

## TECHNICAL REPORT

## Surface Water Quality

# Factors controlling shallow subsurface dissolved reactive phosphorus concentration and loss kinetics from poorly drained saturated grassland soils

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**Abstract**

Shallow subsurface pathways dominate dissolved reactive phosphorus (DRP) losses in grassland soils that are: poorly drained, shallow, or have a perched water table in wetter months causing saturation-excess runoff. Saturated conditions can lead to anoxia, which can accelerate phosphorus (P) loss. Two scales of investigation were utilized in this study. First, at the field scale, soil cores were extracted to 2.5 m, subdivided and samples extracted using water extractable P (WEP) and sodium-bicarbonate-dithionite extractable P (NaBD-P). Second, at the laboratory scale, detailed incubation studies using field-moist grassland topsoils from sites in Ireland and New Zealand examined the kinetics of WEP under anoxic (WEP<sub>anox</sub>) and oxic (WEP<sub>ox</sub>) conditions with imposed temperature and soil P fertilizer input treatments. Results from soil-core samples showed that redox-sensitive NaBD-P concentrations were depleted where artificial drainage lines were installed (100 cm deep), but WEP concentrations available to shallow flow were enriched in topsoil. The laboratory scale incubation experiment investigated the influence of temperature (3 vs. 18 °C), anoxia (designed to simulate saturation following a rainfall event), and superphosphate fertilizer (10 to 60 kg P ha<sup>-1</sup> yr<sup>-1</sup>) on WEP concentrations over 24 h in three grassland topsoils (clay, silt, and sandy loam textures). Concentrations increased with fertilizer rate, temperature, and—in two soils—anoxic conditions. This was commensurate with nitrate (NO<sub>3</sub><sup>-</sup>) depletion and the reductive dissolution of iron and manganese. The release of P during anoxia was complete within 24 h. The results highlighted late winter to spring as the riskiest period for topsoil P losses in shallow subsurface flow due to wet soil conditions, increasing temperatures, and low soil NO<sub>3</sub><sup>-</sup> concentrations. This knowledge highlights the necessity to consider and refine

**Abbreviations:** DRP, dissolved reactive phosphorus; NaBD-P, sodium-bicarbonate-dithionite extractable phosphorus; WEP<sub>anox</sub>, anoxic water-extractable phosphorus; WEP<sub>ox</sub>, oxic water-extractable phosphorus.

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tests used to assess topsoil P loss risk, where in the landscape P losses are likely, and what strategies can be used to mitigate losses.

## 1 | INTRODUCTION

Globally, phosphorus (P) is essential for productive agriculture, and regular fertilizer P inputs into agricultural soils are necessary to maintain that productivity (Haygarth et al., 1998; Leinweber et al., 2002). However, P from agricultural soils can be lost to local surface water bodies via subsurface and surface pathways, leading to water quality issues such as eutrophication and excessive aquatic plant growth (Hansen et al., 2002; Heathwaite & Dils, 2000; Leinweber et al., 2002; Owens & Shipitalo, 2006). Many studies have concentrated on characterizing losses of P in particulate and dissolved forms via surface or subsurface pathways (Dupas et al., 2017; I. Thomas et al., 2016). The storage of dissolved reactive P (DRP) and kinetics of release in near-surface, saturated lateral pathways have not received much attention. Such pathways are highly relevant to P losses in grassland soils that are poorly drained, shallow, and have a perched water table, where there is a large seasonal difference in water table depths and where sites are predisposed to saturation-excess runoff (Z. Thomas et al., 2016).

Saturation excess conditions could cause changes in P loss kinetics along a lateral pathway via reductive dissolution (Gu et al., 2019; Warrinnier et al., 2020). When soil becomes saturated, respiring microbial communities will quickly deplete oxygen ( $O_2$ ) and nitrate ( $NO_3^-$ ) sources for energy by reducing them to solubilized forms via redox reactions. When these are depleted, the soil profile becomes anoxic and microbes rely on reducing P-sorptive manganese- (Mn) and iron- (Fe) oxides to create energy (McMahon & Chapelle, 2008). Any P sorbed to Mn- and Fe-oxides is dissolved. However, current tests, such as water extraction, predict the potential for P loss to runoff in oxic conditions (McDowell & Condon, 2004) and therefore may underestimate P loss under saturated (anoxic) conditions. More recent tests have tried to overcome this by quantifying P that is released from reduced Fe and Mn over the long term, referred to as “reducible-P” (or R-P; Smith et al., 2021).

In laboratory incubations, the time that microbial activity can induce anoxic conditions varies (Holford & Patrick, 1978; Leinweber et al., 2002; Patrick & Khalid, 1974; Zhang et al., 2010). For instance, Gu et al. (2019) incubated riparian wetland soils and sediments in a soil-to-solution ratio of 1:20 at room temperature and found DRP release plateaued after 24 h. However, Heiberg et al. (2012) incubated peat and sandy soils

under cereal crop rotation and found that reduction of Fe(III) began immediately after incubation, and DRP release under anoxic conditions leveled off after 45 days at room temperature (23 °C). Clearly, more information is required on how quickly anoxic conditions will cause P release in soils, especially grassland soils that are saturated intermittently. Here, it is important to know if the period of saturation is long enough that DRP release along surface and subsurface runoff pathways under anoxic conditions is significant.

As redox is microbially mediated, local soil temperature will influence the rate or occurrence of Fe-P dissolution. Sallade and Sims (1997) demonstrated that there was more rapid and pronounced Fe dissolution at an incubation temperature of 35 °C, compared to 7 °C. However, the cooler temperature is warmer than that exhibited by many temperate grassland soils during late autumn to early spring. For instance, at this time, most soils under grassland in Ireland and southern New Zealand would have a temperature range of 2–10 °C, in the first meter (Ceccon et al., 2011; Huang et al., 2014). More importantly, this is also when saturated conditions and P loss are most likely (Srinivasan & McDowell, 2009) leading to the question, “Will this cooler temperature significantly reduce the rate of P release under anoxic conditions?”

