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Diurnal variations in the content and oxygen isotope composition of phosphate pools in a subtropical agriculture soil

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ABSTRACT

Phosphorus (P) is an essential nutrient that affects crop yields, soil ecosystems and environmental pollution. The oxygen isotope composition of phosphate ($\delta^{18}O_P$) in soil is mainly controlled by P source, temperature, the $\delta^{18}O_W$ of soil water, and biological processes and has been used as a unique tracer for the sources and interconversion of P pools within soil. Understanding if and how the diurnal variation of the environmental factors affects the $\delta^{18}O_P$ of soil P pools is thus indispensable to elucidating the biogeochemical cycling of different P pools and to a better use of soil $\delta^{18}O_P$ as a tracer. Soil samples from the same location with different moisture contents were collected every 3 or 12 h for 7 days for the measurement of the content and oxygen isotope composition of soil water and P pools. The results show that soil temperature, moisture and the $\delta^{18}O_W$ of soil water had clear diurnal variations, with $\delta^{18}O_W$ decreasing with increasing soil moisture. However, both the content and $\delta^{18}O_P$ of different soil P pools did not show a significant diurnal variation. These characteristics indicate that short-term diurnal changes in environmental conditions and biological activities have little effect on the content and $\delta^{18}O_P$ of soil P pools. Therefore, the diurnal sampling time has no effect on the content and $\delta^{18}O_P$ of soil P pools. However, the equilibrium $\delta^{18}O_P$ value represents the complete exchange of oxygen isotopes between phosphate and water. Comparisons of the measured values with the equilibrium value could decipher the degree of P recycling by organisms. This study shows that the mean, not the measured (at the sampling time), temperature and $\delta^{18}O_W$ of soil water should be used to calculate the representative equilibrium $\delta^{18}O_P$ value of soil to avoid bias.

1. Introduction

Phosphorus (P) is essential for crop yields and soil ecosystem; understanding its cycling processes and mechanisms continues to be an important research topic (Bünemann, 2015; Hinsinger, 2001). P in soil mainly derives from rock weathering, biological debris decomposition and chemical fertilizers (Bünemann, 2015; Jaisi and Blake, 2014; Kruse et al., 2015). Soil P exists in different chemical forms and pools and can interconvert between the pools (Hedley et al., 1982; Joshi et al., 2016; Walker and Syers, 1976). Different P pools may have different bioavailability and environmental implications. Therefore, the sources and transformations of P pools are key issues of soil biogeochemistry (Bray and Kurtz, 1945; Hinsinger, 2001; Olsen et al., 1954). However, proper tracers are needed to quantitatively understand these processes. The most direct and widely used tracers are radioisotopes of phosphorus (^{32}P and ^{33}P), which can also provide cycling rate information. However, their short half-lives (14.3d for ^{32}P, 25.3d for ^{33}P) limit their applications to only short-time processes. In recent years, the oxygen isotope composition of phosphate ($\delta^{18}O_P$) has been widely used to trace the source and cycling of P in environmental samples (Davies et al., 2014; Jaisi and Blake, 2014; Paytan and McLaughlin, 2012; Tamburini et al., 2014). Some studies have shown that different P pools in soil have different $\delta^{18}O_P$ values (Joshi et al., 2016; Roberts et al., 2015; Zohar et al., 2010a,b) and that $\delta^{18}O_P$ thus has the potential to trace the sources and interconversions of different soil P pools.

The most important characteristic of the oxygen isotope of phosphate is that phosphate does not exchange oxygen isotopes with water under the natural temperature and pH ranges of the earth surface without enzymatic/biological activity (Blake et al., 1997; Lécuyer et al., 1999; O'Neil et al., 2003). However, oxygen isotope exchange is rapid in the presence of enzymes or biological activity (Blake et al., 1997, 2005; Chang and Blake, 2015; Paytan et al., 2002; Stout et al., 2014).

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When the oxygen isotopes are completely exchanged, the system reaches an equilibrium state and the equilibrium $\delta^{18}O_P$ value ($\delta^{18}O_{P\text{-}Equ}$) is only determined by the temperature and $\delta^{18}O$ value of water ($\delta^{18}O_W$) (Chang and Blake, 2015; Longinelli and Nuti, 1973).

The $\delta^{18}O_P$ value of natural samples is mainly controlled by P sources, biological activity, temperature and $\delta^{18}O_W$. However, these parameters can be highly variable at different time scales. Therefore, it is important to elucidate at what time scales the $\delta^{18}O_P$ of the studied samples can be changed and by what mechanisms. For soil samples, some studies have shown that $\delta^{18}O_P$ can be changed during 3 to 50 days of incubation (Bauke et al., 2017; Gross and Angert, 2015; Jiang et al., 2017; Zohar et al., 2010b). In addition, other ^{18}O-spiked studies have shown that the $\delta^{18}O_P$ values of added phosphate can vary at time scales of days (Johansen et al., 1990; Melby et al., 2013) or months (Joshi et al., 2016; Larsen et al., 1989; Middelboe and Johansen, 1992). Angert et al. (2011) reported seasonal $\delta^{18}O_P$ variations in resin-extractable P in natural soil. The diurnal time scale is much more important because it determines the effects of sampling time (e.g., morning vs. afternoon or daytime vs. evening). The temperature of surface soil has significant diurnal variations, which can further lead to the variations of soil water evaporation, $\delta^{18}O_W$ (Gat, 1996) and biological activity (Jackson et al., 1997; Verhoef et al., 2006). All these factors have the potential to affect $\delta^{18}O_P$. However, systematic studies of the diurnal variations in $\delta^{18}O_P$ signatures of natural soil P pools are lacking.

