Journal of Environmental Quality

TECHNICAL REPORTS

Vadose Zone Processes and Chemical Transport

Temperature and freezing effects on phosphorus release from soils to overlying floodwater under flooded-anaerobic conditions

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Abstract

Increased phosphorus (P) availability under flooded, anaerobic conditions may accelerate P loss from soils to water bodies. Existing knowledge on P release to floodwater from flooded soils is limited to summer conditions and/or room temperatures. Spring snowmelt runoff, which occurs under cold temperatures with frequent freeze–thaw events, is the dominant mode of P loss from agricultural lands to water bodies in the Canadian Prairies. This research examined the effects of temperature on P dynamics under flooded conditions in a laboratory study using five agricultural soils from Manitoba, Canada. The treatments were (a) freezing for 1 wk at −20 °C, thawing and flooding at 4 ± 1 °C (frozen, cold); (b) flooding unfrozen soil at 4 ± 1 °C (unfrozen, cold); and (c) flooding unfrozen soil at 20 ± 2 °C (warm). Pore water and surface water were collected weekly over 8 wk and analyzed for dissolved reactive phosphorus (DRP), pH, calcium, magnesium, iron (Fe), and manganese (Mn). Soils under warm flooding showed enhanced P release with significantly higher DRP concentrations in pore and surface floodwater compared with cold flooding of frozen and unfrozen soils. The development of anaerobic conditions was slow under cold flooding with only a slight decrease in Eh, whereas under warm flooding Eh declined sharply, favoring reductive dissolution reactions releasing P, Fe, and Mn. Pore water and floodwater DRP concentrations were similar between frozen and unfrozen soil under cold flooding, suggesting that one freeze–thaw event prior to flooding had minimal effect on P release under simulated snowmelt conditions.

1 | INTRODUCTION

Phosphorus (P) loss from agricultural soils via runoff, leaching, and erosion is a major contributor of P to aquatic systems (King et al., 2015; Smith et al., 2015). This is an environmental concern because P is the most limiting nutrient for freshwater eutrophication (Schindler, 1977; Schindler, Carpenter, Chapra, Hecky, & Orihel, 2016; Schindler, Hecky, & McCullough, 2012). Both the release and mobilization of P from soils are regulated by various biogeochemical and hydrological processes. When soils are flooded for prolonged periods, the development of anaerobic conditions changes the chemistry of soils, often through microbially mediated reactions. These reactions may enhance the release and mobilization of P to overlying floodwater and thus accelerate P loss from soils to water bodies (Amarawansha, Kumaragamage, Flaten, Zvomuya, & Tenuta, 2015;

Abbreviations: DAF, days after flooding; DRP, dissolved reactive phosphorus.

Jayarathne, Kumaragamage, Indraratne, Flaten, & Goltz, 2016; Tian, Dong, Karthikeyan, Li, & Harmel, 2017). This effect has been mostly attributed to the reductive dissolution of iron (Fe) and manganese (Mn) constituents releasing adsorbed and/or precipitated phosphates (Amarawansha et al., 2015; Young & Ross, 2001).

In the Canadian Prairies, dissolved P in snowmelt runoff is the dominant source of P exported from watersheds to surface waters (Corriveau, Chambers, & Culp, 2013; Tiessen et al., 2010). During the snowmelt period, vegetation and soils are subjected to repeated freeze–thaw cycles of varying durations (Liu et al., 2019b). Snowmelt events in the Canadian Prairies are also characterized by flooding because the quick snowmelt process generates high volumes of snowmelt runoff, in combination with slow water infiltration through frozen soils (Liu et al., 2019a) and poor surface drainage due to the relatively flat landscape (Bedard-Haughn, 2009; Buttle et al., 2016). Each spring the duration of flooding on fields may range from a few days to several weeks.

Most of the documented evidence on enhanced P release from soils to floodwater has focused on summer flooding conditions and/or flooding under room temperatures (Amarawansha et al., 2015; Dharmakeerthi, Kumaragamage, Indraratne, & Goltz, 2019b; Tian et al., 2017). The magnitudes of P release from flooded soils under spring snowmelt conditions or cold temperatures are poorly represented in the literature (King et al., 2015). In a previous study by Sallade and Sims (1997), P released from flooded sediments at 7 °C was found to be significantly lower than at 35 °C. Dharmakeerthi, Kumaragamage, Goltz, and Indraratne (2019a) reported significantly lower P release to pore water and floodwater from flooded soils under simulated spring snowmelt (previously frozen soils flooded at 4 °C) compared with simulated summer (unfrozen soils flooded at 22 °C) flooding. However, it is uncertain whether the decreased P release was caused by the lower temperature during flooding, the freezing event prior to flooding, or both. The objective of this study was to further investigate the effects of cold temperatures with and without freezing on P release from flooded soils to pore water and floodwater using soils typically found in the Canadian prairies. The potential for anaerobic conditions to develop under flooded conditions should still exist under cold temperatures with freezing events; however, we hypothesized that low temperature and freeze–thaw events would reduce P release from flooded soils when compared with summer flooding conditions.

2 | MATERIALS AND METHODS

2.1 | Soil collection and analysis

Five surface (0–15 cm layer) agricultural soils were collected from different sampling sites in the Red River

Core Ideas

- P release from flooded soils was greater under warm than cold temperatures.
- Floodwater DRP concentrations were much greater under warm than under cold flooding.
- Development of anaerobic conditions in soils was very slow under cold flooding.
- Redox reactions responsible for P release were less dominant under cold flooding.
- One pre-flood freezing event had minimal effect on P release under cold flooding.