Long-term P fertilizer practices may also affect P losses. Previous work has shown a relationship between Olsen P concentrations and surface and subsurface P loss (McDowell & Condon, 2004). However, it is unclear how soils of different Olsen P would release DRP under anoxic conditions.

To address these issues, two scales of inter-linked investigation were undertaken: a soil coring field study on a hillslope grassland site in Ireland to examine the behavior of P forms readily available to short-term shallow subsurface flow (water extractable P [WEP]) and long-term loss under saturation (sodium-bicarbonate-dithionite extractable P [NaBD-P]) with depth. A laboratory scale incubation study examined the controls of P loss from field-moist surface grassland topsoils from the cored site in Ireland and another grassland site in New Zealand to (a) determine the concentration and kinetics of P release in water extracts under anoxic ( $WEP_{anox}$ ) and oxic ( $WEP_{ox}$ ) conditions over a 24-h period—deemed representative of a subsurface flow producing rainfall event and (b) to establish the importance of temperature and soil P fertility, representative of winter and summer seasons and typical P application rates (0–60 kg P ha<sup>-1</sup> yr<sup>-1</sup>).

## 2 | MATERIALS AND METHODS

### 2.1 | Intact soil core study

To determine the availability of P to be lost to water, five soil profile cores were taken every 30 m along a 100 m transect across a permanent grassland field at Johnstown Castle, Wexford (Ireland). This site is gently sloped with moderately to poorly drained soils. An artificial in-field pipe drainage system with a herringbone format is installed at 1 m intercepting and controlling a perched water table on site (Ibrahim et al., 2013).

A window sampling coring set (Van Walt) enabled soil sampling above and below the water table. Intact cores excavated to 2.5 m below ground level captured the distribution of P in unsaturated and saturated depths of the profile. Each profile core was divided into 0–10, 10–45, 45–70, 70–100, 100–130, 130–150, 150–175, 175–200, 200–215, and 215–250 cm below ground level sampling intervals, air-dried, sieved (< 2 mm) and analyzed for WEP<sub>ox</sub> and NaBD-P. The WEP<sub>ox</sub> test represents the readily available P in soil, while the NaBD-P test represents the long-term pool of R-P (Smith et al., 2021).

Additional data were collected to establish the likelihood of saturation occurring at the site. Soil water table data were collected daily from November 2017 to March 2019 using water level data loggers (TD-Divers, Eijkelpamp Soil & Water) within 2 m of each cored point to between 3 and 4 m below ground level. Soil profiles were cored adjacent to these piezometers on the slope. Alongside this, soil moisture deficit (SMD) data (Met Éireann, 2020) were used to estimate how often the site would reach saturation excess.

A subsample of the bulked cores from each replicate plot was analyzed (air-dried, < 2 mm sieved, Table 1) for WEP<sub>ox</sub>; NaDB-P, Olsen P (Olsen et al., 1954), anion sorption capacity (ASC)—a measure of the soil's P sorption capacity (Saunders, 1965), pH, total nitrogen (TN) and total carbon (TC) by LECO C/N analyzer, and particle size density (Gee & Bauder, 1986).

#### Core Ideas

- Reductive dissolution reactions increasing phosphorus (P) release can take <24 h.
- Oxygen, temperature, and fertilizer affected the amount and rate of P loss.
- Late winter to spring was the riskiest period for topsoil P losses in shallow subsurface flow. P losses occur due to wet soil conditions, increasing temperatures, and low soil nitrate.

### 2.2 | Incubation study

Surface soil from the three different grassland sites were sampled between 2018 and 2019: two in Ireland and one in New Zealand. These sites will be referred henceforth using their dominant soil textural classes: clay loam, sandy loam, and silt loam (Rickard & Moss, 2012).

The sites were classified using the New Zealand (Hewitt, 1998) or the Irish soil classification system and the equivalent Food and Agriculture Organization (FAO) classification (IUSS Working Group WRB, 2015) in parentheses. The clay loam (from Co. Wexford in Ireland) was a moderately drained stagnic brown earth (Cambisol) and was the same soil used in the intact coring study. The sandy loam (from Co. Cork in Ireland) was a well-drained typical brown earth (Cambisol). The silt loam was a well-drained Pallic firm brown soil (Cambisol) from Canterbury in New Zealand.

Initial soil test P values for these soils are listed in Table 2. The clay loam and sandy loam each have had three P treatments for a decade, receiving annual P applications of 20, 40, or 60 kg P ha<sup>-1</sup> yr<sup>-1</sup>. Each soil site selected received the same nitrogen treatment (300 kg N ha<sup>-1</sup>). Grass was not grazed but periodically harvested for hay/silage. There were three replicates of each treatment. The silt loam had three treatments receiving annual applications of single superphosphate (SSP) for over 60 years at rates of 10, 20, and 30 kg P ha<sup>-1</sup> yr<sup>-1</sup>. Each SSP treatment had four replicate plots and were grazed

TABLE 1 Mean soil chemical and physical parameters of the three sites used in the incubation of field-moist soils.

Texture profile <sup>a</sup>	Location	FAO soil group <sup>b</sup>	Irish/NZ Soil classification <sup>a</sup>	Temp	Rainfall	pH	TN	TC	ASC	Particle size	
				°C	mm yr <sup>-1</sup>		g kg <sup>-1</sup>	g kg <sup>-1</sup>	%	Sand %	Clay %
Clay loam	Co. Wexford, Ireland	Cambisol	Stagnic brown	10	1060	6.0	2.2	27	18	40	30
Sandy loam	Co. Cork, Ireland	Cambisol	Typical brown	10	1029	6.8	3.0	42	14	62	15
Silt loam	Canterbury, NZ	Cambisol	Pallic firm brown	11	781	5.4	3.9	43	26	30	25

Abbreviations: ASC, anion sorption capacity; TC, total carbon; TN, total nitrogen.