The objectives of this study were to 1) examine the diurnal characteristics of temperature, moisture content and the $\delta^{18}O_W$ of soil water in a subtropical soil and 2) elucidate how changes in environmental conditions and $\delta^{18}O_W$ affect the content and $\delta^{18}O_P$ of different P pools in the soil. This type of study should provide new insights into deciphering the effects of diurnal variations of temperature, moisture content and the $\delta^{18}O_W$ of soil water on the content and $\delta^{18}O_P$ of P pools in soil, and thus the better use of $\delta^{18}O_P$ as a tracer for P cycling in the environment.

2. Materials and methods

2.1. Materials

The chemical reagents included $NaHCO_3$ and $NH_3{\cdot}H_2O$ (ultrapure, Tianjin Guangfu Chemical Co. Ltd.), HCl, HNO_3, $MgCl_2{\cdot}6H_2O$, NH_4Cl, $(NH_4)_6Mo_7O_{24}{\cdot}4H_2O$, $Mg(NO_3)_2{\cdot}6H_2O$ and $AgNO_3$ (analytical reagents, Xilong Chemical Co. Ltd.), and NaOH (analytical reagents, Sinopharm Chemical Co. Ltd.). Both the cation exchange resin, AG50W-X8 (H^+ form), and the anion exchange resin, AG1-X8 (OH^- form, 200 mesh), were purchased from Bio-Rad (CA, USA).

2.2. Sampling

The soil was collected from a cropland that was fertilized by compound fertilizer and used to cultivate crops (24°3645′ N; 118°3979′ E, Xiamen China). The bedrock is igneous rock. The large particles (e.g., gravels and weeds) were excluded during sampling. After sieved (< 1.4 mm) in the field, the soil was well mixed and placed into 3 shallow bottom-flat pits in discarded cropland for 36 days under field conditions including moisture and temperature. The thickness of the treated soil was approximately 15 cm. During this period, there was heavy precipitation from August 8 to 11 (8.6–29.5 mm) and from August 27 to September 1 (10.8–29.6 mm) and occasional light precipitation events. Then, after ten consecutive sunny days, two pits were watered with tap water with a phosphate concentration below the detection limit; the pits are referred to as "watered-I" and "watered-II", and the watering times are shown in Fig. 1a and b, respectively. The third pit was never watered and is referred to as "non-watered" soil (Fig. 1c). All three pits were sampled at the same time every 3 h, except for the period from 19:00 of Sep. 12, 2015 (19–12, hour-date) to 19:00 of Sep.15, 2015 (19–15), during which they were sampled every 12 h. The watered soils were sampled both before and after watering, except for 19–12, 07–13 and 19–13, which were only sampled before watering. There was no precipitation during the sampling period. The surface soil temperatures were also measured by inserting a temperature sensor (TR-6D, Shunkeda Co. Ltd.) into the soil (~1 cm depth) during sampling. Each time, ~300 g soil was successively sampled from the outside of the soil layer (~3 cm) by a stainless steel scoop. The samples were collected in clean plastic bags and processed in the laboratory within 1 h after sampling. Approximately 100 g of each sample was used to extract water for the measurement of moisture content and $\delta^{18}O_W$. The remaining samples were stored at −20 °C and then freeze-dried and sieved (< 150 μm) to measure the contents and $\delta^{18}O_P$ values of different P pools.

2.3. Soil water extraction and $\delta^{18}O_W$ measurement

The soil water extraction method was modified from the distillation-purging method of Ignatev et al. (2013). Approximately 2–3 mL water was extracted from each sample and airtightly frozen and stored until the analysis of $\delta^{18}O_W$. The content of soil moisture (%, w:w) was calculated by dividing the mass difference before and after the water extraction with the original mass. $\delta^{18}O_W$ was measured by the CO_2-H_2O equilibrium method (Epstein and Mayeda, 1953) using the Gas-Bench – isotopic ratio mass spectrometer (Thermo Fisher, Delta V advantage). Three laboratory reference waters (−6.6, −4.6 and 0.2‰) were measured after every 10 samples to scale the measurement. The laboratory reference waters were scaled by IAEA VSMOW (0‰) and IAEA SLAP (−55.50‰) standards.

2.4. Content and $\delta^{18}O_P$ measurement of P pools

P pools were extracted by the method of Tiessen and Moir (1993), which was modified from Hedley et al. (1982). In brief, 20 g of dried soil was sequentially extracted by H_2O (loosely sorbed P), 0.5 M $NaHCO_3$ (sorbed P), 0.1 M NaOH (Fe- and Al-bound P) and 1 M HCl (apatite and occluded P). The volume of each extractant was 1000 mL. The solution was separated from the residual solids by centrifugation (3500 rpm for 15 min). Except for the extraction of H_2O, residual solids of all other extractants were rinsed by 0.5 M NaCl to wash out the re-adsorbed P. Each extracted solution and the corresponding rinse solutions were combined and 20 mL was taken out to measure the inorganic P (P_i) and total P (TP) concentrations by the method of Tiessen and Moir (1993) and Koroleff (1983), respectively. Organic P (P_o) in each extracted phase was calculated by the difference between TP and P_i. After the extraction of 1 M HCl and the rinsing of the residual solid by 0.5 M NaCl, an aliquot of the residual solid (0.4 g) was ashed at 550 °C for 2 h to decompose the organic P compounds. Then, the ashed samples were extracted with 20 mL of 1 M HCl for 16 h to measure the content of the residual organic P_o (R-P_o) (Ruttenberg, 1992).

