



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% $\text{Ca}(\text{OH})_2$ 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 ($\pm 0.5\%$) 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 m³ 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 ($\pm 0.5\%$) 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

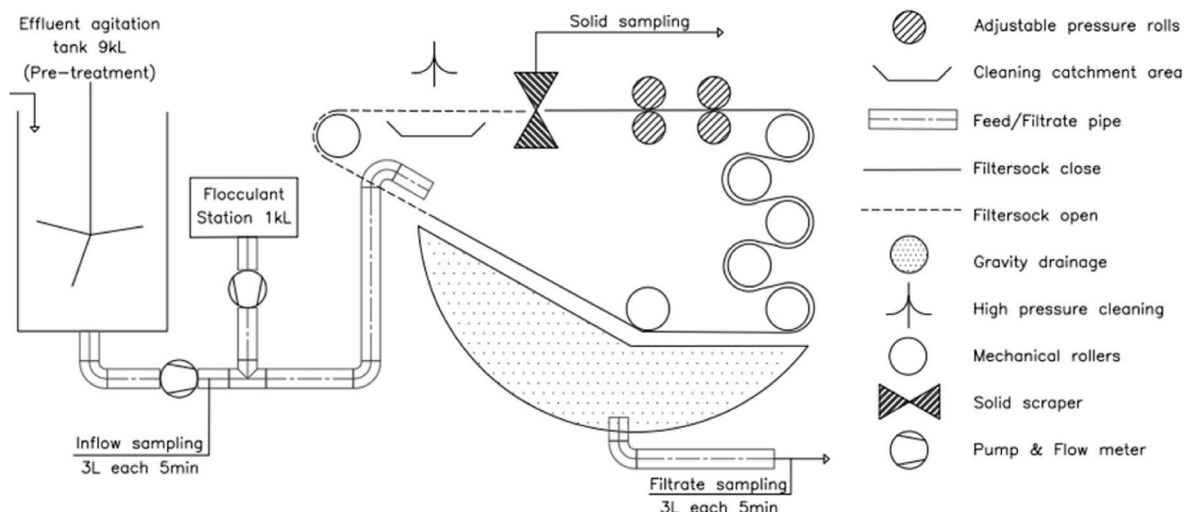


Fig. 1. Illustrated schematic of the Z-Filter with its important features.

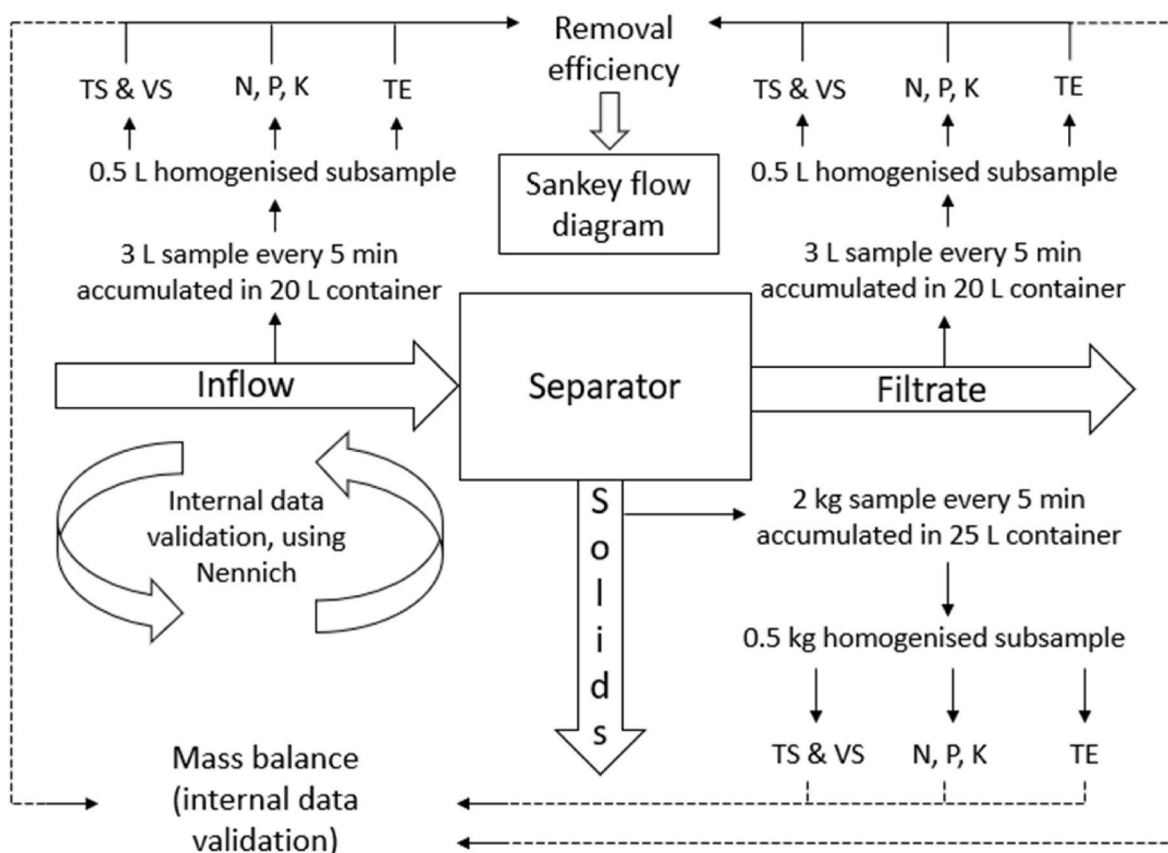


Fig. 2. Graphical summary representation of the test methodology.

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

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.

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.

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 H₂O₂ 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):

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

where $c(x)_{\text{influent}}$ and $c(x)_{\text{filtrate}}$ 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.