Valley and Interlake Region in Manitoba, Canada. Soils belonged to Eigenhof (Orthic Black Chernozem), Red River (Gleyed Rego Black Chernozem), Osborne (Rego Humic Gleysol), Fairford (Eluviated Eutric Brunisol), and Balmoral (Rego Humic Gleysol) series according to the Canadian system of classification (Canadian Agricultural Services Coordinating Committee, 1998), with U.S. soil taxonomy equivalents of Typic Cryoboroll, Aeric Calciaquoll, Histic Calciaquoll, Eutrochrepts, and Histic Calciaquoll (Soil Survey Staff, 2014), respectively. Approximately 10–12 samples were obtained from each site and combined to obtain a composite soil sample. Composite soil samples were passed through a 10-mm mesh screen and stored at room temperature under field moisture conditions before initiating the experiment. A representative subsample from each soil was air-dried and sieved (2-mm mesh). Soils were analyzed for pH (1:2.5 soil/water) using a Fisher Accumet AB15 pH meter (Fisher Scientific) and texture by the pipette method (Gee & Bauder, 1986). Soil test P was determined using the Olsen method (Olsen, Cole, Watanabe, & Dean, 1954); soils were extracted with 0.5 mol L^{-1} NaHCO$_3$ solution at pH 8.5 and P concentrations in filtered extracts were determined by the molybdate blue color method (Murphy & Riley, 1962), measuring the absorbance at the 882-nm wavelength using an Ultraspec 500 pro UV/visible spectrophotometer (Biochrom). Total P was determined using the H$_2$SO$_4$–H$_2$O$_2$ digestion method (Parkinson & Allen, 1975), and the P concentration in the digested solution was measured by inductively coupled plasma atomic emission spectroscopy (iCAP 6500, Thermo Scientific). A single point P sorption study was conducted by equilibrating a soil sample with a solution containing 150 mg P L^{-1} (Amarawansha, Kumaragamage, Flaten, Zvomuya, & Tenuta, 2016) and calculating the P sorption capacity using the difference between the initial and equilibrium solution P concentrations. The degree of P saturation was calculated by expressing Olsen P as a percentage of P sorption capacity (Amarawansha et al., 2016; Kumaragamage et al., 2019). Organic matter and calcium carbonate equivalent

were also analyzed using a modified loss on ignition method (Dean, 1974). Cation exchange capacity was determined by displacing all cations by Na^+ and subsequently extracting Na^+ and measuring the Na concentration in the extract (Hesse, 1971) using atomic emission spectroscopy (AAnalyst 400, PerkinElmer).

2.2 | Incubation study under simulated snowmelt and summer flooding conditions

Field-moist soils were packed into 1.5-L glass incubation vessels with 10 cm internal diameter (nine vessels for each soil) to a depth of 7 cm and a bulk density of 1.1 ± 0.05 g cm^{-3}. A Rhizon-flex pore water sampler with an outer diameter of 2.5 mm and 0.15 mm pore size (Rhizosphere Research Products) was installed at 5-cm depth in each incubation vessel during soil packing to extract pore water. An Eh probe with a platinum sensor (Paleo Terra) was installed at a depth of 5 cm to monitor the changes in Eh in each vessel during flooding. Of the nine vessels for each soil, for 1 wk three were frozen at −20 °C, another three were kept at 4 ± 1 °C, and the remaining three were kept at 20 ± 1 °C. The vessels with frozen soils were thawed for 3 d at 4 ± 1 °C and then flooded using cool, deionized water (18 MΩ cm, Millipore) to a 5-cm ponding depth and incubated under cold temperature (4 ± 1 °C) for 56 d (frozen, cold treatment). It should be noted, however, that under natural field conditions, freezing occurs only from the surface, whereas in the experimental setup, the vessels were exposed to low-temperature conditions from all directions, which may influence the rapidness of freezing and the soil temperatures. We selected −20 °C for freezing and +20 °C to simulate summer flooding based on the typical temperatures for the winter and the summer seasons in the region. The second set of vessels that were not frozen but kept under cold temperature were flooded similarly and incubated at 4 ± 1 °C for the 56-d flooding period (unfrozen, cold treatment). The third set of vessels with unfrozen soils were flooded with deionized water to a 5-cm ponding depth and incubated under warm temperatures (20 ± 2 °C) for 56 d (warm treatment). During incubation, all vessels were kept covered with perforated Parafilm M (Fisher Scientific) to minimize evaporation. The experiment was conducted using a randomized complete block design with three replicates.

Redox potential was determined at 5 cm depth on the day of flooding and thereafter at 7-d intervals by temporarily inserting a reference electrode (Ag/AgCl saturated with KCl, coupled to a permanently installed Pt redox probe) into floodwater just enough to reach the soil surface. On the day of the flooding and thereafter at 7-d intervals, pore water and floodwater samples were taken from each vessel. Samples of pore water were taken using a 20-ml syringe attached to the end of the Rhizon flex sampler. Floodwater was extracted using

a 20-ml syringe from the center of the vessel and immediately filtered through a 0.45-µm membrane filter. Pore water samples were immediately analyzed for dissolved reactive P (DRP) concentration. Floodwater samples were analyzed within 6 h using the molybdate blue color method (Murphy & Riley, 1962), and absorbance was measured using an Ultraspec 500 pro UV-visible spectrophotometer (Biochrom). All water samples were analyzed for pH (Fisher Accumet pH meter) within 24 h of sampling. Total Ca, Mg, Fe, and Mn concentrations were determined using flame atomic absorption spectroscopy (AAnalyst 400, Perkin Elmer).

2.3 | Statistical analysis

Analysis of variance for pore water and floodwater DRP concentrations was performed for each soil separately using the GLIMMIX procedure in SAS Version 9.4 (SAS Institute, 2018) with flooding treatment (Frozen cold, Unfrozen cold, and Warm) as the fixed effect, replicates as blocks, and days after flooding (DAF) as the repeated measures factor. Based on the Akaike information criterion (Littell, Henry, & Ammerman, 1998), the covariance structure used in the final mixed models was heterogeneous compound symmetry, and the DRP concentrations were modeled as lognormal distribution. The Tukey multiple comparison procedure was used to compare least square means when three or more treatment means were compared. Simple linear regression analysis was performed separately for each soil and flooding treatment to identify the relationships between floodwater DRP and soil Eh, pore water and floodwater pH, and cation concentrations in pore water and floodwater. For all statistical analyses, significance was determined at $\alpha = .05$.

