# Repeated applications of farm dairy effluent treated with poly-ferric sulphate did not adversely affect soil phosphorus availability, P fractions and pasture response — a 4-year field plot study

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**Abstract**

**Purpose** Land application of farm dairy effluent (FDE) can cause phosphorus contamination of freshwater due to its high nutrient content especially phosphorus (P) in the animal dung. A novel FDE treatment technology has been developed that uses poly-ferric sulphate (PFS) to treat the FDE and recycle water for washing farmyard and reduce the risk of water pollution from P leaching from through the soil. It is important that the application of PFS-treated FDE (TE) does not cause any adverse impacts on soil fertility or plant growth when the TE is applied to the soil.

**Materials and methods** A multi-year field plot study was conducted to determine the effect of repeat applications of FDE and PFS-treated FDE (TE) on soil P availability, P fractionations, plant yield and nutrient uptake. Eight applications of untreated FDE, TE and water as control were applied to replicated soil plots over the period of 4 years. The soil samples were collected on 1 December 2020, and nine pasture samples were harvested during the 2021–2022 dairy milking season. Measurements included soil chemical properties, soil phosphorous fractionations, plant biomass and plant phosphorus and nitrogen uptake.

**Results and discussion** The results indicated that the majority of soil fertility indices and soil P fractions had no significant difference between the FDE and TE applications, with the exception of labile P which was significantly higher in the TE (122.7 mg kg$^{-1}$) than in the FDE treatments (103.0 mg kg$^{-1}$) at 0–10-cm soil depth and was also significantly higher in the TE (114.6 mg kg$^{-1}$) than in the FDE treatments (74.0 mg kg$^{-1}$) at 10–20-cm soil depth. Similarly, plant P uptakes and dry matter yields were also the same between the TE and FDE treatments with the average of being 54.4 kg P ha$^{-1}$ and 12.8 t ha$^{-1}$, respectively.

**Conclusions** Repeated applications of PFS-treated FDE had no adverse effect on soil P availability or plant growth when compared to untreated FDE application and had the potential to benefit soil fertility compared to control.

**Keywords** Soil phosphorous · Phosphorus fractionation · Farm dairy effluent · Land application · Pasture biomass

## 1 Introduction

Dairy farming generates substantial quantities of effluent containing urine and faeces from cattle that have been diluted with wash-down water. Many countries (such as the UK, USA, Brazil, Korea, Australia and New Zealand) have adopted land application of farm dairy effluent (FDE) as their primary method of FDE management. Land application of FDE recycles valuable nutrients, increases pasture yield and improves soil structure (Woodard et al. 2002; Bolan et al. 2004; Hawke and Summers 2006; Luo et al. 2008; Bhandral et al. 2010; Dumont et al. 2012; McDowell and Nash 2012; Ghezzehei et al. 2014; Li et al. 2014; Choi 2016; Manono et al. 2016). However, land application of farm dairy effluent can also cause pollution of freshwater bodies (i.e. rivers, lakes and groundwater), due to its high content of phosphorus (P) and nitrogen (N) (Di et al. 1999; Dougherty et al. 2004; McDowell et al. 2016; Veltman et al. 2018). Many waterways in New Zealand have high nitrogen concentrations

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(Scarsbrook and Melland 2015), but phosphate is often the limiting element for the growth of aquatic plants and algae (McDowell et al. 2009; Abell et al. 2010). Hence, to prevent further degradation of the health of waterways, it is necessary to reduce the amount of phosphate leaching that occurs from effluent application areas into rivers and lakes.

Moreover, the long-term application of organic animal waste may result in lower soil P adsorption capacity than with the application of inorganic fertilizers (Varinderpal-Singh et al. 2006; Jiao et al. 2007). At the same time, soils receiving the organic form of animal waste have a higher potential to desorb P than those soils receiving inorganic fertilizer because organic acids in soils amended with manure compete for sorption sites (Jiao et al. 2007). As such, continuous P inputs from organic sources may lead to an immediate increase in soil P pools, but P may not be directly available for plants. Repeated application of FDE can result in the accumulation of P within the soil, which can act as a source of P for plant uptake but also increase the risk of P leaching. In an intensive dairy farm system, the largest soil P surpluses generally result from land receiving effluent/manure or dung from livestock (Barlow 2003; Gourley et al. 2015). Moreover, Toor et al. (2004) demonstrated that if the inputs of the P (through FDE) remains above the soil's P fixation capacity for several years, P-fixation sites may become saturated and continued application of FDE may result in increased losses of inorganic P (Po).

Recently, a new FDE treatment technique, called ClearTech®, was created to reuse water for cleaning the dairy milking yard. In order to flocculate and settle the colloidal particles in FDE, the ClearTech® treatment system adds and mixes the coagulant poly-ferric sulphate (PFS) with FDE. In addition to significantly reducing water waste, this innovative FDE treatment technology also reduced methane ($CH_4$) emissions when treated effluent was stored in the storage pond (Cameron and Di 2019, 2021). Additionally, the new FDE treatment approach has been shown to minimise P and *Escherichia coli* leaching losses with land application of treated effluent (TE), compared to the application of FDE. (Cameron and Di 2019; Wang et al. 2019; Chisholm et al. 2020; Che et al. 2022). The significant decrease in P leaching losses mainly resulted from the less available form of P in the TE. After treatment, the P in FDE combined with iron from PFS and formed iron phosphate which was less soluble and less likely to be leached, compared to the P in the original FDE. When the soil is not P-saturated, introducing PFS in the soil as an amendment may also increase soil P sorption capacity by adding additional sorption sites (Li et al. 2010; Loloei et al. 2014).

Total soil P concentrations range substantially from 100 to 3000 mg kg$^{-1}$ (Frossard et al. 2000; Haygarth et al. 2013), but only a small fraction of this is immediately plant-available, in the form of orthophosphate present within the soil solution (Hinsinger 2001; Condron 2004). There are numerous soil P tests available to help understand soil P distribution. For example, water extractable P (WEP) is an indicator for potential dissolved P losses from surface runoff via soil surface; calcium chloride extractable P ($CaCl_2$-P) is widely used to indicate the potential for soil P leaching losses; Olsen P is the most common test for plant-available P (McDowell and Condron 2004). However, the biochemistry of P in soils is complicated, that the availability of P depends on its chemical composition. To understand the effect of land application of FDE on the speciation of soil P, soil P fractionation can be used to operationally define the distributions of the various forms of soil P such as recognize plant-available (labile P) and not-available forms (moderately labile P, stable P and residue P) of soil P. The distribution of the different forms of soil P is affected by the changes in land use at different stages of ecosystem development and is constantly changing to maintain equilibrium between the solid soil phase and the soil solution. Thus, soil P fractionation is a useful indicator of the relative importance of P cycling via geochemical and soil biological processes, indicating the P transfer, accumulation and losses.

