter 5.

Additionally, the levels of earthworm toxicity in raw and composted solid fractions are assessed to explore the potential of composting to ease the toxicity of C-PAM. The mitigation of toxicity by this biochemical process is of interest for agricultural waste management (Chapter 2). Particularly considering the elimination of negative effects of compounds such as C-PAM. It can play a relevant part to control separation efficiency (Chapter 5).

It is necessary to note that due to technical complications in the experimental composter infrastructure that lied beyond the author's control, the experiments failed. Nevertheless, the experimental procedures for the unsuccessful experiments are yet described in detail to potentially facilitate future investigations on the important topics of this chapter. While the results in this part are preliminary, they may also inform further research.

B.1. Methodology

B.1.1. Materials

The dairy solids used for composting were sourced and sampled from the commercial dairy farm that is described in Section 3.3. Three different types of dairy manure solids (Table 1) were sampled, each time weighing approximately 200 kg. Afterwards the material was immediately shipped to the University of Queensland for composting analysis. To ensure optimal preservation, these samples were stored at 4ºC for no longer than about 3 weeks prior to the test.

Prior to the experiment, the samples were acclimatised at room temperature for about 5 days to allow for microbial activation.

B.1.2. Experimental set up and design

For the designated compost period of 30-45 days, 60-liter customized compost chambers (Figure 1) were operated. The essentially important parameters for composting, temperature and aeration, were monitored and controlled. In order to match specific processing conditions, for instance achieving temperatures in the range of 45-60°C, to facilitate faster microbial turn-over (degradation), these parameters were adjusted manually. The oxygen needed in composting was supplied by metered forced aeration drawing in ambient air. The experimental composting chamber consists of two vessels, an interior one, designated for the placement of composting material that sits within an outer vessel (Figure 1). The space between the vessels is filled with insulating material, thereby imitating the insulation characteristics observed in sizable compost piles. To enable control of thermal conditions that imitate the heat produced by microbial activity during the decomposition process, the inner reservoir is equipped with a heating coil positioned around its outside. Spatial temperature readings may be obtained by utilising dual temperature sensors situated at both the edge and center of the chamber. This configuration allows to capture the intrinsic thermal gradient present inside the compost piles. The integrated air supply and exhaust system serves two purposes simultaneously. It regulates the aerobic conditions within the compost and enables constant or temporary monitoring of exhaust gases, which is essential for understanding the biochemical reactions and emissions taking place.

The exhaust air was lead through an outlet tube connected to the compost chambers. It featured a T-piece, that could be attached to a sampling tube to extract the exhaust air. The T-piece was also connected to a Masterflex pump using a flexible tube.

The regular outlet was sealed, ensuring an exclusive pathway from the composting chamber to the sample location.

Figure 10 Schematic and actual view of small-scale composting reactors

Gas sampling

The gas samples were systematically collected over a 30-day period, with the sampling frequency being adjusted to obtain more detailed data during the first two weeks. While samples were taken daily during the first two weeks, the repetition changed to every other day during week three, and once every three days in the final week. Throughout each sampling event, the forced aeration was put on hold to ensure accuracy when sampling. The outlet needed to be properly closed and to prevent a potential backflush with ambient air. To extract gas at a rate of $1L\cdot min^{-1}$ from the compost chamber's headspace, the The Masterflex pump (See above) was accordingly calibrated. The needle of a 25-mL gas-tight syringe was inserted into the open tube end, that was located on the discharge side of the pump, ensuring no dilution with ambient air. The gas sample then was drawn by the pump through the open tube end. At a steady rate of 5 mL every 10 seconds, samples were drawn until a total of 25 mL, accumulated through these multiple smaller gas volume collections. Through these confugurations, the sampled gas effectively represented an aggregate grab sample of the headspace gas in the composter.

Once collected, the sampling material was promptly injected into pre-evacuated exetainers (20mL) and stored at a temperature of 4^{o}C. It was intended to be analysed by the main analytical laboratory at Queensland University of Technology using gas chromatography for CH_4, CO_2, and N_2O (not carried out due to failed experiment).

Table 8 Reactor conditions and analysis

Reactor	Solid samples	Temperature [^{o}C]	Aeration [L/min]	Chemical characteristics	Gases
1	No C-PAM	40(±5)	0.5	TS, VS, N, P, K, C	CO_2 CH_4 N_2O
2	No C-PAM	55(±5)	2		
3	5% C-PAM	40(±5)	0.5		
4	5% C-PAM	55(±5)	2		
5	5% C-PAM & lime	40(±5)	0.5		
6	5% C-PAM & lime	55(±5)	2		

B.1.3. Earthworm toxicity test

Materials

The Earthworm acute toxicity (described in detail by Li et al. (2021a)) was tested on a raw and composted solid fraction of dairy effluent with and without adding polymer flocculant.

