



Drying/rewetting cycles stimulate release of colloidal-bound phosphorus in riparian soils



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ABSTRACT

Drying and rewetting (DRW) events in soils cause the release of molybdate-unreactive dissolved phosphorus (MUP) into soil solutions, which has been historically considered biologically-derived phosphorus (P) from microbial cell lysis. This unreactive P, however, could also represent P bound to soil colloids/nanoparticles, whose releases are also known to be physically stimulated during DRW events. To explore this possibility, two riparian wetland soils (A and B) with contrasting soil P speciation were subjected to three successive DRW cycles in soil columns. Leachates were successively filtrated/ultrafiltrated with 0.45 μm , 30 kDa and 5 kDa pore size membranes to separate the different colloidal/nanoparticulate fractions, in which molybdate-reactive P (MRP), total P (TP), MUP (defined as TP minus MRP), Fe, Al and dissolved organic carbon (DOC) concentrations were measured. For both soils, TP concentrations peaked at the beginning of each rewetting event then decreased rapidly upon leaching. MUP concentrations appeared similar variations as TP, but MRP concentrations remained relatively constant during leaching. Soil B showed larger TP release peaks than soil A and contained different P forms (more MUP and MRP, respectively). In both soils, colloidal/nanoparticulate P was an important fraction (up to 70%) of TP (< 0.45 μm) in leachates immediately after rewetting, then this proportion decreased markedly at the end of each DRW cycle. On average, the proportion of colloidal/nanoparticulate P in TP was much higher in soil B than in soil A (45% vs. 17%). Results suggested that the colloidal/nanoparticulate carriers likely consisted of a mixture of Fe/Al oxides and organic matter, and highlighted a higher colloidal/nanoparticulate P release capacity in soil B with higher soil organic matter content and porosity. In addition, colloidal/nanoparticulate P and truly dissolved MUP had a greater response to DRW events and were exhausted more rapidly than truly dissolved MRP and smaller nanoparticulate P, suggesting potential differences in their sources and production mechanisms. Thus, this study demonstrates that colloidal/nanoparticulate P can represent an important part of the unreactive P fraction released during DRW events in soils and further highlights the importance of the colloidal/nanoparticulate P fraction in the transport and cycling of P in soils and waters.

1. Introduction

Drying and rewetting (DRW) events are one of the most common abiotic stresses that soils experience (Blackwell et al. 2010). Recent studies revealed that DRW events could cause the release of considerable amounts of dissolved phosphorus (P) into soil solutions (Blackwell et al. 2009; Bünemann et al. 2013; Turner et al. 2003; Turner and Haygarth 2001).

Originally, the dissolved P released during DRW events was attributed to physical processes such as disruption of dried soil aggregates upon rewetting followed by the subsequent mobilization of particles and associated P (Powlson and Jenkinson 1976). Recent attention, however, has

focused on the role of soil microbial biomass, with the hypothesis that the released P could derive from microbial cells that are lysed by osmotic shock upon rewetting (Blackwell et al. 2009, 2010, 2013; Bünemann et al. 2013; Turner et al. 2003; Turner and Haygarth 2001). Turner et al. (2003) demonstrated that at least 88% of total dissolved P (TP) released during DRW events originated from lysed bacteria cells. Blackwell et al. (2009) later confirmed this, and found that up to 70% of soil microbial biomass could be killed by the osmotic shock caused by DRW events. More recently, another study on grassland soils in Switzerland also attributed the pulse release of P during DRW events to microbial biomass, through emphasizing at the same time the importance of physicochemical processes (Bünemann et al. 2013).

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Most studies published so far have shown that the dissolved P released during DRW events is mainly molybdate-unreactive, suggesting that it is organic (Blackwell et al. 2009; Turner et al. 2003). Although this molybdate-unreactive nature is consistent with the hypothesis of microbial cell lysis, it does not confirm that the dissolved P released is organic. In fact, dissolved P is operationally defined as P that can pass through 0.45 µm membranes, whose size lies within the range of colloids (1–1000 nm). The molybdate-unreactive P could also consist of inorganic P adsorbed onto, or incorporated into soil nanoparticles and colloids, a possibility that has yet to be fully studied. This knowledge gap is potentially problematic for two reasons: i) an increasing number of studies show that colloids/nanoparticles are important carriers of P in soil solution (Heathwaite et al. 2005; Hens and Merckx 2002; Jiang et al. 2015; Liu et al. 2014; Regelink et al. 2013; Siemens et al. 2008), and ii) drying is known to stimulate the production of soil colloids and nanoparticles, which are then released into soil solutions upon soil rewetting (Klitzke and Lang 2007; Majdalani et al., 2008; Mohanty et al. 2015).

Thus, studying the influence of DRW events on the release of P bound to colloids/nanoparticles into soil solutions is essential. In this study, we estimated this release by ultrafiltrating leaching solutions obtained during laboratory rewetting experiments conducted on two air-dried soils with different organic matter (OM) content, soil microbial biomass, and soil P speciation. The two soils studied come from two riparian wetlands located in an agricultural watershed. We chose these soils because field-monitoring data indicated that both could release P upon rewetting after drying under natural field conditions and the released P was observed to enter the stream network (Dupas et al. 2015; Gu et al. 2017). The same field monitoring data indicated that the two soils responded differently to DRW events in term of the speciation of the P released, with one releasing mainly molybdate-reactive P (MRP) and the other releasing mainly molybdate-unreactive P (MUP) (Gu et al. 2017).