<sup>a</sup>Sourced from: Teagasc & Cranfield University (2007) and Landcare Research (2020).

<sup>b</sup>IUSS Working Group WRB (2015).

**TABLE 2** Soil phosphorus (P) tests for each soil and fertilizer treatment ( $n = 3$  per soil  $\times$  fertilizer treatment) and depth. Means ( $\pm$  standard error [SE]) contrasted via a two-way analysis of variance

Soil texture	P fertilizer treatment kg ha <sup>-1</sup> yr <sup>-1</sup>	WEP mg L <sup>-1</sup>	NaBD-P mg kg <sup>-1</sup>	Olsen P mg kg <sup>-1</sup>
Clay loam	20	0.07a (0.01)	50a (1)	31a (<1)
	40	0.07a (0.01)	66b (1)	33a (1)
	60	0.16b (0.02)	68b (<1)	47b (1)
Sandy loam	20	0.10a (0.01)	70a (<1)	52a (1)
	40	0.16b (0.01)	73b (1)	58b (<1)
	60	0.15b (0.01)	88c (<1)	70c (2)
Silt loam	10	0.11a (0.02)	55a (<1)	27a (1)
	20	0.09a (0.01)	66b (1)	39b (2)
	30	0.22b (0.01)	103c (1)	50c (1)
P <sub>Soils</sub>		<0.01	<0.001	<0.001
P <sub>Fertilizer</sub>		<0.001	<0.001	<0.001
P <sub>Soils <math>\times</math> Fertilizer</sub>		ns	ns	ns

Note: Different letters indicate significant difference between fertilizer treatment, within each soil type. NaBD-P, sodium-bicarbonate-dithionite extractable P; WEP, water extractable P.

by separate flocks of sheep at 10, 14, and 18 stock units ha<sup>-1</sup> (McDowell & Condon, 2012). Ten fresh, field-moist topsoil cores (0–10 cm) were hand-augured from random points in each plot and bulked.

The incubation experiment treatments were O<sub>2</sub> content (oxic vs. anoxic); temperature (3 vs. 18 °C); and fertilizer rate (20, 40, or 60 kg P ha<sup>-1</sup> yr<sup>-1</sup> for the clay loam and sandy loam, and 10, 20, and 30 kg P ha<sup>-1</sup> yr<sup>-1</sup> for the silt loam), with four replicates per treatment. Each bulked field-moist soil sample was sieved to < 4 cm and homogenized. Samples of 2 g were weighed into containers and prepared in either an anoxic glove box purged with nitrogen gas (N<sub>2</sub>) or in ambient air. In the glove box, deionized (DI) water was bubbled for 20 min with N<sub>2</sub> until anoxic (< 0.5 mg dissolved oxygen [DO] L<sup>-1</sup>), added to the soil to reach a ratio of 1:20 dry weight soil to DI water, stirred, and sealed before removal. Samples were placed on orbital shakers (170 rpm) and shaken and incubated in the dark at 18 or 3 °C. The same process was carried out for the oxic treatment but in ambient air (> 7 mg DO L<sup>-1</sup>) with untreated DI water. Chloroform was not used to sterilize soils, as it may have affected the ability of the soils to maintain an anoxic environment and have increased P concentrations by lysing microbial cells (Reddy et al., 1998). The four resulting treatment combinations (oxic 3 °C, anoxic 3 °C, oxic 18 °C, and anoxic 18 °C) were sampled sacrificially in triplicate at each time point (0, 60, 240, 480, and 1440 min).

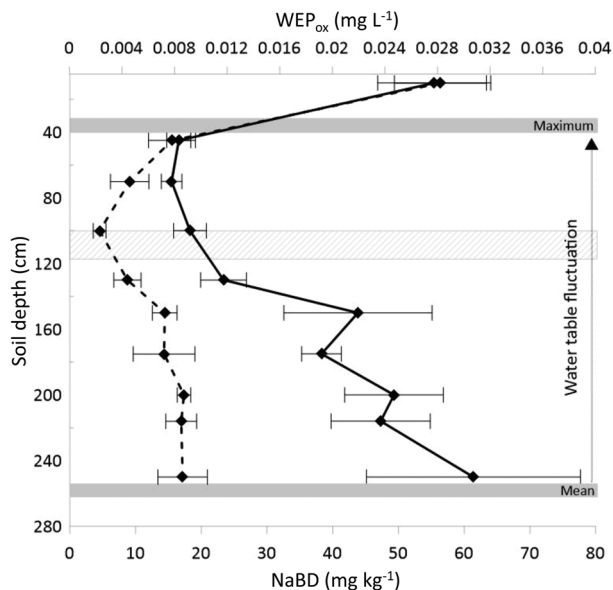
Samples were filtered to 0.45  $\mu$ m, acidified (1 mL 10% HCl) to prevent precipitation of redox-sensitive species (Gu et al., 2019) and analyzed immediately for DRP using a standard colorimetric procedure (Watanabe & Olsen, 1965). The main constituent in DRP is thought to be orthophos-

phate, although it is recognized that some organic P species may undergo acid hydrolysis and increase orthophosphate measurements by 10%–20% (McDowell & Sharpley, 2001). Redox-sensitive cations (Fe<sup>2+</sup> and Fe<sup>3+</sup>, Mn<sup>2+</sup>) were analyzed via inductively coupled plasma optical emission spectrometry and anions (NO<sub>3</sub><sup>-</sup>, SO<sub>4</sub><sup>2-</sup>) using Dionex suppressed ion-exchange chromatography.

