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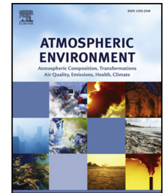

Isotopic signature of atmospheric phosphate in airborne tree pollen

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ABSTRACT

In non-desert areas primary biological aerosol particles, including pollen, are an important source of phosphorus (P). During the flowering season large quantities of pollen from wind pollinated trees, are observed in boreal and temperate lakes and forest, which contain high P concentrations. However, there are different estimations for the contribution of pollen to the atmospheric P cycle, and there is no accepted method for the identification of phosphate from pollen origin. In this study we measured the P concentrations in pollen from various locations. In addition, the oxygen stable isotopes ratio in phosphate ($\delta^{18}O_P$) was measured for the first time for both the bioavailable pool (P extractable by anion-exchange resin), and for the HCl-extractable pool. The resin and HCl-extractable $\delta^{18}O_P$ values were on average (± SE) 25.6 ± 3.4‰, and 25.8 ± 2.9‰, which are distinctively higher than mineral dust $\delta^{18}O_P$ values (average 22.1 ± 0.7‰). Pollen unique isotopic signature in phosphate can be used as a tool to identify P in aerosols from pollen origin, and hence to better understand the atmospheric P cycle. The high P concentrations found in this study (average (± SE) resin-P and HCl-P values were 0.67 ± 0.10 and 0.75 ± 0.9 mg P g^{-1}), confirm that pollen is important to the atmospheric P budget, and can help to solve the uncertainties regarding its role in the global P cycle.

1. Introduction

Atmospheric aerosol deposition is a significant source of phosphorus (P) in many terrestrial and marine ecosystems worldwide (Campo et al., 2001; Jassby et al., 1994). While mineral dust is considered the most dominant airborne P source to ecosystems downwind major deserts, in non-dusty areas anthropogenic sources (Du et al., 2016) and primary biogenic aerosols are also an important source of P (Mahowald et al., 2008; Tipping et al., 2014; Rösel et el., 2012). Pollen is one of the components of primary biogenic aerosol particles which also include bacteria, fungal and fern spores, viruses, and fragments of animals and plants (Artaxo and Hansson, 1995; Despres et al., 2007). Pollen grains have high nutrient concentrations, which could contribute a significant part of the nutrient dynamics in terrestrial and aquatic ecosystems (Stark, 1972; Doskey and Ugoagwu., 1989). In addition pollen of anemophilous (wind pollinated) plants is usually dispersed in large amounts over a short time period and can be transported over long distances during favorable meteorological conditions because of their aerodynamic qualities for wind dispersion (Doskey and Ugoagwu, 1989; Lee et al., 1996a).

Estimating the magnitude of the atmospheric P flux to ecosystems can be challenging, since existing data of biogenic aerosols is lacking leading to uncertainties in global models (Bristow et al., 2010; Krishnamurthy et al., 2010; Mahowald et al., 2008; Wang et al., 2015, 2017). For example, different studies provide different P concentrations in pollen and annual pollen assessments also vary between studies (Hoose et al., 2010; Jacobson and Streets, 2009). Additionally, the contribution of biogenic particles to the global P budget is yet unclear and studies give different estimations (Mahowald et al., 2008; Wang et al., 2015). Furthermore, there is no accepted method for measuring the contribution of pollen to the atmospheric P cycle. Since little information regarding biogenic P is currently available at the global scale, accurate estimations of the impacts of the aerosol-P on the productivity of ecosystems can be enhanced by reliable data on the contribution of the biogenic aerosols to the P budget (Wang et al., 2017).

Here we suggest a method, for tracing and identifying biogenic atmospheric P using oxygen stable isotopes in phosphate. Since the relative abundance of oxygen isotopes in phosphate varies naturally, accurate measurements of the oxygen isotope ratios in phosphate, $\delta^{18}O_P$, provide a unique mean of tracing the P sources. The use of $\delta^{18}O_P$ as a tracer of P is based on the chemical stability of the P-O bond, which under surface temperature conditions is broken only by enzyme mediated reactions (Jaisi et al., 2010; Kolodny et al., 1983; Longinel and Nuti, 1973; Tudge, 1960) followed by specific isotopic fractionation. Specific isotopic fractionations reflect biological processes, which involve P compounds. As mentioned earlier, no isotopic fractionation is

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Table 1
P concentrations in mg P g^{-1} and Resin and HCl $\delta^{18}O_P$ (‰) of pollen from different tree species (organic P concentration n = 2, $\delta^{18}O$ resin and HCL n = 3, ± SD).