The objectives of the present study were to i) evaluate the role of colloids/nanoparticles in the P release flushes observed during DRW cycles in soils, ii) determine the composition of the colloidal/nanoparticulate P fraction and compare it with truly dissolved MRP and MUP, iii) estimate the influence of soil properties and soil P speciation on the release of colloidal/nanoparticulate P and iv) further constrain the mechanisms that produce the various P species in soils during DRW events. More general objectives were to provide insight into the capacity of riparian zones, where DRW events naturally occur, to release colloidal/nanoparticulate P to surface water, and to assess potential consequences of this release on water eutrophication.

2. Materials and methods

2.1. Soil properties and preparation

The studied soils come from two riparian wetlands in the Kervidy-Naizin watershed, a 5 km² agricultural headwater watershed located in Brittany, western France (48.012° N, 2.835° W). The two wetlands (wetlands A and B) are located in the same topographic position, at the interface between cultivated fields and the stream network. They differ in three main characteristics: i) mean slope (3.6% for A vs. 2.8% for B); ii) mean saturation time (< 10 days for A vs. > 25 days for B), and iii) vegetation cover (unfertilized herbaceous species for A vs. shrubs and trees for B).

Both wetlands were monitored for three years to understand the role of water table fluctuations on P release and P speciation in soil solutions (Dupas et al. 2015; Gu et al. 2017). Results indicated pulses of dissolved P release in phase with the water-table rise and subsequent soil rewetting, particularly in autumn when rainfall resumes after the dry summer season. Stream data indicated that the dissolved P released was transferred to the adjacent stream, making the two wetlands the main sources of dissolved P in the stream at the watershed scale. However,

while the dissolved P released in wetland A consisted of mainly MRP (mean = 72%), that in wetland B was mainly MUP (mean = 63%). This difference in dissolved P speciation was related to differences in soil P speciation (more inorganic P in wetland A vs. more organic P in wetland B), which was interpreted as due to differences in topography and water saturation dynamics (Gu et al. 2017).

Soils from wetlands A and B (soils A and B, respectively) are silt loams, classified as Luvisols. Dupas et al. (2015) and Gu et al. (2017) described their chemical and mineral compositions; here, we provide only the information necessary to interpret our results (Tables S1 and S2, Fig. S1 in Supporting Information). Briefly, soils A and B have similar concentrations of clay, silt and sand (24%, 61% and 14% vs. 29%, 60% and 11%, respectively), N contents (3.7 vs. 4.7 g/kg), C:N ratios (11.2 vs. 12.9) and pH (6.1 vs. 6.2). In contrast, soil A differs from soil B in having lower OM content (70.7 vs. 104.0 g/kg), lower microbial biomass (437.5 vs. 791.7 mg/kg), and higher concentrations of TP (1.21 vs. 0.68 g/kg), Olsen-P (0.08 vs. 0.04 g/kg), and Dyer-P (0.288 vs. 0.048 g/kg). Soil A also has a degree of P saturation 7 times as high as that in soil B (21% vs. 3%) (Table S1). Most importantly, sequential extraction experiments (using Hedley fractionation) indicated that soil P speciation is predominantly inorganic (64% of TP) in soil A, which being predominantly organic (57% of TP) in soil B (Table S2). Soil B's higher OM content, microbial biomass and proportion of organic P are related to wetland B's slower mineralization rate, caused by its flatter topography and longer water saturation time (Gu et al. 2017).

Approximately 1.5 kg of soil (fresh weight) from the surface horizon (0–15 cm) was sampled in the two wetlands with a 75-mm diameter auger in October 2015. Sampling sites lay within 1 m of the lysimeters used by Dupas et al. (2015) and Gu et al. (2017) to monitor P concentrations in soil solution (WetUp-A and WetUp-B sites, respectively, in their studies). The surface horizon was selected because it is more prone to DRW events and field monitoring indicated that it releases more P during DRW events than deeper soil horizons (Gu et al. 2017). After sampling, soils were transferred to the laboratory in plastic bags and air-dried in a clean room (20 days, at 25 ± 2 °C). Soils were sieved to < 2 mm after removing all visible non-soil materials. Soils were then stored in a refrigerator at 4 °C before use.

2.2. Experimental setup and conduct of DRW experiments

PVC syringes 11.5 cm long with a 3.1-cm inner diameter were used as leaching columns. For each soil, three columns were run in parallel, to provide triplicate measurements of P concentration dynamics (Fig. 1). A solution of 10 mM NaCl was used as the influent solution since its ion strength was similar to the natural solutions recovered in these soils. All column tops were sealed with rubber plugs with a hole pierced in the middle. Soft PVC tubes were used to inject the influent solution and leachates were drained through the soil column using a peristaltic pump. All junctions between rubber plugs, columns and soft tubes were tightly sealed with parafilm. Each column was filled with 20.0 g of dried soil. Glass fiber (0.5 g, 8 µm in diameter, Sigma-Aldrich) was placed at the top and bottom of each column to avoid soil mass loss/disturbance during solution injection and leaching.

In total, three consecutive DRW cycles were conducted for each soil (referred to as cycle-1, -2, and -3), using protocols (column volume, rewetting rate, leaching time) comparable to those used in previous studies (e.g. Blackwell et al. 2009; Butterly et al. 2009; Li et al. 2013; Li et al. 2015). Each leaching phase after drying began with a gentle rewetting procedure to raise soil moisture to the soil's field capacity. About 15 ml of solution was required for this purpose, with no or only a few drops of leachate recovered at the bottom of each column. The columns were then sealed, and the leaching phases began. The leaching rate was controlled to 1.0–1.5 ml/min. Draining of leachates generated negative pressure in the column, which created a circulation gradient between the solution reservoir and the column. A water head of ca. 0.5 cm was maintained at the top of the column during the leaching

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