The long-term repeated application of dairy manure and mineral fertilizer affected the concentrations and proportions of all P fractions in Inceptisol and Ultisol soils (Condron and Goh 1989, Tran and N'dayegamiye 1995, Sharpley et al. 2004). Significant increases in Olsen P, total P, WEP and $CaCl_2$-P were reported by repeated application of FDE (Toor et al. 2004; McDowell et al. 2005, 2019; Hawke and Summers 2006; Raghunath et al. 2016). However, adding PFS to FDE changes the form of P in TE. FDE contains higher concentrations of dissolved reactive phosphorous (DRP) and total dissolved phosphorous (TDP) than TE, while the P in TE is mainly associated with iron surfaces on particulates (Longhurst et al. 2000; Cameron and Di 2019; Du 2019). As such, it might expect the speciation of P within soils treated with untreated FDE and TE to vary with possible implications for plant availability and concerns regarding pasture yields. Currently, little understanding of the distribution of labile P, moderately labile P, stable P and residual P with repeated applications of FDE or TE are known. Thus, a knowledge gap exists regarding the effect of multiple years of application of FDE vs TE on the P fraction. Additionally, the differences in P losses from the soil are related to the source of P input (Toor et al. 2004; Hawke and Summers 2006; He et al. 2006), and the previous studies in PFS-treated FDE were all short-term studies (Wang et al. 2019; Chisholm et al. 2020; Che et al. 2022). Thus, the comparative effects of repeated applications of TE vs FDE on the pasture dry matter yield and plant P uptake are currently unknown.

Therefore, the objectives of this study were to compare the effect of repeated applications of TE and untreated FDE on (i) the forms of P in the soil and (ii) pasture biomass and plant P uptake. The study hypothesis is that repeated applications of TE will result in different forms of soil P, different pasture yields and different amounts of plant P uptake when compared to repeated applications of untreated FDE.

## 2 Materials and methods

### 2.1 Multi-year field plot trial

The study was conducted on a 4-year pasture field plot trial that was established to compare the effects of land application of TE versus FDE (Cameron and Di 2019). The trial is located at the Lincoln University Research Dairy Farm (LURDF, 43° 38′ S, 172°27′ E) located 20 km of Christchurch. Soil type is Templeton silt loam soil classified as Immature Pallic soil, derived from weakly weathered greywacke alluvium (NZ classification, Hewitt (2010) and Udic Haplustepts, Survey Staff (1998)). The pasture was white clover (*Trifolium repens* L.) and perennial ryegrass (*Lolium perenne* L.). The field plots received a mean annual rainfall of 606 mm and a mean annual irrigation input of 450 mm, with a mean annual temperature of 12 °C. The trial was initiated in 2016 and consisted of 3 treatments with 17 replicate plots arranged in randomized blocks. The treatments comprised: (i) untreated effluent (FDE), (ii) treated effluent (TE) and (iii) control (water only). Each plot was 2.0 m long with 0.5 m wide, and 0.5-m buffers were around each side between plots.

### 2.2 Farm dairy effluent

FDE was collected from the LURDF, which contained Friesian-Jersey dairy cows (also known as 'Kiwi-cross') grazing outdoors on the grassland, consisting of perennial ryegrass and white clover mixture. On each treatment application day, untreated FDE was collected from the effluent sump tank directly after morning milking. The PFS treatment method is according to the guideline provided by Cameron and Di (2019). PFS was added as a coagulant to coagulate the colloidal solids in the effluent. After 60 min of settling, the effluent was separated into two layers: approximately two-thirds of the top of the mixing tank was the clarified water, as the recycled water for cleaning the farmyard, and treated effluent (TE), comprised the sludge settled at the lower part of the tank which was emptied into a storage pond.

The application rate of FDE and TE treatments was at 200 kg N ha$^{-1}$ year$^{-1}$, following local effluent application regulations (Hide 2008). The effluents were applied twice a year, one in the southern hemisphere spring (November) and another in autumn (April), at 100 N ha$^{-1}$ at each application. The volume of each effluent type required was calculated to match the same nitrogen application rate, and additional water was added to standardise the liquid input from each treatment application, thus, causing the differences in P input rate between the FDE and TE treatments. The Control plots received the equivalent amount of water as other treatments. The P concentrations and rates of each application for FDE and TE treatments are shown in Table 1. From 2016 to 2020, the plots received eight applications, with

the average of 14.0 and 25.8 kg P ha$^{-1}$ year$^{-1}$ phosphorus application rates from FDE and TE treatments, respectively. And no extra fertilizer was applied to all plots before the soil sampling. Treatments were applied manually using watering jugs to avoid the application flowing out of each plot area.

### 2.3 Soil sampling

Four years after the trial started, on 1 December 2020, soil samples were collected randomly from six replicate plots of each treatment (FDE, TE and control). Soil samples were collected by digging a block of soil 20 cm wide × 20 cm long × 20-cm deep soil with a spade in each plot. The whole block of soil was collected, and then the soils were partitioned into two different depths (0–10 and 10–20 cm). The soil samples in two soil depths were homogenised, and then the subsamples are taken for further analysis.

### 2.4 Soil analysis

Fresh soil subsamples were analysed for microbial biomass phosphorus within 3 days of sampling. The remainder of each soil sample was air-dried at 65 °C for 48 h, crushed and sieved through a 2-mm sieve for further analysis.