3 | RESULTS

3.1 | Initial soil properties

Initial soil properties are presented in Supplemental Table S1. All soils were slightly alkaline (pH 6.5–7.5). Clay content varied from 360 to 780 g kg^{-1}, giving textures of clay to clay loam. All soils had medium to high organic matter contents ranging from 47 to 71 g kg^{-1}. The calcium carbonate equivalent ranged from 23 to 124 g kg^{-1}, indicating that the studied soils were weakly to moderately calcareous. The cation exchange capacity values were moderate, ranging from 21.6 to 33.0 $cmol_c$ kg^{-1}. The Olsen P concentrations ranged from 28.5 to 69.8 mg kg^{-1}, and total P in soil ranged from 701 to 1066 mg kg^{-1}. Phosphorus sorption capacity ranged from 452 to 756 mg kg^{-1}, resulting in a high variability in degree of P saturation from 6.3% in Fairford and Balmoral series to 12% in Red River series.
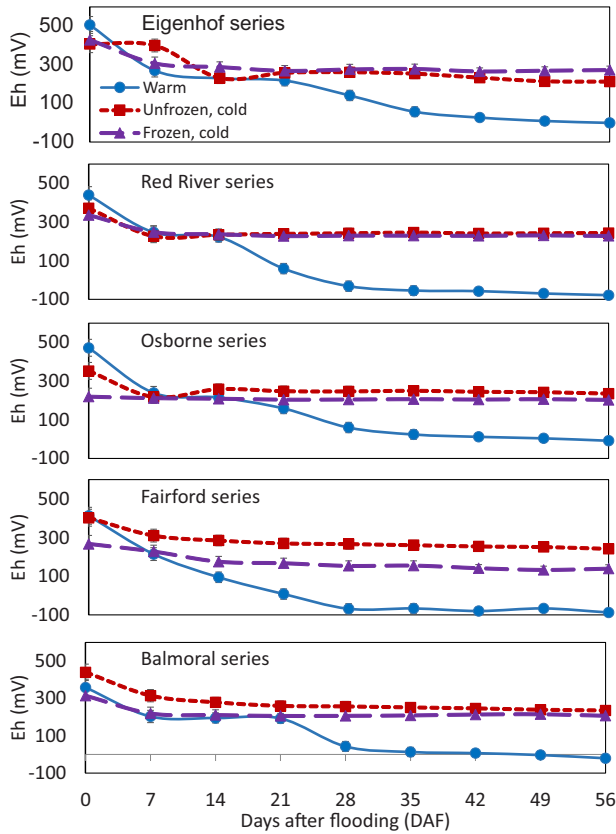
FIGURE 1 Change in mean soil redox potential (Eh, mV) with flooding time for different flooding treatments (unfrozen soils flooded under warm and cold temperatures, and frozen soils flooded under cold temperatures). The vertical bars at each week indicate the standard error of mean ($n = 3$)

FIGURE 2 Change in dissolved reactive P (DRP) concentrations (geometric least squares mean) in pore water with flooding time for different flooding treatments (unfrozen soils flooded under warm and cold temperatures, and frozen soils flooded under cold temperatures). Vertical bars at each week indicate SEM ($n = 3$). Trt, treatment

3.2 | Effect of flooding conditions on soil redox potential changes with flooding time

Soil Eh measured at the 5-cm depth ranged from 220 to 506 mV on the day of flooding and decreased with time of flooding; the magnitude of decrease was largely influenced by the flooding treatment (Figure 1). During the initial stages of flooding for up to ≈7–21 DAF (depending on the soil), similar Eh values were observed under different flooding treatment. Beyond 21 DAF, Eh values in all soils declined sharply with warm-temperature flooding. However, Eh values in both unfrozen and frozen soils under cold-temperature flooding decreased only slightly or remained relatively stable (Figure 1). By the end of the flooding period, soil Eh under warm flooding dropped to values between 0 and −88 mV, whereas Eh under cold flooding of unfrozen and frozen soils remained in the ranges of 213–243 and 139–273 mV, respectively. Whereas the Eh values beyond 28 DAF in all soils were lower under warm flooding than the corresponding Eh values under cold flooding, the Eh values among frozen
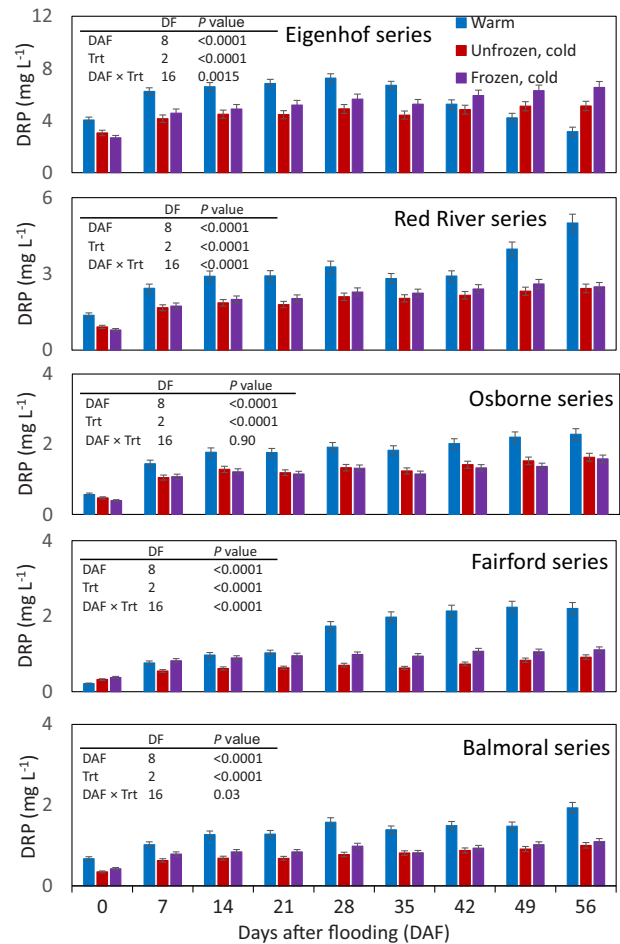
and unfrozen soils flooded under cold temperatures were similar (Figure 1).