For $\delta^{18}O_P$ analysis, the extracted P_i needed to be purified by a series of steps and finally converted into pure silver phosphate (Ag_3PO_4) (Jiang et al., 2017). Briefly, magnesium-induced coprecipitation (MAGIC) was used to concentrate P_i. Next, P_i was purified by sequential precipitation and dissolution of ammonium phosphomolybdate (APM) and magnesium ammonium phosphate (MAP). Then, the solutions were treated with cation resin (AG50W-X8, BIO-RAD) followed by anion resin (AG1-X8, BIO-RAD) to remove cations and anions, respectively. The purified P_i was finally converted to Ag_3PO_4 by the ammonia volatilization method (Firsching, 1961). The $\delta^{18}O_P$ values were measured by HTC-IRMS with the pyrolysis temperature, GC temperature and helium carrier gas flow rate of 1380 °C, 85 °C and 80 mL/min, respectively (Chen et al., 2015; Yin and Chen, 2014). The $\delta^{18}O$ values were calibrated by two international oxygen isotope reference materials: the IAEA-601 benzoic acid ($\delta^{18}O_{VSMOW} = 23.2‰$) and Ag_3PO_4 ($\delta^{18}O_{VSMOW} = 21.7‰$, Elemental Microanalysis Ltd.). The

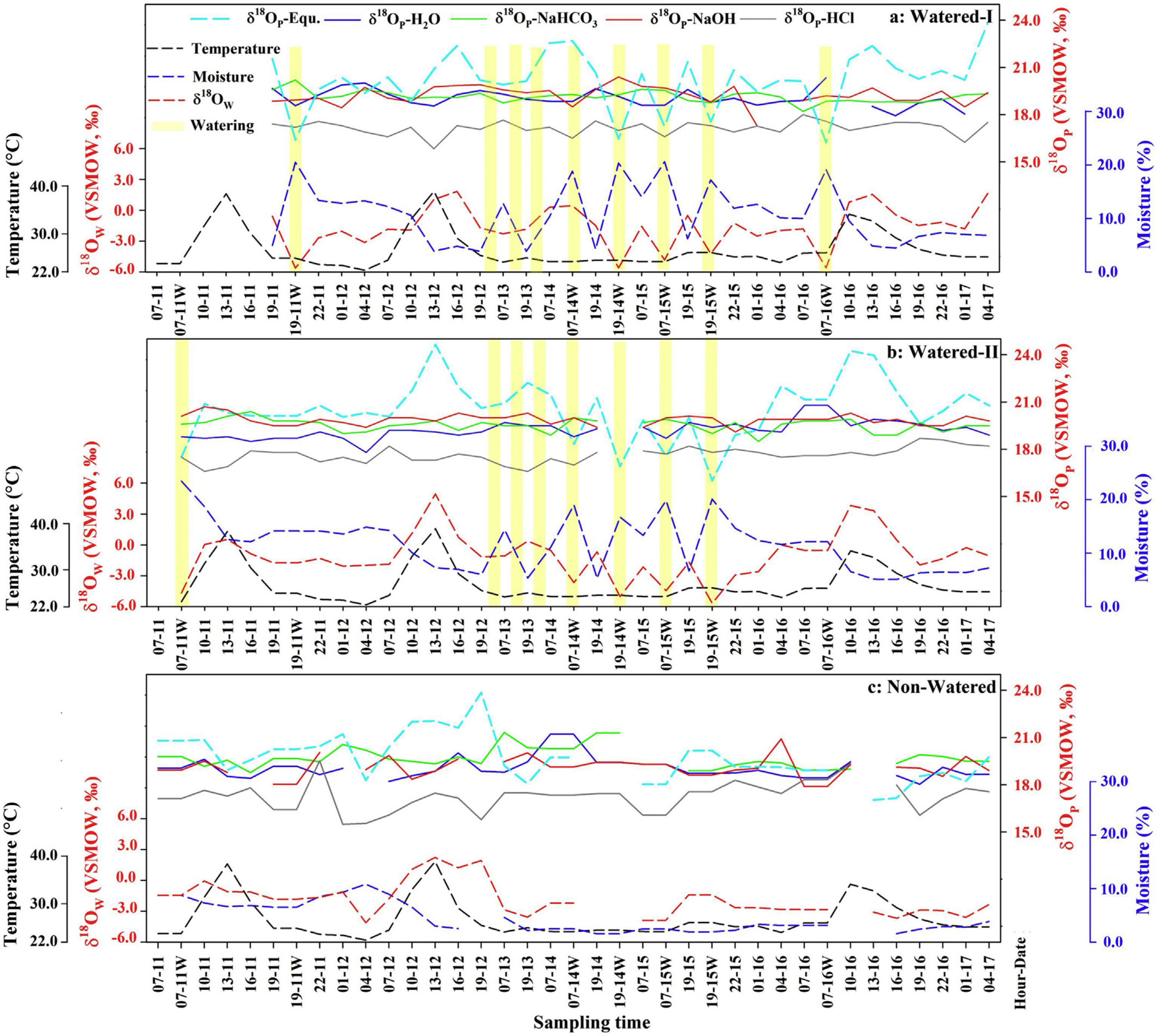

Fig. 1. Diurnal characteristics of temperature, soil moisture, $\delta^{18}O_W$ and $\delta^{18}O_P$ of soil P_i pools. $\delta^{18}O_{P\text{-}Equ}$ was calculated by the equation of Chang and Blake (2015) using the measured soil temperature and $\delta^{18}O_W$ of soil water at the sampling time. "W" denotes sampling immediately after watering. For the samples that were not watered, the value at the sampling time with "W" is identical to that of the same sampling time without "W".

measurement error was ± 0.3‰.