#### 2.4.1 Soil chemical analysis

All the soil samples from both depths were analysed for soil pH in deionised water with a 1:2.5 soil to water ratio of volume (Blakemore et al. 1987). Soil Olsen P was determined by extracting soil samples at 1:20 (w/v) ratio with 0.5 M NaHCO$_3$ at pH 8.5 (Olsen 1954). P concentration was determined colourimetrically by the Murphy and Riley

**Table 1** The P concentrations and rates of each application prior to the soil sample collection

| Applications | P application concentration (mg P L$^{-1}$) | | P application rate (kg P ha$^{-1}$) | |
|---|---|---|---|---|
| | FDE | TE | FDE | TE |
| 1 | 24.7 | 57.3 | 12.3 | 28.6 |
| 2 | 21.7 | 71.5 | 10.8 | 35.7 |
| 3 | 28.3 | 61.0 | 14.2 | 30.5 |
| 4 | 27.9 | 47.2 | 14.0 | 23.6 |
| 5 | 33.5 | 44.6 | 16.7 | 22.3 |
| 6 | 35.3 | 42.5 | 17.7 | 21.3 |
| 7 | 30.6 | 48.8 | 15.3 | 24.4 |
| 8 | 21.4 | 39.2 | 10.7 | 19.6 |
| Total | 223.3 | 412.1 | 111.7 | 206.1 |
| Mean | 27.9 | 51.5 | 14.0 | 25.8 |

*FDE* farm dairy effluent, *TE* treated effluent

(1962) method from the extracted supernatant using a Lachat Flow Injection Analyser. Total soil carbon and nitrogen were determined by a combustion method and analysed by a machine (Elementar Vario-Max CN Elemental Analyser, Hanau, Germany). Soil organic matter (%) was calculated by multiplying the soil organic carbon (%) by 1.724 (Blakemore et al. 1987; Peverill et al. 1999). Soil exchange cations (calcium, potassium, magnesium and sodium) were extracted by 1 M ammonium acetate at pH 7 with soil to solution ratio of 1:20. The filtered suspensions were then analysed using micro wave plasma atomic emission spectroscopy (MP-AES) for exchangeable bases (Rayment and Higginson 1992). The soil cation exchange capacity (CEC) was determined by the summation of the exchangeable cations including hydrogen (estimated by measuring the changes in pH of the cation extraction solution) (Brown 1943). Water extractable P (WEP) was determined at the soil to solution (deionised water) ratio of 1:300 with 45 min of end-over-end

shaking. Calcium chloride extractable P ($CaCl_2$-P) was determined at the soil to solution (0.01 M $CaCl_2$) ratio of 1:5 with 30 min end over end shaking (McDowell and Condron 2004; Dodd et al. 2012). Soil microbial biomass of P was determined using a chloroform fumigation and extraction method with a 1:20 extraction ratio of soil to chloroform (Blakemore et al. 1987; Wu et al. 1990; Grace et al. 2006).

### 2.4.2 Fractionation of P analysis

Soil P fractionations were determined according to the method described by Boitt et al. (2018), which was modified from the method of Hedley et al. (1982) (Olsen and Sommers 1982; Condron and Goh 1989; Condron et al. 1996; Condron and Newman 2011). The fractionation scheme is shown in Fig. 1. A 50-mL centrifuge tube was filled with 0.5 g of air-dried soil. Then 30 mL 1 M $NH_4Cl$, 30 mL 0.5 M $NaHCO_3$ at pH 8.5, 10 mL 0.1 M NaOH, 10 mL 1 M HCl and 10 mL 0.1 M
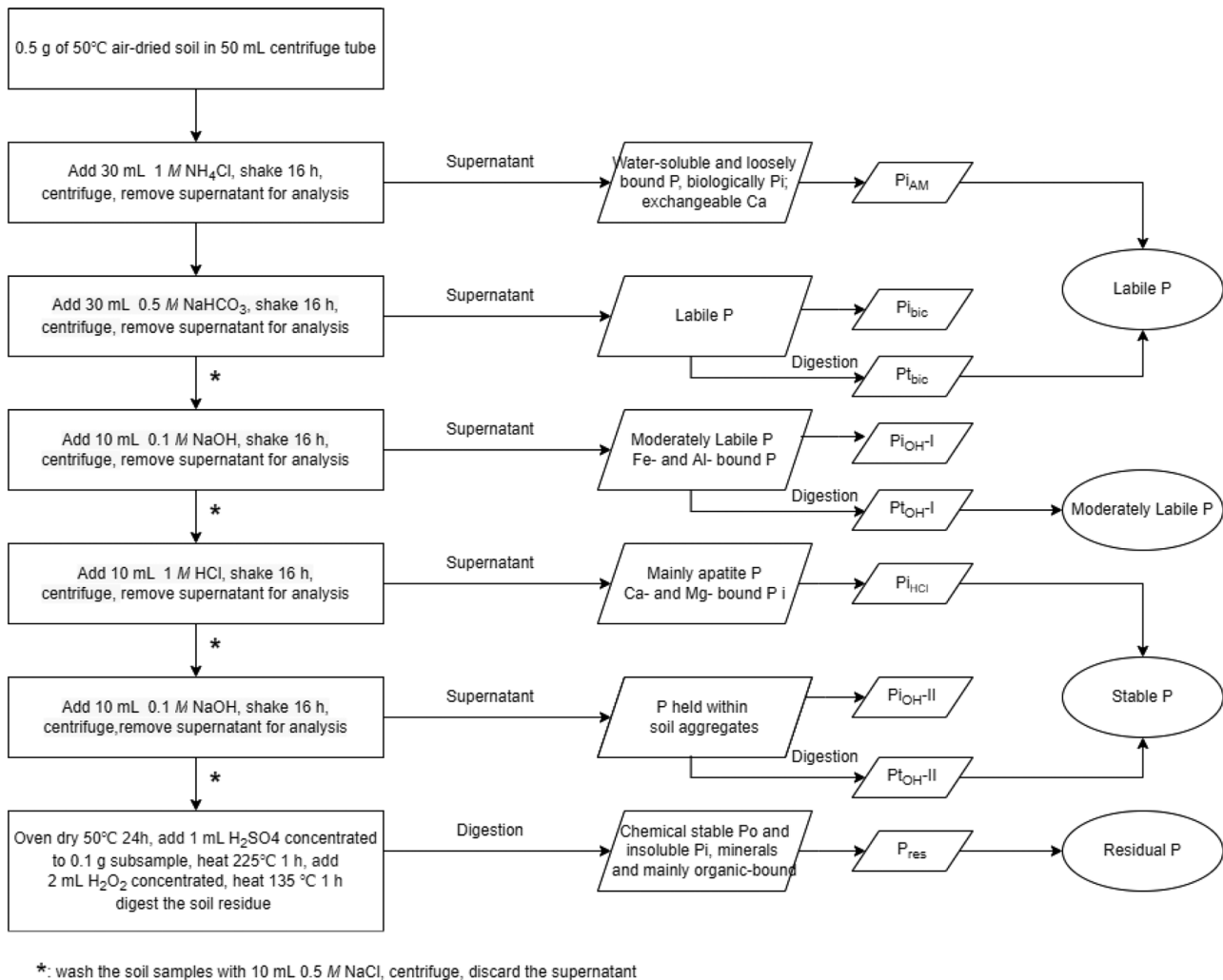