3.3 | Dissolved reactive phosphorus concentration changes in pore water and floodwater with flooding time under different flooding conditions

During the initial stage of flooding (up to about 28 DAF), the pore water DRP concentrations steadily increased in all soils irrespective of the flooding treatment (Figure 2). Thereafter, the pore water DRP concentration either decreased, remained relatively stable, or continued to increase, depending on the soil and the flooding treatment. For pore water DRP concentrations, the two-way interaction of flooding treatment × DAF was significant ($P < .05$) in all but the Osborne
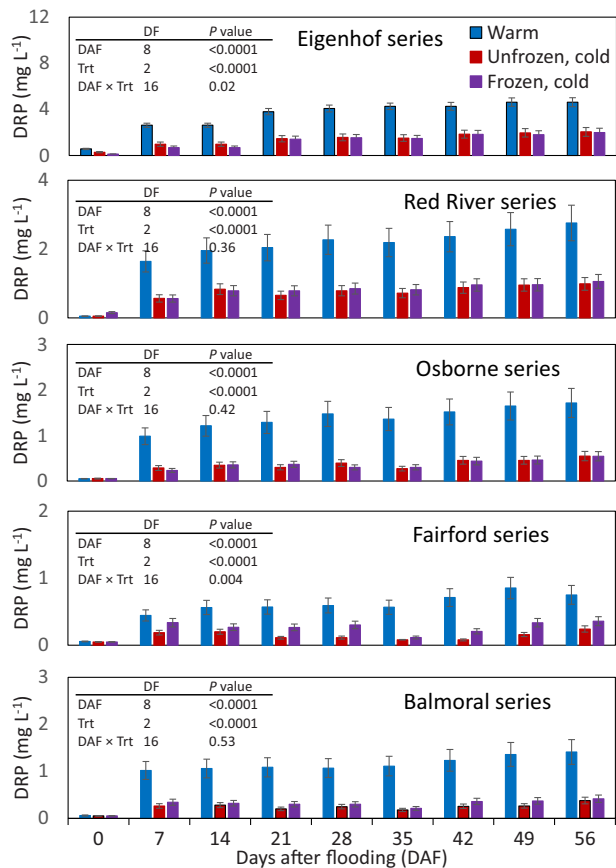
FIGURE 3 Change in dissolved reactive P (DRP) concentrations (geometric least squares mean) in floodwater with flooding time for different flooding treatments (unfrozen soils flooded under warm and cold temperatures, and frozen soils flooded under cold temperatures). Vertical bars at each week indicate SEM ($n = 3$). Trt, treatment

soil; however, even in Osborne soil, the main effects of DAF and flooding treatment were highly significant ($P < .0001$).

Under warm flooding, the pore water DRP concentration in general, increased substantially and significantly in all soils; however, under cold flooding of frozen and unfrozen soils, pore water DRP concentrations remained relatively stable or increased only slightly beyond 28 DAF. In most soils, pore water DRP continued to increase from 28 DAF to 56 DAF. However, the exception was the Eigenhof soil, for which pore water DRP concentration decreased beyond 28 DAF with warm flooding (Figure 2).

Floodwater DRP concentrations showed a sharp increase up to 7 DAF and then continued to increase at a slower rate in most soils under the different flooding treatment (Figure 3). In Balmoral and Fairford soils, a slight decrease in floodwater DRP concentrations was observed from 14 to 35 DAF for unfrozen and frozen soils flooded under cold temperatures, but concentrations increased again after 35 DAF. The DRP concentrations were significantly ($P < .05$) greater under warm than under cold flooding in both pore water (Figure 2)

and floodwater (Figure 3), with a few exceptions. The exceptions were pore water DRP concentration in Eigenhof soil from 35 to 56 DAF and Fairford soil during 0–14 DAF.

3.4 | Changes in pore water and floodwater cation concentrations with flooding time under different flooding conditions

Pore water concentration changes with time of flooding showed a similar trend for Ca and Mg (Table 1). Under warm flooding of unfrozen soils, the concentrations remained stable or slightly declined up to about 21–28 DAF and thereafter steadily increased with DAF in all except Osborne soil, where the concentrations remained relatively stable. The increase was largest in Fairford soil, with about twofold increase in pore water Ca and Mg concentrations at 56 DAF compared with respective concentrations at 0 DAF. In acidic Eigenhof soil, the concentrations decreased significantly up to 21–28 DAF and then increased, reaching concentrations similar to those at 0 DAF by 56 DAF. In contrast to warm flooding, pore water Ca concentrations under cold flooding of frozen and unfrozen soils increased to 7 DAF and then decreased with time of flooding. Pore water Mg concentrations fluctuated during the first 28 DAF and then decreased in most soils.

Pore water Fe and Mn concentrations were below detectable levels (0.05 and 0.16 mg L^{-1} for Fe and Mn, respectively) in soils flooded under cold conditions. When flooded under warm conditions, detectable concentrations were observed, but only at latter stages of flooding. Under warm flooding, pore water Fe concentrations were detectable at 35 DAF in Red River and Fairford soils, whereas pore water Mn concentrations were detectable at 14 DAF in Fairford soil; at 28 DAF in Red River soil; and at 35 DAF in Eigenhof, Osborne, and Balmoral soils (Table 1).

Floodwater Ca and Mg concentrations increased with DAF in general, under all flooding conditions, with greater increases under warm flooding than cold flooding (Table 2). Floodwater concentrations of Fe and Mn were at less than detectable levels for all soils under all flooding conditions (Table 2).

3.5 | Changes in pore water and floodwater pH with flooding time under different flooding conditions

Pore water pH values tended to decrease in the four alkaline soils after about 28 DAF under warm flooding but increased throughout the flooding period in slightly acidic Eigenhof soil; thus, by the end of the flooding period, all soils had similar pore water pH values in the range of 6.9–7.3 (Supplemental Table S2). However, pore water pH increased with DAF in

TABLE 1 Concentrations of pore water Ca, Mg, Fe, and Mn (mean, $n = 3$) over the flooding period for unfrozen soils flooded under warm and cold temperatures, and frozen soils flooded under cold temperatures