3. Results and discussion

3.1. Diurnal variations of temperature, moisture content and $\delta^{18}O_W$

3.1.1. Temperature

Soil temperature showed clear diurnal variations (Fig. 1). From 7:00 to 13:00, the temperature steadily increased and reached to the peak of ~38 °C at ~13:00. Then, it steadily decreased from 13:00 to 19:00. From 19:00 to 07:00 of the next morning, the temperature had only a small decrease.

3.1.2. Soil moisture content

The soil moisture content ranged from 1.6 to 23.6%. From 07:00 to 13:00, the soil moisture steadily decreased with increasing temperature (Fig. 1). However, from 13:00 to 16:00, the temperature steadily decreased while the soil moisture was stable or still had a small decrease. Soil moisture reached the minimum value at 16:00 and then had a small increase at night except for the samples that were watered at 19:00; for the latter case (19:00 watering), the moisture has no clear changes at night. Watering the sample could increase the soil moisture, and the moistures of the watered-I and watered-II soils were thus higher and had larger variation ranges than that of the non-watered soil (Fig. 1).

These results were consistent with the general diurnal distribution pattern of soil moisture previously reported (Jackson, 1973; Jackson et al., 1997; Raz-Yaseef et al., 2012). The daytime decrease of soil moisture is the result of evaporation, whereas the small nighttime increase is the result of upward water fluxing (Jackson, 1973) and/or the absorption of dew deposition water (Agam and Berliner, 2004). Because the samples watered at 19:00 had high moisture, which could dwarf the upward flux and/or absorbed water, their moisture did not show a clear increase at night.

3.1.3. $\delta^{18}O_W$ of soil water

The $\delta^{18}O_W$ variations of watered-I and watered-II soils were larger than those of non-watered soil (Fig. 1); with their mean values of −1.62 ± 2.03‰ ($n = 32$), −1.05 ± 2.32‰ ($n = 34$) and −1.83 ± 1.74‰ ($n = 28$), respectively. $\delta^{18}O_W$ generally increased with increasing temperature from 7:00 to 13:00 but decreased with the decrease of temperature from 13:00 to 19:00. In addition, $\delta^{18}O_W$ was fairly stable from 19:00 to 07:00 the next morning. Therefore, the variation trend of $\delta^{18}O_W$ is generally similar to that of temperature, but is the reverse of that of soil moisture. As observed for soil moisture, the $\delta^{18}O_W$ variation range of the non-watered soil was smaller than those of the watered-I and watered-II soils.

The $\delta^{18}O_W$ of soil water has been widely used in different studies including in ecology (Dawson et al., 2002; Ehleringer and Dawson, 1992), hydrology (Gat, 1996; Laudon et al., 2004) and soil evaporation calculations (Costelloe et al., 2014; Fontes et al., 1986; Hsieh et al., 1998), although values of $\delta^{18}O_W$ have been shown to be highly variable. Due to the preferential evaporation of isotopically light $H_2{}^{16}O$, the $\delta^{18}O_W$ of remaining soil water will increase (Gazis and Feng, 2004; Hsieh et al., 1998). The change of soil moisture can reflect the degree of soil evaporation. Therefore, temperature, moisture content, and the $\delta^{18}O_W$ of soil water all had an evident diurnal variation, and $\delta^{18}O_W$ had a similar and reverse trend with those of temperature and moisture, respectively (Fig. 1). Thus far, studies of the diurnal change in the $\delta^{18}O_W$ of soil water have been limited. To the best of our knowledge, there is only one semidiurnal study (06:30 to 18:30) of soil $\delta^{18}O_W$, Hu et al. (2014); although their results indicated different $\delta^{18}O_W$ variation patterns among different sampling dates, $\delta^{18}O_W$ generally increased from early morning to late afternoon. Our results in this study also show that soil $\delta^{18}O_W$ increased in the morning but decreased in the afternoon and was stable at night (Fig. 1).

In general, isotopically light water molecules, e.g., $H_2{}^{16}O$, should be preferentially evaporated. Therefore, when the moisture was stable or had a small decrease in the afternoon, the $\delta^{18}O_W$ should be stable or

