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The effect of sample treatments on the oxygen isotopic composition of phosphate pools in soils

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ABSTRACT

The oxygen isotopic composition of phosphate ($\delta^{18}\text{O}_\text{P}$) has been increasingly used as an effective tracer for the biogeochemical cycling of phosphorus (P) in soils and other environments. However, diverse pretreatment methods (e.g. storage, preparation and extraction) are being used for soil samples. For the uniformity of methods as well as for the comparison of results, it is important to understand if specific treatment methods can compromise original $\delta^{18}\text{O}_\text{P}$ values. Here, Ag_3PO_4 and KH_2PO_4 were used to test whether a modified Hedley sequential extraction and purification procedure can alter the $\delta^{18}\text{O}_\text{P}$ values of phosphate standards. Additionally, to test the effect of sample storage and drying conditions, two types of soils were first processed by using eight different pretreatment methods including sterilizing, storing, drying, and sieving and then the $\delta^{18}\text{O}_\text{P}$ values of each soil P pool were measured. Results indicate that the extraction and purification procedure, drying temperature ($< 0^\circ\text{C}$ to 80°C) and sieving mesh (20 to 100) had no significant effect on the $\delta^{18}\text{O}_\text{P}$ values of P_i (inorganic P) pools, but storage at room temperature (without microbial growth inhibitor- HgCl_2 added) can lead to significant changes in $\delta^{18}\text{O}_\text{P}$ values of almost all P pools. For the two soils studied, the $\delta^{18}\text{O}_\text{P}$ values of P_i pools decrease from H_2O ($\text{H}_2\text{O}-\text{P}_\text{i}$) to NaHCO_3 ($\text{NaHCO}_3-\text{P}_\text{i}$), NaOH ($\text{NaOH}-\text{P}_\text{i}$) and HCl ($\text{HCl}-\text{P}_\text{i}$), and organic P was also found in the extraction solution of HCl . Furthermore, the $\delta^{18}\text{O}_\text{P}$ values calculated from isotope mass balance were different from the measured values suggesting variable extraction of different P pools during single and sequential extraction methods. Collectively these results highlight the need for a unified and standard processing and extraction methods for soil samples to allow meaningful intercomparison of results.

1. Introduction

Phosphorus (P) is an essential nutrient for all life. Low P in soils can limit the growth of plants (e.g. crops), but over fertilization can also lead to P release from soils to aquatic environments and degrade water quality (Sharpley et al., 1994). Therefore, soil P cycling has received widespread attention ((Bunemann, 2015; Hinsinger, 2001; Kruse et al., 2015). Phosphorus in soils can be derived from the weathering of rocks, deposition of aerosols, decomposition of biological debris (e.g. manure and dead body), and chemical fertilizers (Bunemann, 2015; Kruse et al., 2015). Due to the reactivity of P, it exists in different pools in soils and

can exchange from one pool to another (Hedley et al., 1982a; Joshi et al., 2016; Walker and Syers, 1976). Therefore, the sources, bioavailability, and interconversion of different P pools are key issues central to soil P research (Bray and Kurtz, 1945; Hinsinger, 2001; Olsen, 1954). The sequential extraction (Hedley et al., 1982a; Tiessen and Moir, 1993), ^{31}P -NMR (Condon et al., 1985) and enzymatic hydrolysis (He et al., 2004) techniques are commonly used to address these questions.

On the other hand, stable isotope ratios have been widely used to trace the biogeochemical cycling of C, N, and S. Phosphorus has only one stable isotope (^{31}P), but occurs in nature mainly as orthophosphate

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(PO_4^{3-}) containing four O atoms. Because the P–O bond in PO_4^{3-} is very strong and in the absence of enzymatic/biological activity, phosphate oxygen does not exchange isotopes with water under normal temperature and pH ranges in earth surface environments (Blake et al., 1997; Kolodny et al., 1983; Longinelli et al., 1976; O'Neil et al., 2003). However, oxygen isotope exchange is rapid in the presence of enzymes/biological activity (Blake et al., 1998; Blake et al., 1997; Blake et al., 2005; Chang and Blake, 2015; Kolodny et al., 1983; Paytan et al., 2002; Stout et al., 2014). Since different soil P pools could derive from different sources or soil biota exert different degrees of influence, they could have different $\delta^{18}\text{O}_\text{P}$ (Amelung et al., 2015; Roberts et al., 2015; Zohar et al., 2010a; Zohar et al., 2010b). Therefore, $\delta^{18}\text{O}_\text{P}$ can offer an ideal tracer for the identification of sources, bioavailability and inter-conversion of different P pools in soils.