Soil	DAF	Warm flooding of frozen soils				Cold flooding of unfrozen soils		Cold flooding of frozen soils	
		Ca	Mg	Fe	Mn	Ca	Mg	Ca	Mg
					—mg L^{-1}—				
Eigenhof	0	67 (7.4)	36 (4.4)	ND	ND	47 (9.8)	31 (7.5)	41 (2.3)	34 (1.1)
	7	66 (6.3)	21 (4.3)	ND	ND	67 (7.4)	19 (4.2)	87 (7.0)	30 (0.9)
	14	43 (3.9)	27 (2.4)	ND	ND	51 (8.0)	23 (3.3)	74 (0.3)	35 (3.2)
	21	36 (3.2)	25 (1.5)	ND	ND	51 (3.3)	28 (2.3)	56 (6.0)	42 (2.8)
	28	40 (3.7)	17 (1.1)	ND	ND	51 (2.6)	19 (2.5)	61 (1.2)	30 (1.9)
	35	47 (3.8)	22 (3.8)	ND	0.4 (0.02)	42 (2.0)	16 (0.5)	50 (0.7)	24 (1.8)
	42	53 (5.0)	27 (3.7)	ND	1.2 (0.03)	43 (2.4)	22 (1.5)	48 (1.5)	23 (3.7)
	49	60 (0.8)	30 (1.6)	ND	2.4 (0.08)	39 (1.8)	17 (1.6)	45 (0.7)	22 (1.3)
	56	69 (1.8)	37 (4.4)	ND	4.3 (0.25)	38 (0.6)	18 (0.9)	45 (2.0)	21 (2.3)
Red River	0	67 (2.8)	44 (0.9)	ND	ND	39 (3.5)	25 (1.5)	55 (14.6)	36 (7.2)
	7	87 (2.1)	31 (1.8)	ND	ND	89 (0.8)	30 (1.2)	100 (1.1)	36 (2.1)
	14	71 (4.8)	37 (2.7)	ND	ND	75 (2.9)	38 (2.0)	92 (1.5)	42 (2.3)
	21	72 (2.3)	45 (1.1)	ND	ND	66 (3.4)	40 (5.6)	81 (1.3)	47 (2.7)
	28	87 (2.3)	38 (3.9)	ND	0.3 (0.02)	67 (0.7)	27 (0.8)	80 (1.5)	37 (6.2)
	35	94 (2.7)	50 (3.7)	0.1 (0.03)	0.8 (0.05)	60 (1.3)	33 (1.2)	69 (1.4)	32 (1.9)
	42	107 (2.7)	67 (3.0)	0.9 (0.11)	1.7 (0.08)	58 (1.8)	28 (4.2)	66 (0.5)	35 (2.1)
	49	129 (6.1)	71 (4.7)	3.2 (0.40)	3.4 (0.29)	55 (0.5)	28 (4.0)	63 (0.5)	33 (1.7)
	56	146 (8.3)	93 (8.0)	6.7 (0.71)	4.9 (0.41)	53 (0.7)	23 (2.1)	61 (2.9)	27 (1.7)
Osborne	0	65 (2.3)	57 (0.6)	ND	ND	44 (2.1)	43 (1.8)	57 (7.9)	54 (3.4)
	7	86 (2.1)	47 (1.8)	ND	ND	97 (2.5)	48 (3.2)	107 (6.3)	55 (3.3)
	14	67 (4.0)	49 (1.2)	ND	ND	86 (2.8)	60 (1.7)	90 (6.3)	64 (4.4)
	21	54 (2.4)	45 (3.2)	ND	ND	69 (2.0)	54 (1.4)	77 (4.9)	62 (5.4)
	28	58 (1.1)	43 (4.0)	ND	ND	65 (3.7)	45 (2.0)	71 (3.2)	46 (3.3)
	35	56 (1.1)	35 (2.6)	ND	0.1 (0.02)	57 (1.8)	40 (1.2)	62 (3.4)	38 (2.7)
	42	57 (0.4)	41 (1.7)	ND	0.2 (0.04)	55 (0.8)	38 (5.1)	59 (1.1)	43 (2.1)
	49	60 (0.8)	43 (3.1)	ND	0.3 (0.04)	51 (1.6)	34 (3.3)	56 (0.8)	36 (1.8)
	56	58 (2.0)	44 (1.7)	ND	0.3 (0.04)	47 (0.6)	32 (2.9)	53 (1.1)	31 (2.5)
Fairford	0	99 (4.0)	48 (1.6)	ND	ND	91 (1.9)	49 (1.6)	68 (8.1)	39 (2.1)
	7	115 (2.7)	33 (2.4)	ND	ND	128 (1)	41 (3.1)	104 (1.0)	32 (1.8)
	14	103 (2.5)	45 (0.2)	ND	0.3 (0.06)	106 (1)	48 (2.7)	92 (0.6)	40 (0.9)
	21	113 (3.1)	60 (3.5)	ND	2.1 (0.27)	90 (1.4)	45 (1.3)	80 (0.5)	41 (0.6)
	28	149 (3.6)	76 (4.2)	ND	5.6 (0.51)	83 (0.7)	41 (1.8)	78 (3.0)	33 (2.3)
	35	160 (7.1)	92 (4.3)	5.7 (0.48)	8.1 (0.34)	74 (1.0)	31 (0.8)	71 (1.0)	28 (3.7)
	42	166 (3.2)	101 (0.3)	8.8 (0.30)	10.4 (0.1)	69 (1.3)	33 (3.5)	68 (1.6)	32 (4.0)
	49	223 (5.4)	104 (1.4)	11.5 (0.4)	11.2 (0.1)	65 (0.3)	27 (0.7)	71 (4.6)	34 (2.8)
	56	232 (4.1)	120 (6.4)	ND	13.5 (0.5)	62 (0.4)	23 (2.1)	65 (0.5)	31 (2.3)
Balmoral	0	64 (5.3)	58 (1.9)	ND	ND	61 (6.8)	52 (3.8)	39 (8.4)	45 (2.7)
	7	91 (0.5)	40 (3.9)	ND	ND	99 (1.2)	51 (3.4)	89 (2.3)	49 (1.3)
	14	72 (2.7)	50 (1.0)	ND	ND	84 (0.9)	59 (3.3)	73 (1.0)	52 (3.4)
	21	62 (1.1)	51 (4.5)	ND	ND	69 (1.2)	56 (0.8)	63 (1.3)	49 (1.8)
	28	70 (2.6)	48 (0.7)	ND	ND	63 (2.4)	39 (2.3)	62 (0.4)	45 (1.0)
	35	76 (0.7)	51 (4.1)	ND	0.2 (0.01)	56 (1.1)	37 (2.1)	55 (0.4)	37 (3.1)

(Continues)

TABLE 1 (Continued)

Soil	DAF	Warm flooding of frozen soils				Cold flooding of unfrozen soils		Cold flooding of frozen soils	
		Ca	Mg	Fe	Mn	Ca	Mg	Ca	Mg
					—mg L^{-1}—				
	42	87 (1.0)	69 (0.5)	ND	0.5 (0.02)	49 (1.5)	36 (5.0)	51 (0.8)	40 (3.5)
	49	99 (1.1)	71 (3.8)	ND	0.9 (0.04)	48 (0.5)	36 (3.0)	48 (0.6)	35 (1.8)
	56	113 (3.0)	85 (3.4)	ND	1.6 (0.01)	47 (0.9)	31 (3.4)	49 (0.2)	33 (1.0)

Note. Iron and Mn were not detectable under cold flooding. DAF, days after flooding. Standard error of mean in parentheses. ND, not detectable.

all soils flooded under cold temperatures (Supplemental Table S2). Floodwater pH values immediately after flooding ranged around 6.0–6.5 in all soils and increased with flooding time in all soils, with greater increases under warm than cold flooding of unfrozen or frozen soils (Supplemental Table S3).