The amounts ( $q_t$ , mg kg<sup>-1</sup>) of DRP desorbed were calculated after fitting an Elovich equation using Curve Expert 14.0. The Elovich equation is widely used in the study of P release from soils and sediments (Sparks, 2003). The Elovich model was selected as it fits the data better ( $r^2$  and standard error) than other commonly used models (pseudo-first-order and second-order equations; Table S1). For each treatment, the natural log of time,  $\ln(t)$ , was plotted against  $q_t$ , and the following Elovich model applied:

$$q_t = (1/\beta) \ln(\alpha\beta) + (1/\beta) \ln t. \quad (1)$$

The model gives the rate of reaction (mg kg min) via the equation constants,  $\alpha$  and  $\beta$ , which represent the initial chemisorption rate (mg g<sup>-1</sup> min) and extent of surface coverage and activation energy of chemisorption (g mg<sup>-1</sup>), respectively (Inyabor et al., 2016; McCallister, 2015; Ramachandran et al., 2011). The slope from the Elovich equation ( $1/\beta$ ) is equivalent to the desorption rate, and the intercept (" $(1/\beta)\ln(\alpha\beta)$ ") approximates the amount of P that is desorbable at time zero (McCallister, 2015). Other studies have quoted decreasing  $\beta$  values and increasing  $\alpha$  values as an indication of an increase in reaction rate, but the validity of this is not robust (Chien & Clayton, 1980; Sparks, 2003).



**FIGURE 1** Mean ( $\pm$  standard error) vertical distribution of sodium-bicarbonate-dithionite extractable phosphorus (NaBD-P, solid line) and water extractable P (WEP, dashed line) to 250 cm below ground level. Gray bars indicate the mean and maximum water table level over the five soil profiles from September 2017 to March 2019. The shaded area at 1 m below ground level indicates the location of artificial drainage.

### 2.3 | Statistical analysis

Analyses were conducted using R Studio and Minitab or Curve Expert 14.0 where specified (Minitab, 2018; R Development Core Team, 2010). A two-way analysis of variance (ANOVA) was used to compare mean soil P test concentrations, and Tukey's Honestly Significant Difference post hoc test was used to find specific significant differences between chemical parameters in the three soil groups.

Analysis by repeated measures was carried out using a general linear model applied to each of the three soils to identify the influence of  $O_2$ , temperature, and fertilizer as factors (and their interactions) on mean DRP, Fe, Mn, and  $NO_3$  concentrations measured over 24 h and on the P desorption rate ( $1/\beta$  from the Elovich model). A crossed method was selected as all combinations of factors are represented in the experimental design. Interactions between factors and P, Fe, Mn, and  $NO_3$  were calculated via a two-way ANOVA.

## 3 | RESULTS AND DISCUSSION

### 3.1 | Soil coring study

The investigation shows that there was a difference in P behavior with depth. For WEP, the amount of extracted P was greatest in the topsoil and decreased with depth (Figure 1). In

contrast, NaBD-P decreased with depth to 60–100 cm, coincident with the location of artificial drainage pipes at 100 cm depth, then gradually increased to concentrations like those recorded in the topsoil (0–10 cm). These data provide context for the potential of short- and long-term pools of P to influence P losses. More specifically, such results suggest that while drainage may be influenced by P that is susceptible to loss under long-term reducing conditions, shallow subsurface flow may be influenced by P that is susceptible to loss in the short term. We examine the influence of time and anaerobic conditions on WEP in the incubation. The possibility of short-term changes (e.g., over 24-h periods) is likely in such systems. For instance, between November 2017 and March 2019, the average soil water table level was 260 cm below ground level, but at some locations along the hillslope, the water table level would periodically come to within 40 cm of the soil surface (as indicated in Figure 1). At the bottom of the slope, the water table was at 100 and 250 cm below ground level for 27% of the period sampled. More importantly, for surface runoff, mean SMD in topsoil was  $\leq 0$  mm (i.e., indicating saturation excess/waterlogging) for 60% of the year between 2017 and 2019, meaning that shallow subsurface flow was likely as frequent as deep drainage (Met Éireann, 2020).

### 3.2 | Incubation study: Soil P concentrations

Mean Olsen P concentrations were enriched above those deemed necessary for optimal pasture production (20–40 mg  $kg^{-1}$ ) for drystock and dairy farming, which may explain why WEP concentrations were high (Table 2; Morton & Roberts, 2016; Roberts & Morton, 2009). Concentrations of NaBD-P in the New Zealand soil (silt loam) were between 55 and 103 mg  $kg^{-1}$ . Concentrations of NaBD-P were below the average of similar soils in national soil surveys from New Zealand (91 mg  $kg^{-1}$ ) and Ireland (92 mg  $kg^{-1}$ ; Smith et al., 2021).

### 3.3 | Factors controlling DRP release during the incubation

Table 3 shows the mean solution DRP concentration after 1440 min (24 h). DRP concentrations increased with fertilizer inputs in the clay loam, sandy loam, and silt loam. The warmer temperature significantly increased DRP concentrations in sandy ( $P < 0.001$ ), silt loams ( $P < 0.001$ ) and clay loam ( $P < 0.05$ ). Examples of DRP release over time are given in Figures 2–4 for each of the soils at the same fertilizer rate (20  $kg\ ha^{-1}$ ). Between 40% and 100% of cumulative DRP desorbed from each treatment occurred within the first 60 min of incubation. However, this varied between the soils. For instance, within 60 min, the sandy loam released between 80% and 100% of its cumulative DRP. Meanwhile, about 50%

**TABLE 3** Mean ( $\pm$  SE) solution dissolved reactive P (DRP) concentrations ( $\text{mg L}^{-1}$ ) averaged over 1440 min for each soil and fertilizer rate under anoxic or oxic conditions at 3 or 18 °C