Fig. 1 Soil fractionation scheme used for the study, modified from Hedley et al. (1982)

NaOH were sequentially used to extract the soil sample followed by digestion with $H_2SO_4 + H_2O_2$. The inorganic P content of the extracted supernatants was measured as $Pi_{AM}$, $Pi_{bic}$, $Pi_{OH-I}$, $Pi_{HCl}$ and $Pi_{OH-II}$, respectively. Subsamples from $Pi_{bic}$, $Pi_{OH-I}$, and $Pi_{OH-II}$ were digested by $(NH_4)_2SO_4$ and $H_2SO_4$ to determine the total P as $Pt_{bic}$, $Pt_{OH-I}$, and $Pt_{OH-II}$, respectively (EPA 1978, Ohno and Zibilske 1991). Organic P (Po) was calculated by the differences between total P and Po from each fraction as $Po_{bic}$, $Po_{OH-I}$ and $Po_{OH-II}$. Non-extractable P was analysed using the digestion method as residue P ($P_{res}$) (Olsen and Sommers 1982). These extracted P forms were grouped as follows:

$$\text{Labile P} = Piam + Pi_{bic} + Po_{bic}$$

$$\text{Moderately labile P} = Pi_{OH\_I} + Po_{OH\_I}$$

$$\text{Stable P} = Pi_{HCl} + Pi_{OH\_II} + Po_{OH\_II}$$

$$\text{Residual P} = Pres$$

$$Po_{bic} = Pt_{bic} - Pi_{bic}$$

$$Po_{OH-I} = Pt_{OH-I} - Pi_{OH-I}$$

$$Po_{OH-II} = Pt_{OH-II} - Pi_{OH-II}$$

$$\text{Total inorganic P (total Pi)} = Pi_{AM} + Pi_{bic} + Pi_{OH\_I} + Pi_{HCl} + Pi_{OH\_II}$$

$$\text{Total organic P (total Po)} = Po_{bic} + Po_{OH\_I} + Po_{OH\_II}$$

All the concentrations of P in extractions were analysed using Murphy Riley's colourimetric method (Murphy and Riley 1962). Total soil P (total P) was analysed separately on an inductive coupling plasma emission spectrometer (Agilent 5110 ICP-OES Analyser, American).

## 2.5 Pasture sampling and analysis

The pasture was harvested to typical post-grazing height and collected during the 2021–2022 dairy milking season. On 18 November 2021, each plot received 50 kg P ha$^{-1}$ of 20% potash sulphur super. Nine harvests (4 August, 16 September, 11 October, 9 November and 9 December in 2021 and 11 January, 9 February, 9 March and 13 April in 2022) were carried out during the dairy milking season (August to May). The fresh weight of the pasture samples was recorded from each plot and dried at 75 °C for 48 h. The dry weight of samples was then measured and recorded, and dried plant samples were grounded and analysed for total P and N concentrations. Total herbage P concentrations were determined

using the same analyser machine for determining soil Total P. Total herbage N concentrations were determined using an Elementar Vario-Max CN Elemental Analyser (Elementar GmbH, Hanau, Germany).

## 2.6 Statistical analysis

To identify the effects of the treatments on the soil chemical values, soil P fractionation results and pasture results, an analysis of variance (ANOVA) was carried out using GenStat (22nd edition, Lawes Agricultural Trust). Tukey's HSD (honestly significant difference) test ($p < 0.05$) was used to determine the significance of the difference between treatments.

## 3 Results

### 3.1 Soil properties

There was no significant difference in soil pH, carbon and nitrogen ratio, CEC, calcium and sodium concentrations between the FDE, TE and the control treatments at both soil depths (Table 2). The concentrations of soil organic matter, total nitrogen, total carbon, exchangeable magnesium and potassium were significantly higher at the top 0–10 cm of soil than at 10–20 cm depth of soil for each treatment. The soils at 0–10-cm depth showed no significant difference ($p > 0.05$) in the soil pH, organic matter, total nitrogen, total carbon, CEC, exchangeable calcium magnesium, potassium and sodium contents between FDE and TE treatments. There were significantly higher concentrations ($p < 0.05$) of organic matter and total carbon in the FDE and TE treatments, compared to the control treatment. In 10–20-cm soil depth, the results of organic matter, total nitrogen, total carbon, CEC, exchangeable calcium, magnesium, potassium and sodium contents showed no significant difference ($p > 0.05$) between the FDE, TE and control treatments.

Mean concentrations of Olsen P values across all three treatments were 18.9 at 0–10 and 13.6 at 10–20 cm (Fig. 2). No significant ($p > 0.05$) difference in the concentrations of Olsen P (Fig. 2A), $CaCl_2$-P (Fig. 2C) and WEP (Fig. 2D) between the soils that received FDE, TE or control at the same depth. No significant difference ($p > 0.05$) was found in the concentrations of microbial biomass P between the FDE and TE applications within the same depth (Fig. 2B). The concentrations of P in the microbial biomass were significantly ($p < 0.05$) lower in the deeper soil layer, compared to the top soils from the same treatment. The mean concentration of microbial biomass P in the soil treated by FDE was 25.23 mg P kg$^{-1}$, which was significantly increased ($p < 0.05$) than the 16.1 mg P kg$^{-1}$ in the control soil at 0–10-cm depth.