4 | DISCUSSION

4.1 | Change in soil redox potential with flooding time and its influence on phosphorus release from soils

Relative to warm temperatures, the slower rate and degree of Eh reduction with time of flooding under cold temperatures is expected. Previous studies have suggested that redox reactions in flooded soils are microbially mediated (Ponnamperuma, 1972; Stres et al., 2008) and thus would be slower under cold conditions that impair microbial activity (Jefferies, Walker, Edwards, & Dainty, 2010; Stres et al., 2008). As such, the slower rate and degree of Eh reduction with time of flooding under cold compared with warm temperatures (Figure 1) are consistent with changes in microbially mediated redox reactions, although microbial activity was not measured for the current study. Previous research has indicated that freeze–thaw cycles can disrupt microbial activity (Jefferies et al., 2010), although that does not appear to have happened for this study because the Eh values with flooding were similar in frozen and unfrozen soils.

With the decrease in Eh, the pore water DRP concentrations often increased in most soils; however, in some soils, pore water DRP concentrations either decreased or remained relatively stable. Similar differences in pore water DRP concentrations with DAF among different soils have been previously reported under summer flooding (or room temperature) conditions (Amarawansha et al., 2015; Jayarathne et al., 2016). In the current study, flooding treatment significantly influenced the change in pore water DRP concentration with time (DAF).

As observed in previous studies (Jayarathne et al., 2016; Smith & Jacinthe, 2014), the initial increase in DRP concentrations in pore water and floodwater with DAF, prior to the intensive reduction in soils, is likely caused by dissolution reactions of sparing soluble P compounds. Once the

soils became anaerobic, reductive dissolution reactions may have continued to release P from adsorbed and precipitated pools, as evidenced by the continued increase in pore water and floodwater DRP concentrations in samples flooded under warm temperatures. The influence of reductive dissolution reactions on P release is clearly documented in the literature (Amarawansha et al., 2015; Jayarathne et al., 2016; Maranguit, Guillaume, & Kuzyakov, 2017), with reduction of Fe and Mn compounds playing a significant role through various mechanisms. Reactions may include reductive dissolution of ferric and manganese phosphates as previously reported for calcareous soil from the same region (Jayarathne et al., 2016) as well as reductive dissolution of ferric oxides releasing adsorbed and occluded P (Rakotoson, Rabeharisoa, & Smolders, 2016). Under warm flooding, the decrease in pore water DRP concentrations in Eigenhof soil beyond 28 DAF with a consistent increase in floodwater DRP concentrations may be a result of rapid diffusion of P from pore water to surface floodwater because of the sandy nature of the soil. It was also noted that pore water DRP concentrations exceeded 7 mg L^{-1} by 28 DAF in Eigenhof soil under warm flooding, which may favor reprecipitation reactions with cations such as Ca and Mg, whereas in other soils, pore water DRP concentrations were well below 3 mg L^{-1} during most of the flooding period.

Our results suggest that P release with flooding is mostly through redox-induced reactions. Severe reduced status with very low Eh values under warm flooding resulted in greater P release to floodwater. Microbial reduction of Fe^{3+} to Fe^{2+} takes place when soil Eh falls below ∼100 mV (Gotoh & Patrick, 1974), whereas reduction of Mn^{4+} to Mn^{2+} takes place at higher Eh than Fe^{3+} reduction (Amarawansha et al., 2015; Patrick & Jugsujinda, 1992). A severe reducing environment causing release of Mn^{2+} and Fe^{2+} was not observed for all soils under unfrozen, cold flooding and for all but Fairford soil under frozen, cold flooding; thus, redox-induced P release was not favored under cold flooding of both frozen and unfrozen soils. We can speculate that the low temperatures with or without a freeze–thaw event may have influenced microbially mediated redox transformations through their effect on biological activity, as reported previously (Campbell, Mitchell, Groffman, Christenson, & Hardy, 2005; Lipson, Schadt, & Schmidt, 2002). This is

TABLE 2 Concentrations of floodwater Ca and Mg (mean, $n = 3$) over the flooding period for unfrozen soils flooded under warm and cold temperatures, and frozen soils flooded under cold temperatures