To conduct the earthworm test in accordance with ethical standards, the animal ethics committee at UniSQ (animal.ethics@usq.edu.au) was consulted. As earthworms are living microorganisms not declared to be animals, the committee confirmed that there were no concerns regarding their use in this experiment. Thus, the experiment did not require any ethical approval. Nevertheless, the experiment was conducted with the utmost care and attention to minimize any potential harm or stress to the earthworms.

Any use of living organisms in scientific research demands high ethical considerations to make certain that the experiments are carried out in a responsible and humane manner.

Adult Eisenia fetida (liveweight of 300 - 600mg) were acquired from an earthworm farm in Toowoomba, Queensland. They were placed in a 5L cotton bag of cow dung, which was periodically moistened with deionised water as described by Li et al. (2021a). The Separated dairy solids were sourced from a Farm in WA as described in Chapter 4.

Test procedure

The solids were tested in four replicates. Each time 500 grams of solids and 10 earthworms were carefully weighed out, documented, and placed together in a sealed plastic container with holes in the lid for oxidation. At the temperature of 20°C ± 1°C, the test continued for 14 days. The procedure was performed in an incubator lit up with continuous light, so that the worms remained in the test medium throughout the duration of the test. The mortality was assessed by emptying the test medium onto a glass tray or plate, sorting worms from the medium, and testing their reaction to a mechanical stimulus at the front end. At the end of the 7-day assessment, the weighed worms were placed back in the test container and observed for any behavioural or pathological symptoms, which when noted were reported. Lastly, the moisture content of the test medium was assessed and reported at the end of the test.

B.1.4. Analytical Procedure

In alignment with the Standard Methods procedure 2540G (APHA) TS and VS were measured. Carbon and nitrogen quantifications in pre-dried (70°C) solid fraction samples were facilitated using the Elementar Vario Macro Combustion Analyzer (Hanau) based on the Dumas method. To determine the elemental composition, including P and K, the inductively coupled plasma optical emission spectroscopy (ICP-OES) was employed, using the PerkinElmer Optima 5300DV (PerkinElmer Corp., Norwalk Ct, USA). Accurate measurements, as described in detail in Chapters 4 and 7 were achieved by adhering to procedures, involving specific pre-digestion protocols and dilution methods.

B.2. Results

B.2.1. Raw material characteristics

The separated dairy manure solids were examined for the characteristics, that are presented in Table 2. Dairy Solids 1 (no C-PAM) was analysed to have a 92% moisture content with a total organic carbon (TOC) and total carbon (TC) content of 45.5%. This material contained 1.13% nitrogen and displayed a carbon/nitrogen ratio of 40. However, the incorporation of a 5% C-PAM in Dairy Solids 2 resulted in a notable decline of the moisture content to 82%, as well as a reduction in total organic carbon (TOC) and total carbon (TC) to 38.6%. The nitrogen levels conversely saw a rise to 2.61%, reflecting increased nitrogen recovery to the solids fraction (Chapter 4), and thereby leading to a reduction of the carbon-to-nitrogen (C:N) ratio of 15. Dairy Solids 3 was added with a combination of 5% C-PAM and lime, resulting in a marginal elevation in moisture content to 84%. Additionally, there was an increase in the levels of total organic carbon (TOC) and total carbon (TC) to 41.1%. Furthermore, the nitrogen concentration reached 2.68%, while the carbon-to-nitrogen (C:N) ratio remained constant at 15.

The separated dairy solids samples were not considered too wet in their native form for composting, which is the reason why no bulking agent such as straw needed to be added. The results indicate that the use of chemical treatments, specifically C-PAM, can significantly modify the physiochemical characteristics of dairy solids.

Table 9 Characteristics of separated dairy solids

Parameter	Dairy Solids 1 - No C-PAM	Dairy Solids 2 - 5% C-PAM	Dairy Solids 3- 5% C-PAM + Lime
Moisture Content (%)	92	82	84
Total Organic Carbon (%)	45.5	38.6	41.1
Total Carbon (%)	45.5	38.6	41.1
Total Nitrogen (%)	1.13	2.61	2.68
Carbon/Nitrogen Ratio	40	15	15
Estimated Organic Matter (% OM)	77.3	65.5	69.9

All analytes are measured on a wet basis

B.2.2. Composting observations

The figure below shows the temperature profiles for the compost reactors. Due to technical problems with the temperature control, the reator temperuteur exeeded 60 degrees celsius for aerated compostingn and 45 degrees for stockpiled conditions, both for approximately 15 degrees.