Site	Clay loam			Sandy loam			Silt loam			
	Fertilizer	20	40	60	20	40	60	10	20	30
kg ha <sup>-1</sup> yr <sup>-1</sup>										
Anoxic (3 °C)	0.05	0.08	0.19	0.08	0.11	0.11	0.17	0.38	0.86	
	(0.01)	(0.02)	(0.03)	(0.01)	(0.01)	(0.05)	(0.02)	(0.04)	(0.08)	
Oxic (3 °C)	0.05	0.06	0.17	0.07	0.06	0.08	0.16	0.37	0.41	
	(0.01)	(0.01)	(0.03)	(0.01)	(0.01)	(0.05)	(0.03)	(0.03)	(0.07)	
Anoxic (18 °C)	0.09	0.14	0.21	0.14	0.16	0.18	0.26	0.46	0.81	
	(0.01)	(0.01)	(0.03)	(0.02)	(0.02)	(0.05)	(0.04)	(0.03)	(0.08)	
Oxic (18 °C)	0.07	0.08	0.27	0.14	0.15	0.15	0.24	0.41	0.63	
	(0.08)	(0.01)	(0.06)	(0.02)	(0.02)	(0.05)	(0.03)	(0.03)	(0.09)	
P <sub>Fertilizer</sub>	<0.001			<0.001			<0.001			
P <sub>Temperature</sub>	<0.05			<0.001			<0.001			
P <sub>Oxygen</sub>	ns			ns			ns			
P <sub>Fertilizer × temperature</sub>	ns			ns			<0.05			
P <sub>Fertilizer × oxygen</sub>	ns			ns			ns			
P <sub>Temperature × oxygen</sub>	ns			ns			ns			
P <sub>Fertilizer × temperature × oxygen</sub>	ns			ns			<0.05			

Note: The *p* value of general linear model (GLM) of the averaged solution dissolved reactive P concentrations within a soil group is given for each treatment and their interaction.

of cumulative DRP was released from the clay loam in the first 60 min. In contrast to the clay loam and sandy loam, there was a significant ( $P < 0.05$ ) fertilizer by temperature interaction, and fertilizer by temperature by O<sub>2</sub> interaction on DRP in the silt loam. Compared to the other two soils, DRP release from the silt loam was more gradual, especially with those incubated at 3 °C. The response of redox-sensitive parameters was not as apparent as that for DRP (Tables S2–S4). NO<sub>3</sub><sup>-</sup> was enriched in the clay loam under oxic and cold conditions (Table S2). The sandy loam maintained between 0.2 and 0.4 mg NO<sub>3</sub><sup>-</sup> L<sup>-1</sup> in solution between 60 min and 24 h (Figure 2), and the silt loam maintained > 0.8 mg NO<sub>3</sub><sup>-</sup> L<sup>-1</sup> (Figure 4). In contrast, the clay loam nearly exhausted NO<sub>3</sub><sup>-</sup> within the first 60 min in the anoxic treatments (Figure 3).

The patterns of Fe release were like those for P across all soils (Figures 2–4). The mean concentrations of dissolved Fe and Mn increased with temperature in the sandy and silt loam soils but not in the clay loam (Tables S3 and S4; Figures 2–4). Apart from enrichment in Fe under anoxia in the sandy loam, no differences were noted for the other soils or treatments.

### 3.4 | The effect of fertilizer, temperature, and O<sub>2</sub> status on DRP release

The Elovich model was applied to all soils and the slope (1/β) used (see also Table S1) to statistically compare the des-

orption rate ( $\text{mg g}^{-1} \text{min}^{-1}$ ) of DRP across treatments within soils (Table 4). A higher value was indicative of a faster release rate (McCallister, 2015). Interactions were significant between fertilizer, temperature, and O<sub>2</sub> (Table 4) for all soil textures. This means that changes in desorption rate were different for each combination of fertilizer rate, temperature, and O<sub>2</sub>. There was a significant increase under anoxia in the clay and sandy loams but not so for silt loam soils. The release of DRP from soil to solution depends on the quantity and strength of P sorbed to the soil relative to the capacity of the soil to sorb P and physical conditions that control the kinetics of P release into solution (Dodd et al., 2013; McDowell, 2012). Briefly, in neutral to acidic soils, the strength of P sorption increases with the quantity of P-sorbing Al and Fe hydrous oxides; the exchange or potential release of P from soils of a coarse texture is greater than from finer textured soils; and less P is sorbed and more desorbed as the quantity of added P increases relative to the number of P sorption sites in the soils (Dodd et al., 2013). In terms of the kinetics of P release, loosely sorbed and easily available P would be released before more strongly sorbed and recalcitrant P (Lair et al., 2009; Scalenghe et al., 2002). No fertilizer effect was expected in the redox-sensitive parameters (Fe, Mn, and NO<sub>3</sub><sup>-</sup>). This was largely the case. The exception was an increase in NO<sub>3</sub><sup>-</sup> in the silt loam (Table S2), this was due to the fact that stock numbers, and therefore urine patches containing N, increased with fertilizer rates (McDowell & Smith, 2012).

**TABLE 4** Mean ( $\pm$  SE) DRP desorption rates ( $1/\beta$ , mg g<sup>-1</sup> min<sup>-1</sup>) calculated via the Elovich model after 1440 min for each soil and fertilizer (Fert) rate under or oxic (Ox) conditions at 3 or 18 °C