**Table 2** Mean chemical properties of the soil at 0–10- and 10–20 cm following repeated applications of TE and FDE

| | | pH | | Organic matter % w/w | | Total nitrogen g kg⁻¹ | | Total carbon g kg⁻¹ | | Carbon/ nitrogen ratio | | CEC cmolc kg⁻¹ | | Exch-Ca cmolc kg⁻¹ | | Exch-Mg cmolc kg⁻¹ | | Exch-K cmolc kg⁻¹ | | Exch-Na cmolc kg⁻¹ | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 0–10 cm | FDE | $6.1\pm0.2$ | a | $4.4\pm0.3$ | c | $2.4\pm0.2$ | c | $25.7\pm1.8$ | c | $10.7\pm0.5$ | a | $11.7\pm0.8$ | a | $5.7\pm0.6$ | a | $0.69\pm0.13$ | c | $0.2\pm0.03$ | c | $0.22\pm0.04$ | a |
| | TE | $5.9\pm0.1$ | a | $4.5\pm0.2$ | c | $2.4\pm0.1$ | c | $26.3\pm1$ | c | $11\pm0.6$ | a | $12.3\pm1$ | a | $5.5\pm0.9$ | a | $0.6\pm0.12$ | bc | $0.17\pm0.03$ | bc | $0.2\pm0.07$ | a |
| | Control | $6\pm0.1$ | a | $4\pm0.3$ | b | $2.2\pm0.2$ | b | $23.3\pm1.6$ | b | $11\pm0.6$ | a | $11.8\pm0.8$ | a | $5.7\pm0.9$ | a | $0.47\pm0.14$ | abc | $0.14\pm0.02$ | ab | $0.16\pm0.06$ | a |
| 10–20 cm | FDE | $6.1\pm0.2$ | a | $3.5\pm0.1$ | a | $1.9\pm0.3$ | a | $20.3\pm0.5$ | a | $11\pm1.3$ | a | $10.8\pm1$ | a | $4.9\pm1$ | a | $0.47\pm0.18$ | abc | $0.12\pm0.03$ | a | $0.16\pm0.05$ | a |
| | TE | $6\pm0.1$ | a | $3.6\pm0.2$ | a | $1.8\pm0.1$ | a | $20.6\pm0.9$ | a | $11.5\pm0.5$ | a | $11.3\pm0.5$ | a | $4.9\pm0.5$ | a | $0.43\pm0.05$ | ab | $0.12\pm0.01$ | a | $0.12\pm0.04$ | a |
| | Control | $6\pm0.1$ | a | $3.5\pm0.2$ | a | $1.8\pm0.1$ | a | $20\pm0.9$ | a | $11.2\pm0.8$ | a | $11.5\pm1$ | a | $4.9\pm1$ | a | $0.38\pm0.1$ | a | $0.11\pm0.01$ | a | $0.13\pm0.07$ | a |

Values with different letters indicate significant differences between the treatments at $p < 0.05$

*FDE* farm dairy effluent, *TE* treated effluent

## 3.2 P fractionation

There was no significant difference ($p > 0.05$) in Pi, Po and total P between the soil that received FDE and TE treatments at the same depth (Fig. 3A–C). The concentrations of Pi varied from 205.8 to 263.2 mg P kg⁻¹ at 0–10 cm. In the top 0–10 cm of soil treated with TE, Pi concentrations were significantly higher ($p < 0.05$) than in control soil. Similarly, the mean total P concentration in TE-treated soil was 742.9 mg P kg⁻¹, in the 0–10-cm soil, which was significantly higher ($p < 0.05$) than in the control soil at 644.1 mg P kg⁻¹. The mean concentrations of soil Po in the deeper layer were lower than those in the topsoil layer for the same treatment, with average values of 188.4 mg P kg⁻¹ and 322.3 mg P kg⁻¹, respectively.

The soil that received the repeated applications of TE showed significantly higher ($p < 0.05$) labile P than the soil that repeatedly received FDE applications at both depths (Fig. 4A). No significant difference ($p > 0.05$) was detected in moderately labile P between the soils treated with FDE, TE and control at the same depth (Fig. 4B). The concentrations of moderately labile P were significantly greater ($p < 0.05$) in the 0–10-cm soil layer with an average of 360.1 mg P kg⁻¹ than in the 10–20-cm soil layer with 264.2 mg P kg⁻¹. There was no significant difference ($p > 0.05$) in stable P between the soils treated by FDE and TE treatments (Fig. 4C). There was also no significant difference ($p > 0.05$) in soil residue P between FDE, TE treatments and control with a mean concentration of 34.5 mg P kg⁻¹ across all depths (Fig. 4D).

## 3.3 Plant yield, P and N concentrations

The results showed no significant difference ($p > 0.05$) in pasture growth among FDE, TE treatments and control (Fig. 5). Similarly, the amounts of herbage P uptake were not significantly different ($p > 0.05$) among all three treatments, ranging from 52.8 kg P ha⁻¹ in the control to 55.2 kg P ha⁻¹ in the FDE treatment. Total herbage N uptake ranged from 378.0 kg N ha⁻¹ in control to 407.5 kg N ha⁻¹ in FDE treatment, and no significant difference ($p > 0.05$) among all treatments was found.