Soil	DAF	Warm flooding of frozen soils Ca	Mg	Cold flooding of unfrozen soils Ca	Mg	Cold flooding of frozen soils Ca	Mg
				mg L^{-1}			
Eigenhof	0	0.5 (0.29)	1.4 (0.33)	0.8 (0.64)	1.4 (0.44)	0.2 (0.06)	0.9 (0.34)
	7	10.9 (1.55)	5.6 (0.19)	3.2 (0.76)	2.7 (0.46)	0.6 (0.19)	1.0 (0.17)
	14	13.1 (0.30)	8.2 (0.10)	8.1 (1.56)	4.3 (0.68)	2.1 (1.00)	2.7 (0.31)
	21	10.5 (0.69)	9.0 (0.28)	5.9 (0.86)	6.1 (0.65)	6.1 (1.84)	4.8 (0.44)
	28	14.4 (2.77)	8.5 (0.38)	14.1 (3.48)	7.6 (1.03)	5.9 (0.32)	5.2 (0.09)
	35	18.4 (2.56)	10.4 (0.37)	17.7 (2.63)	8.1 (0.59)	19.1 (1.58)	6.9 (0.41)
	42	18.0 (1.30)	10.8 (1.10)	15.5 (3.19)	7.6 (0.77)	17.1 (1.38)	8.2 (0.52)
	49	20.5 (0.42)	13.0 (0.49)	20.1 (1.48)	12.3 (1.60)	19.4 (0.24)	8.8 (1.72)
	56	18.5 (2.38)	8.5 (0.37)	16.0 (2.86)	7.8 (1.71)	16.1 (1.97)	10.2 (0.94)
Red River	0	1.9 (0.86)	2.9 (0.77)	2.4 (0.89)	2.3 (0.22)	2.2 (0.51)	3.3 (0.66)
	7	23.3 (1.69)	4.9 (1.11)	12.8 (0.70)	5.5 (0.57)	4.1 (0.50)	3.3 (0.14)
	14	26.6 (3.92)	15.2 (1.35)	19.4 (1.30)	8.8 (0.37)	12.8 (1.51)	7.8 (0.64)
	21	23.6 (2.60)	17.3 (0.83)	17.0 (1.54)	10.5 (0.20)	14.0 (1.19)	8.6 (0.07)
	28	33.3 (0.31)	15.9 (1.58)	28.9 (2.00)	10.9 (1.08)	21.9 (1.97)	11.0 (1.50)
	35	35.5 (0.46)	19.2 (0.89)	24.4 (0.70)	9.9 (0.92)	24.0 (0.70)	9.5 (0.90)
	42	41.2 (1.41)	25.2 (0.85)	25.4 (0.31)	13.8 (0.35)	24.6 (0.44)	14.8 (1.32)
	49	49.1 (1.25)	29.7 (0.39)	26.7 (0.44)	14.0 (0.68)	26.5 (0.87)	12.9 (1.50)
	56	51.6 (0.89)	29.2 (0.34)	25.8 (0.13)	12.1 (1.33)	24.9 (0.98)	12.5 (1.19)
Osborne	0	1.8 (0.50)	3.5 (0.34)	2.6 (1.20)	2.8 (0.75)	0.2 (0.05)	1.1 (0.08)
	7	18.8 (2.12)	10.3 (0.80)	7.7 (1.29)	6.5 (0.42)	12.2 (8.18)	4.8 (0.79)
	14	32.1 (2.50)	26.1 (1.60)	16.0 (3.30)	12.9 (1.47)	15.6 (5.37)	9.9 (1.51)
	21	25.4 (2.50)	25.1 (2.21)	14.7 (0.77)	15.4 (1.11)	14.1 (1.78)	14.0 (1.01)
	28	30.9 (0.96)	26.6 (2.45)	22.4 (2.97)	15.4 (1.50)	23.6 (0.59)	15.5 (0.96)
	35	32.3 (1.03)	21.9 (0.17)	26.1 (0.82)	17.6 (0.47)	25.4 (0.59)	16.3 (0.48)
	42	28.6 (1.04)	24.4 (0.99)	25.5 (0.79)	18.2 (1.29)	24.7 (1.46)	20.2 (0.61)
	49	31.9 (0.11)	28.1 (1.29)	25.9 (1.58)	20.6 (1.95)	29.5 (0.89)	22.1 (1.68)
	56	32.1 (0.69)	22.4 (1.93)	26.8 (0.54)	17.0 (0.71)	23.8 (1.68)	17.8 (1.96)
Fairford	0	0.5 (0.13)	1.7 (0.27)	1.0 (0.43)	1.3 (0.36)	0.2 (0.07)	0.7 (0.18)
	7	19.0 (2.04)	4.1 (0.38)	3.8 (0.27)	1.8 (0.12)	2.0 (0.50)	1.8 (0.40)
	14	32.5 (2.93)	14.9 (1.03)	18.4 (1.17)	6.9 (0.42)	19.1 (3.70)	7.4 (0.16)
	21	33.3 (1.68)	15.4 (0.92)	15.9 (1.54)	8.6 (0.33)	16.8 (1.24)	8.2 (0.23)
	28	42.7 (1.36)	16.6 (0.56)	25.0 (0.60)	10.1 (1.66)	23.0 (0.29)	9.2 (1.40)
	35	46.8 (0.58)	20.9 (1.18)	25.9 (0.30)	11.6 (1.24)	26.3 (1.97)	9.3 (0.97)
	42	52.6 (0.62)	26.6 (0.34)	25.8 (1.15)	11.9 (0.60)	22.5 (2.83)	11.7 (1.11)
	49	63.0 (0.36)	32.7 (3.19)	25.4 (1.81)	13.1 (1.00)	27.1 (1.49)	11.4 (1.25)
	56	69.3 (1.45)	30.4 (1.57)	28.3 (0.44)	11.5 (0.65)	26.2 (0.11)	11.6 (0.40)
Balmoral	0	0.7 (0.48)	2.1 (0.57)	1.8 (1.09)	3.2 (0.93)	0.2 (0.03)	1.2 (0.12)
	7	25.6 (2.69)	5.7 (1.14)	4.1 (1.96)	2.9 (0.54)	3.1 (0.43)	4.2 (0.80)
	14	23.7 (5.16)	21.8 (1.18)	17.3 (1.66)	9.4 (0.95)	11.2 (3.48)	9.4 (0.36)
	21	28.5 (1.06)	22.7 (0.77)	8.1 (0.47)	11.3 (1.54)	12.9 (3.33)	11.7 (1.11)
	28	30.9 (0.30)	22.5 (1.66)	20.6 (0.37)	13.7 (0.47)	21.1 (0.40)	12.5 (0.13)
	35	30.0 (0.41)	20.8 (1.10)	16.1 (2.16)	14.9 (0.62)	16.4 (1.26)	14.4 (1.27)

(Continues)

TABLE 2 (Continued)

Soil	DAF	Warm flooding of frozen soils		Cold flooding of unfrozen soils		Cold flooding of frozen soils	
		Ca	Mg	Ca	Mg	Ca	Mg
				mg L^{-1}			
	42	32.9 (2.22)	25.2 (0.28)	19.5 (1.81)	15.7 (0.88)	17.2 (3.31)	18.3 (0.54)
	49	34.6 (2.34)	30.3 (1.77)	19.9 (1.59)	17.5 (1.01)	19.5 (2.07)	19.0 (0.64)
	56	40.7 (0.46)	29.2 (1.70)	20.5 (0.57)	12.5 (2.40)	16.3 (1.20)	14.1 (0.77)

Note. Iron and Mn were not detectable in floodwater. DAF, days after flooding. Standard error of mean in parentheses.

also clearly indicated by the highly significant ($P < .001$) negative relationship between soil Eh and floodwater DRP concentration under warm flooding in all soils (Supplemental Table S4), whereas under cold flooding of frozen and unfrozen soils the relationship was weaker and often not significant. Consistent with these observations, previous studies reported significantly lower P release from flooded sediments at 7 °C than at 35 C (Sallade & Sims, 1997) and from frozen soils flooded at 4 °C than unfrozen soils flooded at 20 °C (Dharmakeerthi et al., 2019a), suggesting a lower redox-induced P release under spring snowmelt than summer conditions.