Site Fertilizer	Clay loam			Sandy loam			Silt loam		
	20	40	60	20	40	60	10	20	30
kg ha <sup>-1</sup> yr <sup>-1</sup>									
Anoxic(3 °C)	0.21 (0.01)	0.26 (0.01)	0.77 (0.10)	0.45 (0.01)	0.31 (0.01)	0.16 (0.01)	0.63 (0.05)	0.71 (0.06)	0.46 (0.46)
Oxic (3 °C)	0.20 (0.20)	0.16 (0.02)	0.66 (0.02)	0.32 (0.01)	0.22 (0.02)	0.16 (0.01)	0.63 (0.64)	0.59 (0.06)	0.51 (0.05)
Anoxic (18 °C)	0.37 (0.02)	0.31 (0.03)	0.75 (0.07)	0.66 (0.02)	0.51 (0.03)	0.16 (0.01)	1.07 (0.13)	0.89 (0.01)	0.84 (0.24)
Oxic (18 °C)	0.30 (0.02)	0.23 (0.02)	1.04 (0.08)	0.63 (0.01)	0.52 (0.03)	0.17 (0.01)	0.97 (0.98)	1.09 (0.05)	0.45 (0.01)
P <sub>Fertilizer</sub>	<0.001			<0.001			<0.001		
P <sub>Temperature</sub>	<0.001			<0.001			<0.001		
P <sub>Oxygen</sub>	<0.01			<0.001			ns		
P <sub>Fertilizer × temp</sub>	ns			<0.001			ns		
P <sub>Fertilizer × oxygen</sub>	<0.01			<0.01			ns		
P <sub>Temp × oxygen</sub>	ns			<0.001			ns		
P <sub>Fertilizer × temp × oxygen</sub>	<0.05			<0.001			<0.01		

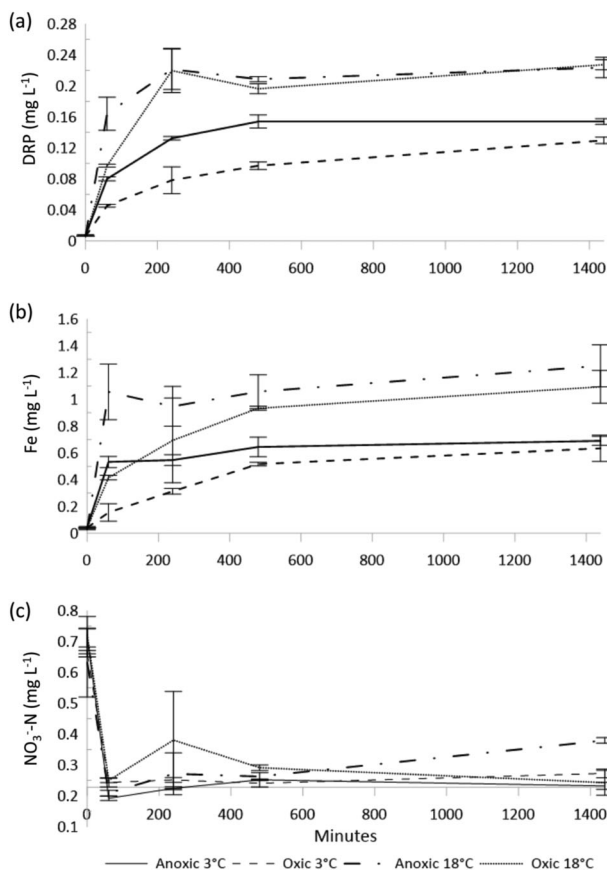
Note: The *p* value of GLM of the mean DRP desorption rates within a soil group is given for each treatment and their interaction.

Of the soils studied, the sandy loam released a majority of its total DRP lost in the first 60 min. Owing to its high sand content, sandy loam and other sandy soils have low concentrations of amorphous and crystalline Fe and Mn oxides (McCallister, 2015), which translates to a low ASC (viz., P sorption capacity). Coupled with a coarse texture, providing easy access to sorption sites, P is desorbed easier than the other soils (Saunders, 1965). The high proportion of cumulative DRP released from the soil in the first 60 min suggests that the low number of sorption sites, indicated by the low ASC and coarse texture, were not only more saturated but more loosely bound than the other soils (McDowell & Sharpley, 2004).

In contrast to the other soils, the clay loam exhibited a delayed release of P. This has been observed in other studies (Moazallahi et al., 2018) and attributed to a two-phase reaction, where loosely bound P is desorbed quickly before the dissolution of more tightly held P occurs (Lair et al., 2009; Moazallahi et al., 2018). The influence of dissolution is supported by simultaneous flushes of DRP, Fe, and Mn into the solution. Coupled with this is a delay in reaction caused by aggregation whereby P is slowly diffused from the inside of aggregates under oxic or anoxic conditions (McDowell & Sharpley, 2003).

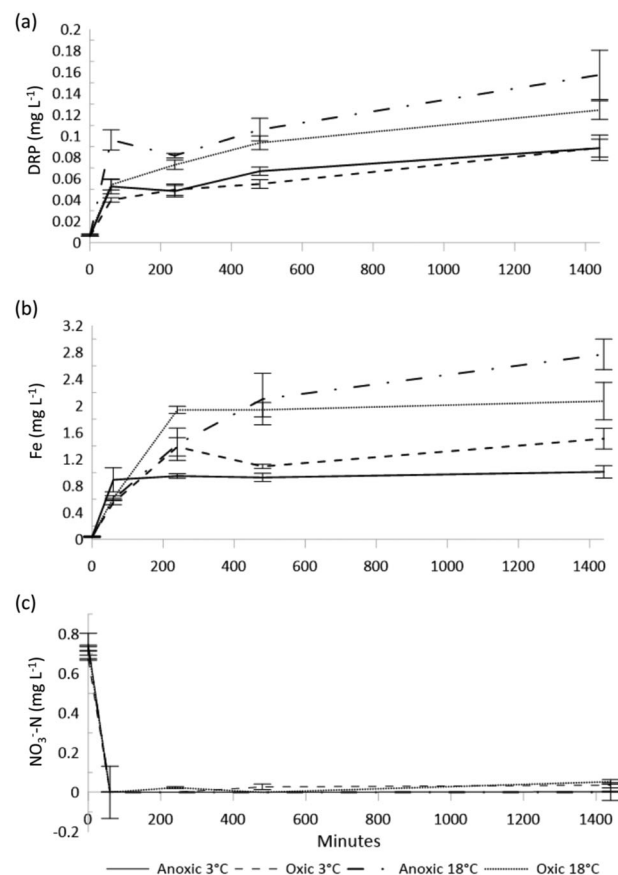
In the present study, temperature was consistently (across all soils sampled) a significant factor controlling DRP desorption rate and solution concentration. Previous research has noted a relationship between a quicker reaction rate and warmer temperatures caused by mineralizing organic P,