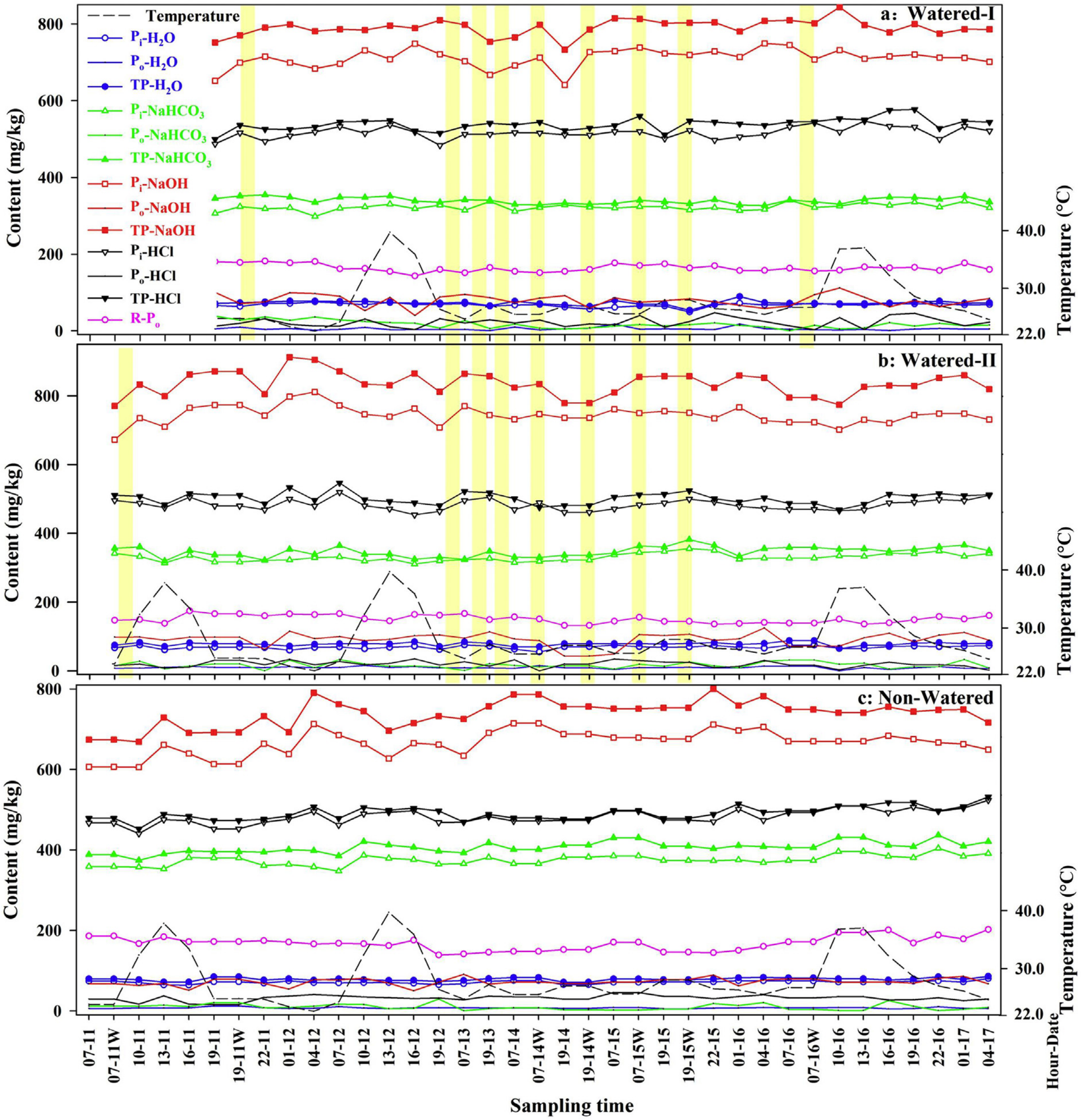

Fig. 2. Diurnal characteristics of the contents of P pools in soil. "W" denotes sampling immediately after watering. For the samples that were not watered, the data at the sampling time with "W" is identical to that of the same sampling time without "W".

continue to increase in the afternoon if the soil moisture was only controlled by evaporation (i.e., no other water was added). However, Fig. 1 shows a clear decrease of $\delta^{18}O_W$ in the afternoon, which suggests that new water with a lower $\delta^{18}O_W$ value must have been added to the soil. If the $\delta^{18}O_W$ of the new water was lower than that of evaporated water and the amount of new water was equal or smaller than that of evaporated water, the result can be stable or small decreases in the moisture and clear decreases in soil-water $\delta^{18}O_W$. Our hypothesis is that this “new” water with lower $\delta^{18}O_W$ may be the water (vapor) that upwelled from the deep soil (Jackson, 1973). Indeed, Fig. 1c shows that the soil moisture of non-watered soil did not continue decreasing with time, nor did $\delta^{18}O_W$ continue increasing with time; these may represent a balance between evaporation and upward water (vapor) fluxes.

3.2. Phosphorus content

3.2.1. The content of P pools

For each extractant, P_i was the main constituent of TP (comprising > 85%), and TP and P_i thus had similar trends (Figs. 2 and 3; Table A1). For each soil, the P_i contents followed the order of NaOH-P_i > HCl-P_i > $NaHCO_3$-P_i > H_2O-P_i, and the content of R-P_o was between those of $NaHCO_3$-P_i and H_2O-P_i. Similar to P_i, NaOH-P_o and H_2O-P_o were also the maximum and minimum organic phosphorus pools, respectively, for all three soils. However, for the watered-I and watered-II soils, the proportion of HCl-P_o (watered-I: 18.0%; watered-II: 14.1%) was slightly higher than that of $NaHCO_3$-P_o (watered-I: 14.0%; watered-II: 11.7%), and for the non-watered soil, the proportion of HCl-P_o (8.5%) was clearly lower than that of $NaHCO_3$-P_o (26.2%) (Figs. 2 and 3).

Phosphate in acidic agricultural soil can be transformed to moderately stable Fe-P and Al-P (Buehler et al., 2002; Liu et al., 2015; McLaughlin et al., 2011) or more stable Ca-P (Bell and Black, 1970; Sato et al., 2005) via long-term transformation and cycling. Therefore, NaOH-P_i and HCl-P_i are generally the major pools in acidic soils (Beauchemin et al., 2003; Buehler et al., 2002; McLaughlin et al., 2011). Because the labile P can be easily assimilated by organisms, transformed into stable pools (Bünemann et al., 2004; Daroub et al., 2000), or lost by runoff (Hooda et al., 2000; Vadas et al., 2005), the contents of H_2O-P_i and $NaHCO_3$-P_i were lower than those of NaOH-P_i and HCl-P_i.