In recent years, there has been a surge in publications involving measurements of phosphate oxygen isotopic composition in soils. However, most of these studies have focused on resin-extraction (Gross et al., 2015; Tamburini et al., 2012; Weiner et al., 2011), dilute HCl-extraction (Angert et al., 2012; Tamburini et al., 2010) or both (Angert et al., 2012; Angert et al., 2011; Tamburini et al., 2012). The resin-extracted P is believed to represent the most bioavailable fraction. Single-step extraction methods such as resin extraction are straightforward, relatively easy to perform and save both time and resource, and still they can provide some useful information. In contrast, there are only a handful of studies employing sequential extraction methods to target more specific P pools for $\delta^{18}\text{O}_\text{P}$ analysis in soils (Joshi et al., 2016; Roberts et al., 2015; Zohar et al., 2010a; Zohar et al., 2010b) or marine/lake sediments (Jaisi and Blake, 2010; Joshi et al., 2015; Markel et al., 1994). One challenge for direct comparisons among these studies is the variability of the sample treatment methods have varied greatly between studies. For example, the published sample storage conditions varied from 4 °C (after drying at 40 °C) (Angert et al., 2012; Angert et al., 2011; Gross et al., 2013; Gross et al., 2015; Weiner et al., 2011) to –20 °C (Gross et al., 2015; Jaisi and Blake, 2010) and –80 °C (Joshi et al., 2015). At the same time, the soil drying temperature is quite variable as well including 24 °C (Zohar et al., 2010b), 30 °C (Roberts et al., 2015), 40 °C (Angert et al., 2012; Angert et al., 2011; Tamburini et al., 2010; Weiner et al., 2011), 60 °C (Gross et al., 2015), and freeze-drying (Jaisi and Blake, 2010; Joshi et al., 2015; Joshi et al., 2016). Unfortunately, the effects of these different sample pretreatment and storage methods on soil $\delta^{18}\text{O}_\text{P}$ values have not been evaluated. We seek here to address these issues by conducting a carefully controlled comparative study including evaluation of the effects of different sample processing and sample storage protocols. We tested the reliability of the extraction-purification procedures by Ag_3PO_4 and KH_2PO_4 , and then the effects of sterilizing, storing, drying and sieving methods on $\delta^{18}\text{O}_\text{P}$ of soil P pools were studied. Finally, a comparison of $\delta^{18}\text{O}_\text{P}$ values between single-step and sequential extraction method was made. Our results provide important insights into the causes and impacts of treatment methods on $\delta^{18}\text{O}_\text{P}$ values of soil P pools.

2. Materials and methods

2.1. Materials

The reagents used in this study include: NaHCO_3 and KH_2PO_4 , both are ultra-pure reagents and produced by Tianjin Guangfu Chemical Co. Ltd.; HCl, $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$, HgCl_2 and $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, all are analytical grade reagents produced by Xilong Chemical Co. Ltd.; NaOH is an analytical grade reagent produced by Sinopharm Chemical Co. Ltd.; AG50W- \times 8 cation exchange resin (H^+ form) and AG1- \times 8 anion exchange resin (OH^- form), both are biotechnology grade produced by Bio-Rad Laboratories Inc., USA.

2.2. Collection and processing of soil samples

For the comparison of soil processing and storage protocols for phosphate O-isotope analyses, two types of soils (soil A and B) were collected and analyzed. Soil A was collected from a cropland near Xiamen University (N: 24°3645', E: 118°3979') on March 30, 2015. This farm is mainly used for the cultivation of strawberry corn and potato and chemical fertilizer is the main source of P_i. Soil B was collected from a wasteland near the soil A site on March 18, 2015. It has not been farmed and fertilized for many years. From both sites, surface soil (0–3 cm) was collected by using a stainless steel scoop and size separated by a 20 mesh sieve ($\Phi = 0.84$ mm).

To preserve original $\delta^{18}\text{O}_\text{P}$ values of the soil P pools, a subset of soil sample was treated with HgCl_2 (0.4%, w:w) solution to prevent microbial growth and extracted immediately following this treatment. For comparison, another subset of soil sample was processed in parallel (without HgCl_2 treatment) to investigate the effect of microbial activity that might alter original isotopic compositions. To investigate the effects of storage time, the third subset of soil sample was stored in a plastic bag (without sealing) at room temperature for 10 and 50 days. All other sub-samples were stored at –20 °C immediately after collection. In order to investigate the effects of drying temperature, a split of sample was taken from the one stored at –20 °C and freeze dried or air dried at 40 °C (for 48 h) or 80 °C (for 24 h) until the weight became constant. In order to investigate the effect of soil size, a subset of freeze-dried and air-dried (at 40 °C) samples were further sieved in 100 mesh ($\Phi = 0.15$ mm). Table 1 shows the detailed treatment methods and terminologies used in this communication.

For all treatment methods, soil P extraction was initiated within 3 h after the specific treatment. Soil A was chosen for all treatment methods of Table 1, except 80 °C-drying, and each method included six replicate samples processed in parallel. Because no 80 °C-drying temperature was used in the literature and we believe the actual drying temperature does not need not as high as 80 °C. So 80 °C-drying method was not conducted on soil A. Soil B was limited to four treatment methods namely 0d sto.- HgCl_2 , 0d sto.-No HgCl_2 , –20 °C sto. & 40 °C dry and –20 °C sto. & 80 °C dry. All treatment included three replicate samples and processed in parallel.

Table 1
Sample treatment methods.

Treatment method	HgCl_2	Storage time (days)	Storage temperature	Drying method	Sieving mesh
0d sto.- HgCl_2	100 mL, 0.4% (w:w)	No	No	No	20
0d sto.-No HgCl_2	No	No	No	No	20
24 °C 10/50d sto.-No HgCl_2	No	10/50	24 °C	No	20
–20 °C sto. & Fre./40/80 °C dry	No	Immediately after collection	–20 °C	Freeze dried or air dried at 40 °C or 80 °C	20
–20 °C sto. & Fre./40 °C dry-100 mesh	No	Immediately after collection	–20 °C	Freeze dried or air dried at 40 °C	100

d = days.

sto. = storage.

Fre. = freeze dried.

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