increasing reduction potential (Eh) in anoxic conditions, or stimulating biological activity that utilizes nutrients and O<sub>2</sub> under oxic and anoxic conditions (Sallade & Sims, 1997; Sparks, 2003). For instance, anoxic incubations on ditch sediments carried out by Sallade and Sims (1997) demonstrated that a similar downward trend in Eh occurred in 7 and 35 °C incubations, but redox potential decreased faster at 35 °C and more than doubled the soluble P concentrations after 21 days. Meanwhile, a study by Gibbons (2015) noted that sediments incubated at 10 and 20 °C had similar release rates, while the 30 °C incubation exhibited greater concentrations and release rates. The soils used in this study are rarely warmer than 20 °C, and never warm in the late winter to early spring periods where waterlogging and anoxia are more likely to occur (Met Éireann, 2020; NIWA, 2019). The 18 °C treatment only modestly increased concentrations in the averaged data, but the rationale was that this was more representative of actual soils conditions that the extreme temperature contrasts noted above. It is worth noting that the level of effect of temperature on DRP concentrations varied between soil types in the present study. The clay loam exhibited a low effect (e.g., 0.04 mg L<sup>-1</sup> difference at 20 kg P ha<sup>-1</sup> yr<sup>-1</sup>), compared to the other soil textural classes (e.g., 0.07–0.1 mg L<sup>-1</sup> difference at 20 kg P ha<sup>-1</sup> yr<sup>-1</sup>), perhaps being buffered by a finer-texture and two-phase reaction process as noted above. Previous work on sediments highlighted how anoxic P release is inconsistent due to the fact that temperature influences a variety of factors that may operate in opposing directions (e.g. Eh, mineralization, O<sub>2</sub> consumption, diffusion rates; Gibbons, 2015).



**FIGURE 2** Example of (a) dissolved reactive P (DRP), (b) dissolved iron (Fe), and (c) nitrate-N ( $\text{NO}_3^-$ ) concentrations, detected over time under the various temperature and oxygen ( $\text{O}_2$ ) treatments for the sandy loam receiving  $20 \text{ kg ha}^{-1} \text{ yr}^{-1}$ . Error bars represent the 95% confidence interval.

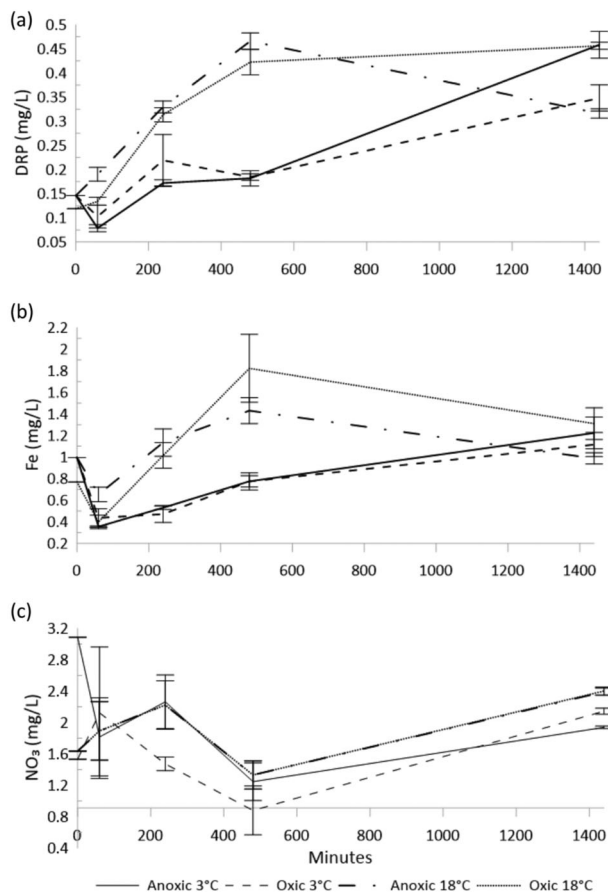
The mean concentrations at each time point and over the length of the incubations suggested that anoxic conditions enriched P concentrations in solution across the soil groups. In theory, dissolution and diffusion should be aided by anaerobic conditions throughout the soil or in anaerobic microsites caused by microbes that enhance P release (Hutchison & Hesterberg, 2004; Roden et al., 2000; Scalenghe et al., 2002). However, while the data presented herein suggested that anoxic conditions increased the rate of DRP release in the clay and sandy loam soils, this was not the case with silt loam soil. The answer to this discrepancy may lie in the greater concentrations of TC and TN in the silt loam soil (Table 1), and the availability of nitrate-N, supporting microbial growth, which in turn would maintain anoxic conditions. It is also likely that  $\text{NO}_3^-$  was formed via the mineralization of organic N (a major proportion of TN in New Zealand pastoral soils) during soil disturbance (Kristensen et al., 2000; Ringuet & Bachmeier, 2006). High  $\text{NO}_3^-$  concentrations were also probably in solution from urine and higher stocking rates used in the silt loam soil that was absent in the clay or sandy loam



**FIGURE 3** Example of the (a) dissolved reactive P (DRP), (b) dissolved iron (Fe), and (c) nitrate-N ( $\text{NO}_3^-$ ) concentrations detected over time under the various temperature and  $\text{O}_2$  treatments for the clay loam receiving  $20 \text{ kg ha}^{-1} \text{ yr}^{-1}$ . Error bars represent the 95% confidence interval.