The main constituents of soil P_o are phosphomonoester, phosphodiester and phosphonate (Stewart and Tiessen, 1987; Turner et al., 2005). Except for inositol hexaphosphate (IP_6), all other P_o molecules can be easily hydrolyzed by phosphatases in soils (Condron et al., 2005; McLaren et al., 2015). Therefore, for each extractant, P_o only constitutes a minor proportion of TP (< 15%) and inositol hexaphosphate may be the main constituent of P_o. Since inositol hexaphosphate can be easily and strongly adsorbed by Fe/Al and their oxides in acidic soils (Giles et al., 2011; Turner et al., 2002), NaOH-P_o was the main constitute of P_o.

3.2.2. Diurnal variations of P pools

Fig. 2 shows no obvious diurnal variations in P_i, P_o or TP for all extractants of the three soils. Soil temperature and moisture, on the other hand, had clear diurnal variations (Fig. 1), which should affect biological activities. Although most studies have suggested that biological activities had a significant impact on the content and transformation of P pools in soil (Daroub et al., 2000; Hedley et al., 1982; Liu et al., 2015), the diurnal pattern of soil P pools remains poorly understood. Vandecar et al. (2009) and Wood et al. (2016) found that the content of Bray-extractable P in wet/humid tropical forest soils had some diurnal variations, with different patterns during different days. Our results showed that the content of P pools had no evident diurnal variations for all the samples. The cause of these differences is unclear. Note that the soil samples of our study were freeze-dried before extraction. Some studies have shown that drying and rewetting of soil can increase the water extractable P (mainly organic P) (Blackwell et al., 2013; Turner and Haygarth, 2001). Therefore, the contents of H_2O-P, $NaHCO_3$-P and even NaOH-P could be overestimated using this procedure.

3.3. Oxygen isotope composition of phosphate

3.3.1. Variations in $\delta^{18}O_P$ of P_i pools

For all the soils and P_i pools, their $\delta^{18}O_P$ values were relatively stable (Fig. 1), and for each P_i pool, their $\delta^{18}O_P$ values were similar between soil samples (Fig. 4a). The mean $\delta^{18}O_P$ values of H_2O-P_i, $NaHCO_3$-P_i, NaOH-P_i and HCl-P_i in all three soils were 19.1 ± 0.6‰ ($n = 91$), 19.4 ± 0.5‰ ($n = 93$), 19.5 ± 0.6‰ ($n = 90$) and 17.4 ± 0.7‰ ($n = 94$), respectively. The equilibrium values were calculated by the revised equation of Chang and Blake (2015) based on the soil temperature of the sampling time and the measured $\delta^{18}O_W$ of soil water. As shown in Fig. 1, most of the calculated equilibrium $\delta^{18}O_P$ values had diurnal variation and were slightly higher than those of the

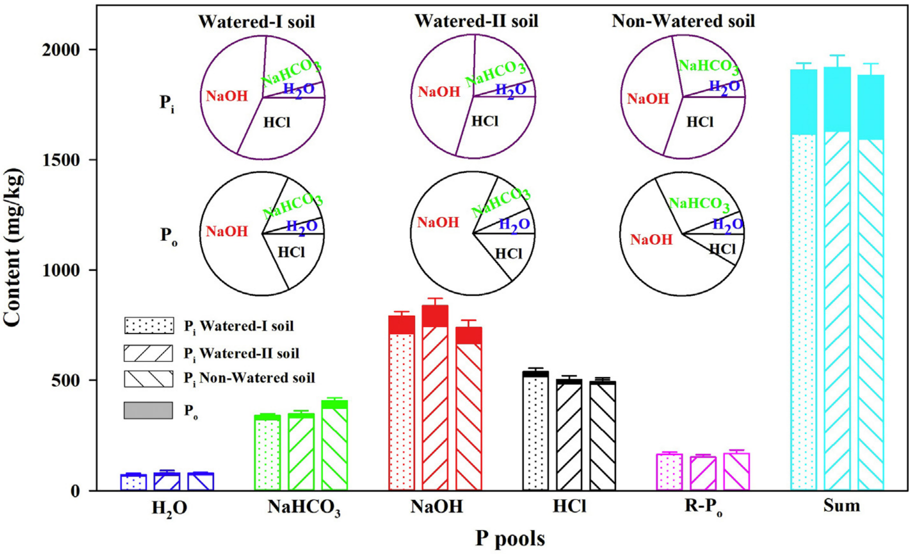

Fig. 3. The mean contents and their fractions of P pools in three soils. The error bar represents the standard deviation of the mean P contents for each soil sampled at different times.