When frozen and unfrozen soils were compared under cold flooding, both pore water and floodwater DRP concentrations were often not significantly different. The results indicate that one freeze–thaw event prior to flooding had a negligible effect on P release after flooding. Previous research investigating the effect of freeze–thaw events on P solubility and availability has yielded contradictory results; some studies showed an enhanced P solubility and availability with freezing and thawing (Messiga, Ziadi, Morel, & Parent, 2010; Yevdokimov, Larionova, & Blagodatskaya, 2016; Ziadi, Whalen, Messiga, & Morel, 2013), with multiple freeze–thaw events intensifying the effect (Messiga et al., 2010), whereas in other studies freezing and thawing did not have a significant effect on P availability (Peltovuori & Soinne, 2005; Xu et al., 2011; Zhao, Zeng, & Fan, 2008). However, these previous studies were conducted using field moist or air-dried soil, whereas in the current research soils were flooded and thus had anaerobic conditions for most of the flooding period.

It is also interesting to note that the differences in DRP concentration between warm and cold flooding treatments at a particular DAF were often greater for surface floodwater than for pore water. This is likely because the change in floodwater DRP concentration depends not only on the release of P from soils to pore water but also on the effective diffusion of P from pore water to floodwater. It is well established that the P diffusion coefficient in soil is temperature dependent (Jungk & Claassen, 1997; Mackay & Barber, 1984), with greater values at higher than lower temperatures. Dharmakeerthi et al. (2019a) also reported a lower rate of P diffusion from pore water to floodwater under cold flooding of frozen soils compared with warm flooding of unfrozen soils.

4.2 | Relationships between pore water and floodwater cation concentrations and floodwater dissolved reactive phosphorus concentrations

Calcium and Mg are not redox-sensitive elements; thus, the increase in Ca and Mg concentrations in most soils with DAF is likely due to enhanced dissolution reactions under warm temperatures, as previously observed (Jayarathne et al., 2016). The decrease in Ca and Mg concentrations may be due to the diffusion of cations from pore water to floodwater. Precipitation reactions involving Ca and Mg may also decrease their concentrations in pore water, as previously reported (Amarawansha et al., 2015). Because all soils except Eigenhof soil are alkaline and moderately calcareous, solubility of Ca and Mg is most probably controlled by carbonates in these soils. Thus, we can speculate that pore water Ca and Mg concentrations under cold flooding were substantially less than under warm flooding in these alkaline soils because of the lower solubility of Ca and Mg carbonates under colder temperatures. Pore water Ca and Mg, in general, did not show significant relationships with floodwater DRP concentrations, particularly under cold flooding (Supplemental Table S4).

Significant ($P < .05$) positive relationships were observed between pore water Fe and/or Mn and floodwater DRP concentrations (Supplemental Table S4) for all except Osborne soil. This suggests that reductive dissolution reactions contributed to P release, with simultaneous release of Fe and/or Mn as previously reported for flooded soils under room temperature conditions (Amarawansha et al., 2015; Young & Ross, 2001). Less-than-detectable concentrations under cold flooding of frozen and unfrozen soils are to be expected because reductive dissolution only takes place under low Eh values (Gotoh & Patrick, 1974; Patrick & Jugsujinda, 1992), which were not reached under cold flooding conditions in this study.

Increases in surface floodwater Ca and Mg concentrations with DAF can be attributed to the diffusion of Ca and Mg from pore water to floodwater, with enhanced diffusion rates under warm temperatures. Highly significant positive relationships between floodwater Ca/Mg with floodwater DRP under warm flooding (Supplemental Table S4) suggest dissolution of Ca and Mg phosphates releasing P and cations to soil

solution, which eventually diffuses to overlying floodwater. Similar observations were reported with flooded soils under room temperature conditions (Jayarathne et al., 2016), where dissolution reactions contributed to increases in Ca, Mg, and DRP concentrations in floodwater, particularly during initial stages of flooding. Under cold flooding, however, the relationships between floodwater DRP with pH, Ca, and Mg were weaker and at times not significant (Supplemental Table S4).

4.3 | Pore water and floodwater pH and their relationships to floodwater dissolved reactive phosphorus concentrations

A convergence of soil pH to neutral is the typical response of mineral soils when flooded for a prolonged period (Ponnamperuma, 1972). However, the trend of pH converging to neutrality with flooding was not observed in frozen and unfrozen soils flooded under cold temperatures. Instead, the pore water pH increased with DAF in all soils flooded under cold temperatures. Pore water pH showed a highly significant ($P < .001$) positive relationship with floodwater DRP concentration under warm flooding in the acidic Eigenhof soil, but the relationship was not significant in the soils that are slightly alkaline (Supplemental Table S4). Even though highly significant positive correlations were observed between floodwater DRP and pH under both warm and cold flooding for all soils (Supplemental Table S4), the reactions and processes involved in raising floodwater pH and DRP with flooding may or may not be related and need to be further investigated.

5 | CONCLUSIONS

Redox-induced P release from flooded soils was greater under warm temperatures than under cold temperatures. Stable soil redox potentials under colder incubation temperatures minimized microbially mediated redox reactions, which decreased P release. Other flooding-induced changes in soils, such as pH changes with flooding time, that may influence P dynamics were less apparent under cold flooding of frozen and unfrozen soils compared with warm flooding. One freeze–thaw event prior to flooding under cold temperatures had a negligible effect on redox-induced P release. Our results suggest that the magnitude of P loss from soils to floodwater under cold temperatures with possible freeze–thaw events would be substantially less during the initial spring snowmelt stage than during the latter stages of snowmelt with warmer temperatures.

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SUPPORTING INFORMATION

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