soils (18 stock units vs. none for the clay and sandy soils, respectively).  $\text{NO}_3^-$  is a limiting factor in Fe-P release, as it will be preferentially used by microbes in redox reactions before Fe and Mn are acted upon (McMahon & Chapelle, 2008; Surridge et al., 2007).  $\text{NO}_3^-$  concentrations in the silt loam were significantly greater than the clay or sandy loam and were hence inhibiting reductive dissolution. No difference between DRP concentrations from the silt loam due to  $\text{O}_2$  content suggests that 24 h was not long enough to sufficiently remove enough  $\text{NO}_3^-$  in the silt loam system for metal oxides to be affected by reductive dissolution or that the conditions were not reduced enough. Previous work has suggested that greater differences occur beyond a 24-h time period (Gibbons, 2015) and that optimum reduction potential occurred after 4 days of incubation. Conversely, the lower TN concentrations in the clay and sandy loam soils ( $1.8$  and  $3.0 \text{ g kg}^{-1}$ ) likely buffered  $\text{NO}_3^-$  concentrations but not at a great enough concentration ( $0.5 \text{ mg NO}_3^- \text{ N L}^{-1}$ ; McMahon & Chapelle, 2008) to prevent anoxic conditions from affecting DRP release.





**FIGURE 4** Example of the (a) dissolved reactive P (DRP), (b) dissolved iron (Fe), and (c) nitrate-N ( $\text{NO}_3^-$ ) concentrations detected over time under the various temperature and  $\text{O}_2$  treatments for the silt loam receiving  $20 \text{ kg ha}^{-1} \text{ yr}^{-1}$ . Error bars represent the 95% confidence interval.

### 3.5 | Implications for management

This experiment aimed to determine if over a 24-h period, P release from topsoils would be enhanced by increased temperature and anoxia. While the enhancement was true for two of the three soils, it is possible that this may be true for soils, only if the soil contained a low soil nitrate-N concentration. Furthermore, many of the effects were found within 60 min (Figures 2–4). Both findings are important for predicting how soils respond to saturated conditions. However, more importantly, the results highlight the need to refine the knowledge of local saturated areas where shallow subsurface flow is likely in topsoils. The present study also highlights the riskiest period of the year for losses due to these processes (i.e., spring). This is when topsoils are still moist and easily saturated with small rainfall events, temperatures are warmer, and most  $\text{NO}_3^-$  had been leached from the soil.

General strategies like decreasing soil Olsen P to an agronomic optimum (Morton & Roberts, 2016) and/or switching to crops that are productive on lower Olsen P soils will

be effective in decreasing P losses from these soils year-round (McDowell & Cosgrove, 2016). Provided the soil type and situation allows it, artificial drainage systems could be redesigned and improved to maximize drainage and avoid saturated conditions in topsoils. However, such systems could end up losing the same load of P by draining a larger amount of more dilute water (Monaghan et al., 2000). To decrease the load effectively, drainage systems could be designed to intercept and filter out P by, for example, lowering the soil P through inversion tillage above the main perforated pipe or using a highly P-sorptive backfill (McDowell et al., 2008). Alternatively, where anthropogenically altering the land is untenable, saturated areas could be identified to inform management decisions, especially during high-risk periods of the year. For instance, strategies specific to springtime could include avoiding the grazing or the application of P fertilizer to wet fields. This includes delaying the grazing of pasture or forage crops in gullies to avoid animals being present in the base of gullies that are likely to be saturated (Monaghan et al., 2017).

Additionally, the results call into question the safety of accumulated P in riparian zones or buffer areas adjacent to waters. If accumulated P is in a form that is vulnerable in the long-term (e.g., NaBD-P), the present study would suggest that buffer areas that have low  $\text{NO}_3^-$  during risky periods (i.e., high temperature, low  $\text{O}_2$ ) may be a potential source of P to waterways. Another example where low  $\text{NO}_3^-$  may pose a risk is in heavy-textured soils in Ireland. In these soils, where artificial land drainage is installed, there have been studies that recorded low  $\text{NO}_3^-$  but high ammonium ( $\text{NH}_4^+$ ; Peyton et al., 2016). Soil N primarily being converted to the  $\text{NH}_4^+$  would not provide the same buffering effect and may increase the potential for reductive dissolution of P and its loss via artificial drainage (Clagnan et al., 2018).

## 4 | CONCLUSION

Over a 24-h period, the mean DRP concentration in water extracts increased with fertilizer application and temperature and in two topsoils, anoxic conditions—commensurate with the depletion of  $\text{NO}_3^-$  and release of Fe and Mn via reductive dissolution. Owing to enriched topsoil N concentrations, anoxia may not have been sustained in a third soil. Treatment effects on the kinetics of P release into water mirrored those of mean concentrations, except that for the clay loam soil, which noted a distinct two-phase release attributed to its finer texture, greater surface area and potentially greater store of P held within soil aggregates than the silt or sandy loams. Importantly, treatment effects such as the enhanced release of P during anoxia were complete for the clay and sandy loam soils within 24 h. Based on the results presented, late winter to spring would be the riskiest season for shallow subsurface P

losses due to this reaction, owing to moist topsoils that are easily saturated with small rainfall events, warmer temperatures, and low soil  $\text{NO}_3^-$  concentrations. This knowledge highlights the necessity to consider and refine where in the landscape P losses are likely and what strategies can be used to mitigate losses.

## AUTHOR CONTRIBUTIONS

**Gen J. Smith:** Data curation; formal analysis; investigation; methodology; writing—original draft; writing—review and editing. **Richard W. McDowell:** Conceptualization; funding acquisition; investigation; methodology; supervision; writing—original draft; writing—review and editing. **Karen Daly:** Supervision; writing—review and editing. **Daire Ó hUallacháin:** Methodology; supervision; writing—review and editing. **Leo M. Condrón:** Supervision; writing—review and editing. **Owen Fenton:** Funding acquisition; methodology; supervision; writing—review and editing.

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## CONFLICT OF INTEREST

The authors declare no conflict of interest.

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## SUPPORTING INFORMATION

Additional supporting information can be found online in the Supporting Information section at the end of this article.

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