## 4 Discussion

### 4.1 Impact of TE vs. FDE on soil P fractions

The multi-year application of TE, but not FDE, significantly increased total Pi and total P in the 0–10-cm soil, compared with the control. These increases in P appear to be within the labile P fraction which was the only significantly increased fraction in the TE treatment, compared to FDE. The PFS-treated effluent had a higher P concentration than untreated FDE. Hence, at the 0–10-cm soil depth, the soil receiving TE
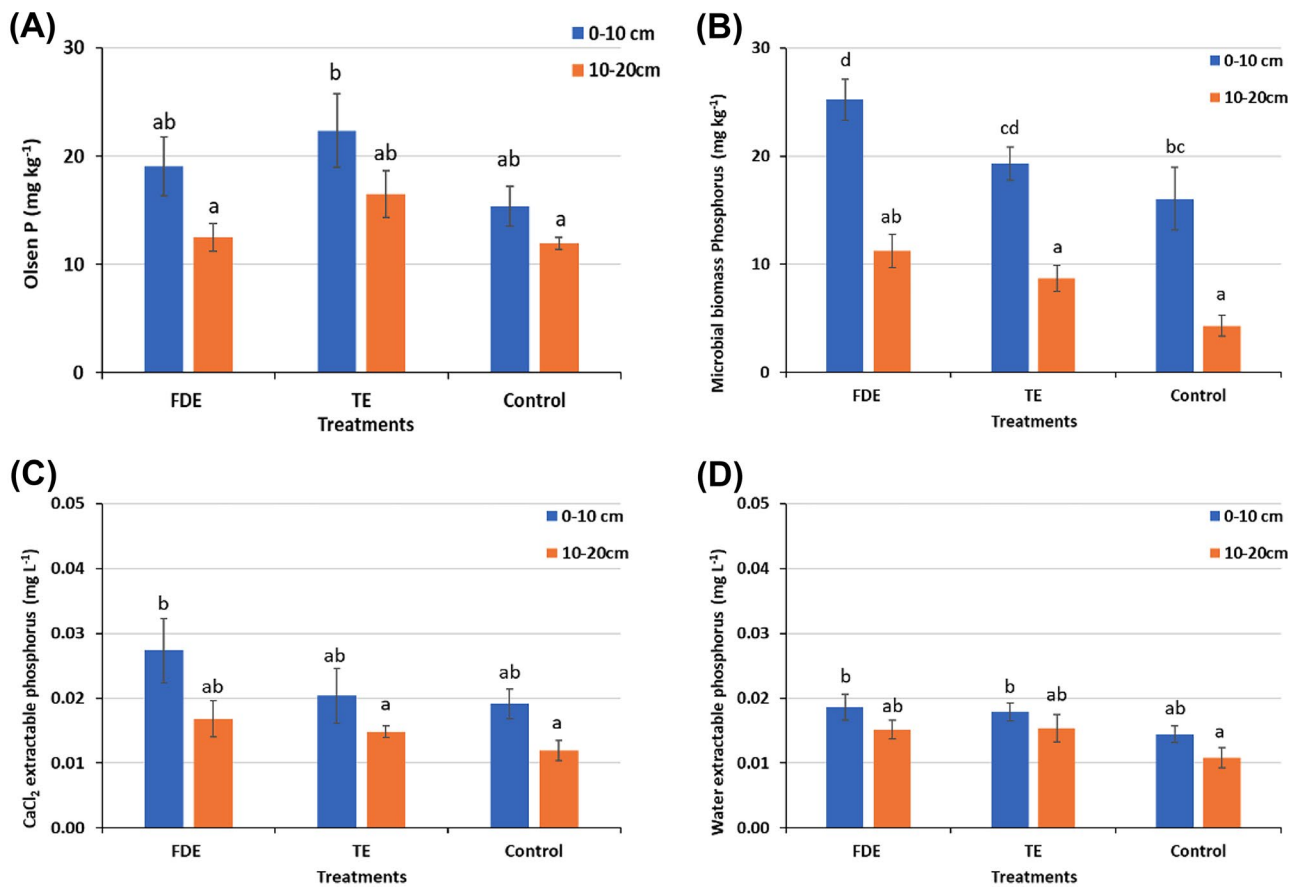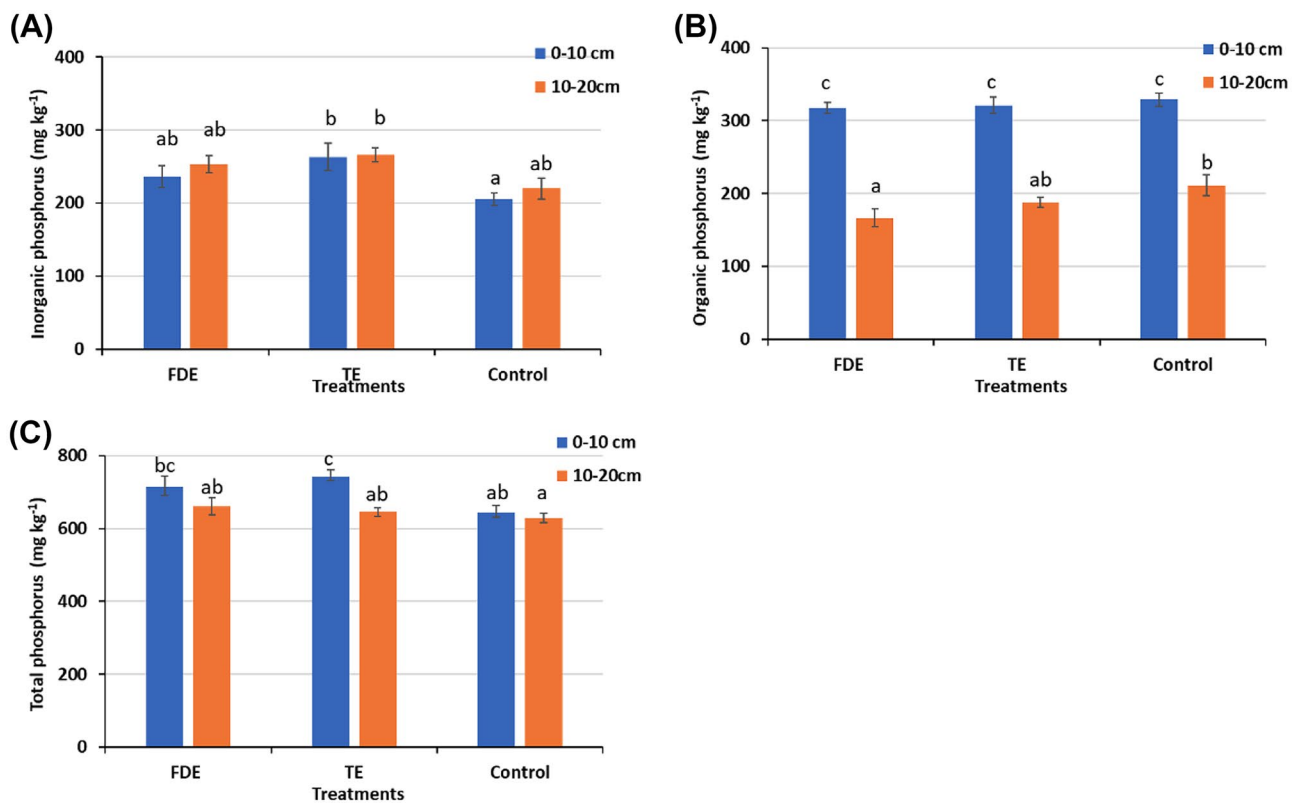
**Fig. 2** Average concentrations of soil Olsen P, microbial biomass P, CaCl$_2$-P and WEP from the soil at 0–10- and 10–20-cm depth