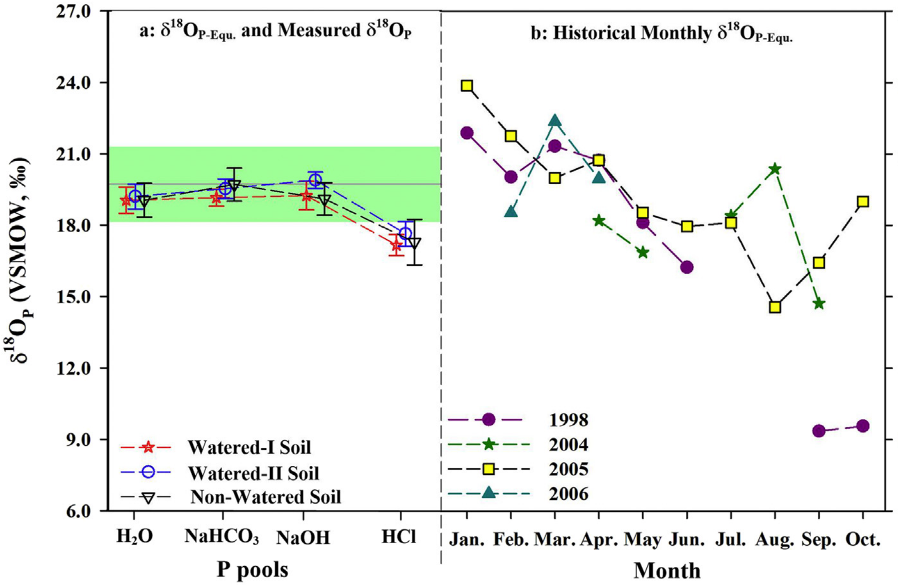

Fig. 4. The mean $\delta^{18}O_P$ of P_i pools in three soils and the calculated average $\delta^{18}O_{P\text{-}Equ}$. a: The symbols represents the results of three soils and the shadow represent the average $\delta^{18}O_{P\text{-}Equ}$ calculated by the measured temperature and $\delta^{18}O_W$ of the non-watered soil. The error bar represents the standard deviation of the mean $\delta^{18}O_P$ for each soil sampled at different times. b: The monthly average $\delta^{18}O_{P\text{-}Equ}$ calculated by the monthly average air temperature and $\delta^{18}O_W$ of precipitation (Cai et al., 2000; Chen et al., 2010). The data were appended in Table A2. All $\delta^{18}O_{P\text{-}Equ}$ values were calculated by the equation of Chang and Blake (2015).

measured values. The $\delta^{18}O_{P\text{-}Equ}$ variations of watered-I and watered-II soils were larger than those of the non-watered soil (Fig. 1); with their mean values of 20.2 ± 1.8‰ (n = 32), 20.7 ± 1.9‰ (n = 34) and 19.7 ± 1.6‰ (n = 28), respectively.

The $\delta^{18}O_P$ values of HCl-P_i were lower than those of other P_i pools but higher than the reported $\delta^{18}O_P$ value of igneous rock (~5.3–7.0‰, Taylor, 1968; Mizota et al., 1992) and the chemical fertilizers (13.8‰ and 12.6‰) applied by local farmers (Lei, 2018). A similar trend was also found by other studies (Angert et al., 2012; Jiang et al., 2017; Joshi et al., 2016; Roberts et al., 2015). Since the HCl-P_i mainly represents apatite and occluded P_i (Hedley et al., 1982), the oxygen isotope data imply that igneous apatite was not the only source of HCl-P_i and that microbially-reworked phosphorus (e.g., available P) should also contribute to the HCl-P_i pool.

The $\delta^{18}O_P$ values of H_2O-P_i, $NaHCO_3$-P_i and NaOH-P_i were similar, which implies that H_2O-P_i, $NaHCO_3$-P_i and NaOH-P_i can interconvert each other. Since we did not use the isotope-labeled method and did not detect $\delta^{18}O_P$ variations for all P_i pools during the sampling period (seven days), we cannot provide further information about the time needed for the complete interconversion. Several previous $\delta^{18}O_P$ studies have shown that the $\delta^{18}O_P$ pattern was not the same among these three P_i pools. Some studies found similar $\delta^{18}O_P$ values among the H_2O-P_i, $NaHCO_3$-P_i and NaOH-P_i pools (Bi et al., 2018; Joshi et al., 2016; Lei, 2018), but others showed that the $\delta^{18}O_P$ values of H_2O-P_i and $NaHCO_3$-P_i were higher than that of NaOH-P_i (Jiang et al., 2017; Roberts et al., 2015; Zohar et al., 2010b). Microbial P_i is more active and has the potential to influence other P_i pools in soil. Two related studies so far have shown that the $\delta^{18}O_P$ values of microbial P_i were either similar to that of resin-extractable P_i (Tamburini et al., 2012) or lower than those of resin-extractable P_i and NaOH-P_i (Tamburini et al., 2018). The effect of microbial P_i on the $\delta^{18}O_P$ of other P_i pools is thus unclear. A ^{33}P-spike study showed that the resin-P_i of some soils (continuous rice and rice green manure rotation) could be quickly converted into $NaHCO_3$-P_i and NaOH-P_i, whereas the conversion of other soils (native savanna and grass-legume pasture) was much slower (Buehler et al., 2002). Therefore, soil types can also affect the $\delta^{18}O_P$ distribution pattern of P_i pools.

3.3.2. Diurnal variation of $\delta^{18}O_P$

Fig. 1 shows that $\delta^{18}O_P$ had no clear diurnal variation for any P_i pools of the three soils. As discussed above, temperature, equilibrium $\delta^{18}O_P$, moisture content and $\delta^{18}O_W$ of soil water all had obvious diurnal variations. Therefore, we can conclude that for natural soil samples studied here, diurnal changes in the environmental conditions did not result in variations in $\delta^{18}O_P$ of P_i pools in the soil.