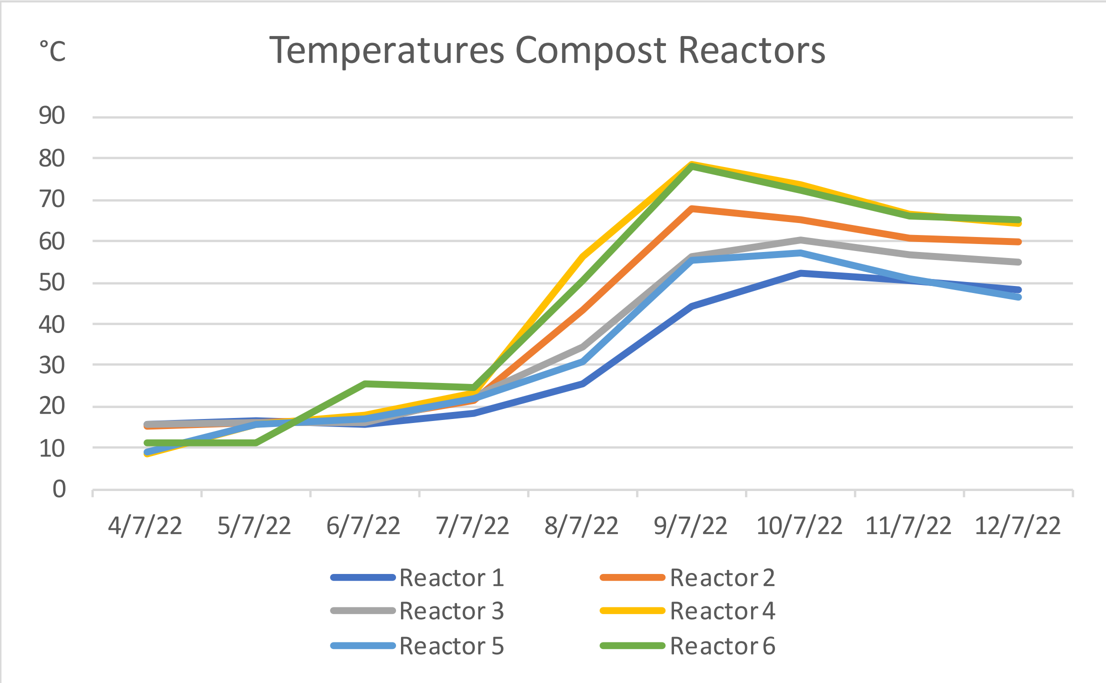

Figure 11 Averaged temperature profile during composting of separated dairy manure solids

B.2.3. Earthworm toxicity test

The earthworm acute toxicity test generated clear results. All worms survived in untreated manure (no flocculant), while the ones in the flocculant treatment died within the first week. This suggests that solids with cationic polymer flocculant directly from the solid-liquid separation process pose a significant risk to soil organisms such as earthworms. The failed experiment did not allow us to conduct the same experiment with composted solids, but it would be important to confirm the efficacy of composting in potentially reducing the adverse impacts of the flocculant.

B.3. Discussion

The objective to evaluate carbon emissions from the composting process of separated dairy residue solids, unfortunately could not be addressed in the current thesis, due to the technical issues at the compost facility. Therefore, only the side objective of the ecotoxicity and potential influence of composting on manure solids that have been treated with cationic polyacrylamide using an earthworm toxicity test could be addressed.

The results show that the death rate of earthworms in the treated separated solid with the C-PAM fraction was greater than that of earthworms in the untreated separated solid fraction. In fact, all earthworms in solids with PAM were found dead. In contrast, in the untreated material all earthworms survived and gained weight on an average basis (+0.4g/worm). These results imply that cationic polymer flocculant treatment may be hazardous to earthworms, which are essential to the ecology of the soil.

It is necessary to highlight that this study has limitations since the polymer flocculant-treated solid fraction was not mixed into agricultural soils and the composting capacity of the materials was not investigated as the focus was on the influence of composting. Further research is required to explore the possible effects on soil biota when introducing polymer flocculant-treated separated solids into agricultural soils. It is well-documented that the ecotoxicological impacts of such chemicals exhibit a dose-dependent relationship, where lower doses typically result in lower toxicity, while higher doses lead to increased toxicity. Therefore, adhering to manufacturer-recommended application rates is essential not only for optimizing waste treatment efficacy but also for minimizing potential adverse effects on soil ecosystems. This approach ensures that the application of CPAM is conducted within a framework that prioritizes environmental safety and sustainability. However, in this study the toxicity test was conducted purely on the separated solids to focus on a potential reduced toxicity of CPAM by composting.

The study's findings indicate that solid-liquid separation done with modular technology could provide improved environmental management options for dairy farmers that want to treat dilute manure effluent to utilise the organic matter and nutrients for the benefit of soil biota. However, the use of cationic polymer flocculant to enhance nutrient recovery should first be carefully evaluated to protect soil microbiota from potential negative impact. The manufacturer's recommendations for soil applications are a safe guideline.