showed a significant increase in labile P, which was likely to have occurred from the higher mean P application rate in the TE application (25.8 kg P ha$^{-1}$ year$^{-1}$), compared with the FDE application (14.0 kg P ha$^{-1}$ year$^{-1}$); because the rates of application were standardised to match N inputs as regulated by local regulatory authorities. The P in TE was mainly combined with iron to form iron phosphate and stored in the moderately labile P pool. However, we did not find a significant increase in this fraction following repeated TE applications. This suggests that the moderately labile P is in dynamic equilibrium with the labile P pool and acts as a reservoir which can rapidly resupply the labile P in reaction to plant uptake (Johnson et al. 2003; Richter et al. 2006; Negassa and Leinweber 2009). The increase in labile P pools indicated that multi-year TE application increased the plant available P in the soil and could improve production long-term (Hawke and Summers 2006).

The differences in results attained for total P and the summation of Po and Pi from the Hedley et al. (1982) fractionation were the result of utilising different analytical so were not directly comparable, and the fractionation process would have resulted in incomplete P recoveries leading to lower total P values, compared with the single combustion method.

However, the relatively higher P application rate of TE did not result in increases in other soil P fractionations, compared to the FDE application. Negassa and Leinweber (2009) summarised that, in a temperate climate, the changes in P fractions depend on the application amount of P rather than the source of P. Low P input and high P input would slightly and substantially, respectively, increase the absolute concentrations and properties of different P fractions slightly and substantially. The mean P application rates from FDE and TE treatments were 14.0 and 25.8.0 kg P ha$^{-1}$ year$^{-1}$, respectively, from each application, both low application rates. This could be the main reason why even though the mean application rate of P with TE was higher than FDE; no significant difference in most soil P fractionations was found from the soil following repeated FDE and TE treatments. Similarly, fallow soils have also been shown to have the limited change in soil P factions in response to a low rate of P or with no P application within a 10-year period (Qian and Schoenau 2000; Hylander and Simán 2006).

Our earlier research has shown significant reductions in the amounts and the concentrations of P leaching losses through the soil received TE application, compared to the untreated FDE application (Wang et al. 2019; Chisholm

**(A)**



**(B)**



**(C)**



**Fig. 3** Average concentrations of soil inorganic P, organic P and total P in the soil from 0–10- and 10–20 cm depth

et al. 2020; Che et al. 2022). These may have resulted from the reaction of the ferric iron in PFS with the water-soluble P in the FDE, which formed a partially soluble/insoluble iron phosphate in the TE. The removal of dissolved reactive P in the TE reduced the P mobility in the soil (Cameron and Di 2019), which results in lower P losses from the TE application, compared to the FDE application. As such, we would have expected TE to increase the moderately labile P related to iron-bonded P (Fe–P). However, no significant difference between TE and FDE or the unamended control was found for this fraction. This suggests that any Fe–P added with TE was able to resupply the labile P pool and over time became available for plant uptake.

WEP and $CaCl_2$-P can be used to assess the potential and the magnitude for P losses from surface runoff (Sharpley and Moyer 2000) and leaching through soil (McDowell and Condron 2004), respectively. No significant differences in WEP and $CaCl_2$-P values between TE and control indicated that TE application did not lead to any increased risk of baseline P losses, compared with control. In the other studies, similar results showed no significant differences in P leaching losses between the land applications of TE and control (Wang et al. 2019; Chisholm et al. 2020; Che et al. 2022). Land application of FDE has been shown to result in higher P losses via leaching, compared to TE treatment and control (Wang et al.

2019; Chisholm et al. 2020; Che et al. 2022). However, the results could be caused by the large P losses immediately following the application of FDE but did not lead to a prolonged increased risk of P leaching after the initial flush of FDE. This agrees with the data shown in earlier studies that the concentrations of P in leachate showed breakthroughs and peaked immediately after the effluent application. Then, the concentrations of P decreased over time and showed no significant difference between TE application and control thereafter. Moreover, the lack of significant differences in WEP amongst the application of FDE, TE and control might be caused by the soil physical characteristic of the Templeton soil used in this study. McDowell et al. (2019) reported similar results that long-term FDE application led to enrichment at depth in Olsen P and WEP in a stony Eyre soil, but no significant difference in WEP and Olsen P in a Templeton soil due to lower porosity and hydraulic conductivity of the Templeton soil (Toor et al. 2005).