Pyrophosphatase (PPase) is the only enzyme currently known that can lead to oxygen isotope exchange between phosphate and water (to reach an equilibrium) (Blake et al., 2005; Chang and Blake, 2015). The exchange can be detected within 34 min and is complete within 818 min (Chang and Blake, 2015). PPase mainly exists in cells, but some studies have also reported PPase occurrence in soils (Baligar et al., 1991; Tabatabai and Dick, 1979). However, the non-diurnal variation of $\delta^{18}O_P$ indicates that pyrophosphatase activity, either outside or inside the cell, is not sufficiently large to change the $\delta^{18}O_P$ of soil P_i pools at a diurnal time scale. Huang (2016) found that the $\delta^{18}O_P$ of plant leaves had a clear diurnal variation that resulted from high enzyme (e.g., pyrophosphatase) activity in the leaves and thus the quick response of $\delta^{18}O_P$ to changes in temperature and $\delta^{18}O_W$. Moreover, because water is both the medium and participator of the oxygen isotope exchange, soil with relatively low water content (compared with leaves) thus may also limit isotope exchange rates.

The uptake and release of P_i by organisms and hydrolysis of organic phosphorus can also increase or decrease $\delta^{18}O_P$. If these processes balance each other, the diurnal variation can also be absent. However, these processes could also affect P_i content. Nonetheless, Fig. 2 shows that the P_i content also has no diurnal variation in any P_i pools. The action of these processes in the soil is less likely to keep P_i contents and $\delta^{18}O_P$ values constant at the same time. Therefore, it is likely that the diurnal variation of these processes is not sufficiently strong to alter the content and $\delta^{18}O_P$ values of soil P_i pools. As discussed in Section 3.2.2, the extraction of dry soil may overestimate the labile P. Some studies attributed the additional P to the direct release of microbial biomass (e.g., Turner and Haygarth, 2001). Based on the results of our leaf studies (Huang, 2016), the $\delta^{18}O_P$ of microorganism should have diurnal variations. Therefore, if the amount of P_i released from the microbial biomass is sufficiently large, diurnal $\delta^{18}O_P$ variations in H_2O-P_i, $NaHCO_3$-P_i and even NaOH-P_i can occur. Overall, the absence of their variations shown in Fig. 1 suggests that this source of P_i is insignificant.

3.4. Implications for the calculation of $\delta^{18}O_{P\text{-}Equ}$

The calculation of equilibrium $\delta^{18}O_P$ values requires both $\delta^{18}O_W$ and temperature data. At present, both the measured $\delta^{18}O_W$ of soil water (Bi et al., 2018; Joshi et al., 2016; Roberts et al., 2015) and the average $\delta^{18}O_W$ of monthly or annual precipitation (Amelung et al., 2015; Angert et al., 2011; Chang et al., 2010; Tamburini et al., 2012) are being used. For the temperature, the measured soil temperature

(Amelung et al., 2015; Joshi et al., 2016; Tamburini et al., 2012), monthly average soil temperature (Angert et al., 2011, 2012; Bi et al., 2018; Granger et al., 2017), annual average soil temperature (Joshi et al., 2016; Roberts et al., 2015) and annual average air temperature (Chang et al., 2010) have all been used. However, our results showed that the soil temperature, $\delta^{18}O_W$ of soil water, and calculated $\delta^{18}O_{P\text{-}Equ}$ all had significant diurnal variations, whereas the $\delta^{18}O_P$ of all P_i pools did not show clear diurnal variations (Fig. 1). Therefore, the use of measured temperature and the $\delta^{18}O_W$ of soil water at the sampling time is not appropriate for the calculation of $\delta^{18}O_{P\text{-}Equ}$. Instead, the average $\delta^{18}O_W$ and temperature should be used. In addition, the best time range that should be used to calculate the average values of temperature and $\delta^{18}O_W$ is the least time that is required to change the $\delta^{18}O_P$ of H_2O-P_i by the variation of temperature and/or $\delta^{18}O_W$.

Because the surface soil temperature is similar to that of the air temperature, both can be used to calculate $\delta^{18}O_{P\text{-}Equ}$. Since the isotopically light $H_2{}^{16}O$ is preferentially evaporated, the $\delta^{18}O_W$ of soil is usually heavier than that of the precipitation. The shadow of Fig. 4a shows the average $\delta^{18}O_{P\text{-}Equ}$ calculated by the measured temperature and $\delta^{18}O_W$ of the non-watered soil. Fig. 4b and Table A2 show the monthly average $\delta^{18}O_{P\text{-}Equ}$ calculated by the monthly average air temperature and the precipitation $\delta^{18}O_W$ of Xiamen in 1998, 2004, 2005 and 2006 (Cai et al., 2000; Chen et al., 2010). Although from Fig. 4 we still cannot tell if the $\delta^{18}O_W$ of soil water or the $\delta^{18}O_W$ of precipitation should be used to calculate $\delta^{18}O_{P\text{-}Equ}$, the $\delta^{18}O_W$ of soil microorganism body water may be the best choice since the oxygen isotope exchange between water and phosphate mainly occurs inside the organism. This deserves further study.

4. Conclusions

1) The $\delta^{18}O_W$ of surface soil water has an evident diurnal variation, showing a similar variation trend with temperature but a reverse trend with soil moisture.
2) The content and $\delta^{18}O_P$ of soil P pools have no evident diurnal variations, suggesting that the sampling time, short time watering and diurnal variation of environmental conditions and biological activities have little effect on soil $\delta^{18}O_P$ at a diurnal time scale.
3) The single point temperature and $\delta^{18}O_W$ of soil water are not representative for the calculation of equilibrium $\delta^{18}O_P$ values and may cause biased $\delta^{18}O_{P\text{-}Equ}$ data.

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Declaration of interest

None.

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