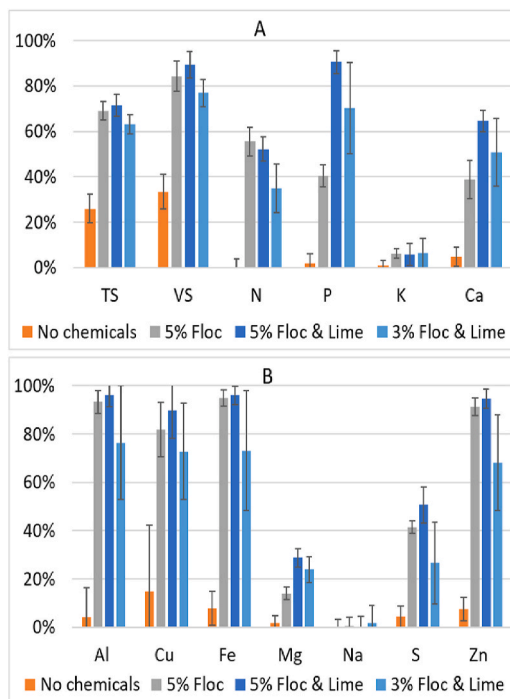


Fig. 3. Removal efficiency (R_E) (%) values (Eq. (1)) for (A) TS, VS, N, P, K, Ca, and (B) Al, Cu, Fe, Mg, S, and Zn. Values given are calculated means in replicates and the error bars are estimated standard deviations.

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).

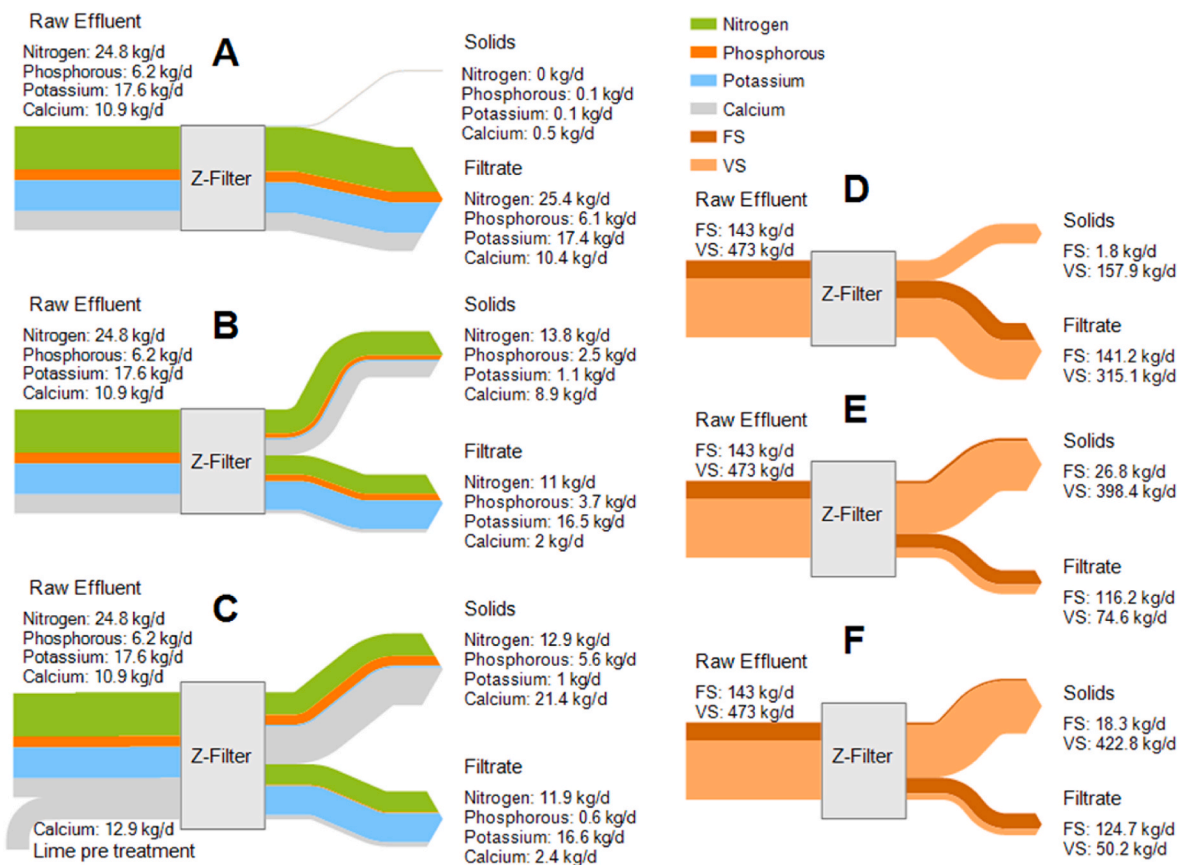


Fig. 4. Average estimated mass flows of analytes during the trial. (Fixed solids (FS) equals TS minus VS). This includes Sankey diagrams (A, D) without any lime or flocculant; (B, E) with 5% flocculant only; and (C, F) with both lime and 5% flocculant.

Table 3

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

TS [% wet mass basis]	VS [% wet mass basis]	VS/TS ratio [-]	N [$\text{g}\cdot\text{kg}^{-1}$]	P [$\text{g}\cdot\text{kg}^{-1}$]	K [$\text{g}\cdot\text{kg}^{-1}$]	Mg [$\text{g}\cdot\text{kg}^{-1}$]	Ca [$\text{g}\cdot\text{kg}^{-1}$]
No Lime or Flocc							
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% Flocc							
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% Flocc & 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% Flocc & 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 overflowing 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 ~ 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

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