The changes in soil microbial biomass P could indicate changes in soil fertility, as it responds quickly to changes in plant or animal residues (Brookes 2001). The significant increase in soil microbial biomass P from FDE application indicates improved soil fertility, compared to control at 0–10-cm depth. Hawke and Summers (2006) found similar results of soil microbial biomass P increases by FDE
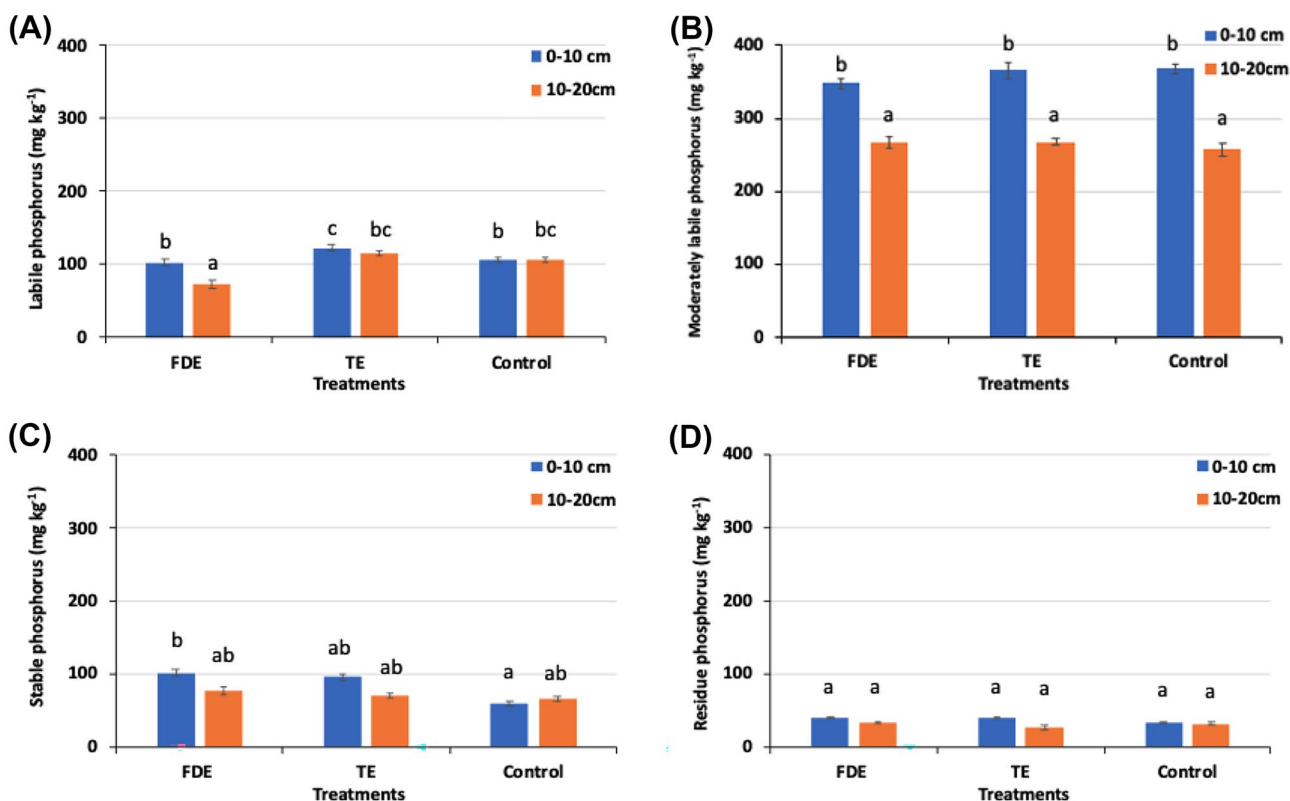
**Fig. 4** Average concentrations of soil labile P, moderately labile P, stable P and residue P in the soil from 0–10- and 10–20-cm depth

application. This increase would likely be caused by the greater nutrient contained in FDE, which stimulated microbial activity and surface soil (Zaman et al. 2002). The increase of microbial biomass can also be affected by the content of carbon in FDE, compared to control, which can lead to a more diverse dynamic microbial system (Xu et al. 2020). No significant difference in soil microbial P between FDE and TE application again indicated that TE application would not adversely affect soil fertility.

These results confirmed that the repeated land application of TE under the local regulation lead to no major changes in



**Fig. 5** Mean herbage dry matter yield and plant P and N uptakes followed by the application of the different treatments

soil P fractionations and availability, compared to repeated FDE application. It further indicated that the repeated applications of TE would not increase the risk of soil P loss, compared to FDE treatment in the long term.

## 4.2 Impact of TE vs FDE on herbage and P uptake

The results of P fractionation in this study showed that liable P was a very minor part of the total P content in the sampled soils ($15.6 \pm 0.2\%$), but the large quantities of P were stored in moderately labile forms which can potentially become plant-available P. However, the results of soil Olsen P did not show an increase with TE application, compared with FDE application and control. Soil Olsen P is the plant-available soil P test in New Zealand and a number of counties globally (Olsen 1954). The lack of differences in Olsen P in the soil between FDE and TE application might also have resulted from the lower P application rates, compared to the requirement of actual dairy pasture production. In this study, the P application rates were low from both TE and FDE applications, compared to the requirement of actual annual dairy pasture production. The Olsen P level of best economic return for local dairy pasture production is about 30 mg kg$^{-1}$ (Morton and Roberts 2009). The lower Olsen P values in 0–10-cm soil for FDE (19.1 mg kg$^{-1}$) and TE treatments
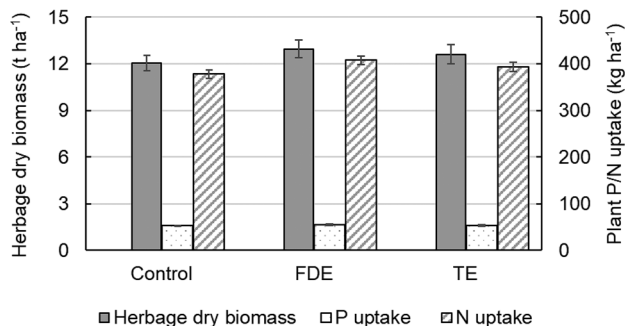
$(22.4 \ \mathrm{mg \ kg^{-1}})$ also indicated a lower P application rate than the local recommended P input level in this study.

Moreover, the lack of a significant difference in plant P uptake also agreed with Olsen P findings. Other short-term studies revealed comparable results in plant biomass and plant nutrient uptakes as this study (Du 2019; Wang et al. 2019; Chisholm et al. 2020; Che et al. 2022). The significant increases in soil organic matter, total carbon and exchangeable potassium with TE and FDE application were caused by the higher nutrient in FDE and TE, compared to water applied in the control (Longhurst et al. 2000; Cameron and Di 2019). These indicated the potential to increase soil fertility by repeated TE application. Thus, the results of this study further present that the repeated application of TE would not alter the plant-available P level, compared to the control and has no adverse effect on pasture yield and plant nutrient uptake.

## 5 Conclusions

Repeated land application of TE did not significantly affect most soil P fractionations, soil properties, herbage yield and plant P and N uptakes, compared to repeated applications of FDE. The only significant increase in soil labile P from TE application indicated the increase of plant-available P in soil and has a higher potential to improve pasture production long-term than FDE application. The hypothesis of this study is therefore rejected, and it is concluded that the repeated application of TE would not create any adverse impacts on soil P fractionations, P availability, plant dry matter yield and plant P uptake, compared with the repeated application of untreated FDE.

## Declarations

**Conflict of interest** The authors declare no competing interests.

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