Tree species	Total	Organic	HCl	Resin	$\delta^{18}O$ Resin	$\delta^{18}O$ HCl
P. Halapinesis IL	2.13	1.44 ± 0.01	0.69	0.42	25.3 ± 0.4	29.0 ± 0.02
P. Nigra IL	N/A	N/A	0.75	0.74	22.7 ± 0.5	N/A
P. Brutia IL	2.07	1.41 ± 0.27	0.66	0.42	25.3 ± 0.2	29.0 ± 0.5
P. Banksiana NH	2	1.31 ± 0.02	0.69	0.71	25.2 ± 0.1	25.4 ± 0.6
P. Banksiana MA	N/A	N/A	0.36	N/A	21.1 ± 0.6	N/A
B. Pendula IL	0.84	0.64 ± 0.03	0.2	0.4	23.8 ± 0.2	26.0 ± 0.4
B. Pendula SE	1.43	0.99 ± 0.08	0.44	0.53	27.5 ± 0.4	26.2 ± 0.1
B. pendula MA	4.28	N/A	0.45	0.77	29.6 ± 0.1	19.2 ± 0.6
C. Sempervirens IL 2016	0.98	0.32 ± 0.04	0.67	0.16	32.7 ± 0.4	26.8 ± 0.5
C. Sempervirens IL 2017	N/A	N/A	0.59	0.38	27.5 ± 0.4	23.3 ± 0.3
O. Carpinifolia 1 2016 IL	2.89	1.66 ± 0.01	1.23	1.24	24.0 ± 0.2	25.8 ± 0.3
O. Carpinifolia 2 2016 IL	1.73	1.02	0.71	0.69	22.0 ± 0.2	24.8 ± 0.4
O. Carpinifolia 1 + 2 2017 IL	N/A	N/A	1.21	0.73	27.2 ± 0.1	26.9 ± 0.5
O. Carpinifolia MA	N/A	N/A	1.48	1.57	27.6 ± 0.4	26.4 ± 0.1

involved in phosphate a-biotic surface-temperature processes (Dahms and Boyer, 1973; Kolodny et al., 1983; Tudge, 1960). Therefore, $\delta^{18}O_P$ is applicable for tracing the different P sources. $\delta^{18}O_P$ has been found useful in various studies as a tracer of various phosphate sources (Elsbury et al., 2009; Gross et al., 2015, 2016; Young et al., 2009), and could be an effective tool for estimating pollen contribution to the atmospheric P cycle.

Pollen deposition and P concentrations are not uniform at the different locations and annual variations are reported in several studies (Table 3) (Cho et al., 2003; Lee et al., 1996a, 1996b; Saito et al., 1991). This variance emphasizes the need for a tracer in order to identify and add information about pollen emissions and its contribution to the global P cycle.

In the current study we aim to identify the signature of pollen oxygen isotopes in phosphate. This will enable the use of this signature for the separation of pollen from other atmospheric P sources. In addition, we evaluated pollen P inorganic and organic concentrations, which are an important parameter to estimate its importance to various ecosystems and to the global airborne-P budget.

2. Materials and methods

2.1. Pollen sampling

Pollen from anemophilous tree species was collected in 4 sites during the flowering season. During the springs of 2016 and 2017, pollen was collected at the Hebrew University of Jerusalem, Safra Campus, Israel (IL), (31°46′03.1″N 35°11′59.6″E), from *Pinus halepensis*, and at the Jerusalem botanical gardens (31°45′55.4″N 35°12′01.9″E) from the following tree species: *Pinus brutia, Pinus nigra*, *Cupressus sempervirens*, *Betula pendula*, and *Ostrya carpinifolia*. *Ostrya carpinifolia* pollen sampled in 2016 was collected in two different locations at the botanical gardens (1, 2). In 2017 the pollen from these locations was combined for analysis because of low pollen quantities. In addition pollen from *Betula Pendula* was collected in 2016 in Stockholm, Sweden (SW), (59°18′29.8″N 18°12′06.2″E) and *Pinus banksiana* in Dublin, United States (NH), (42°54′35.70″N, 72°3′37.91″W). Pollen was also collected in 2017 from the Arnold Arboretum of Harvard University, Boston, (MA), (42°18′26.3″N 71°07′16.1″W), from *Betula pendula* (accession numbers: 1319–85*A, 105–96*A, 105–96*C), *Ostrya carpinifolia* (accession numbers: 398–75*A, 1295–83*B, 106–2000*A), *and Pinus banksiana* (accession number: 1380–83*A). The pollen was collected to plastic bags, sieved to 350 μm, and kept in a freezer at −20 °C until it was analyzed.

Table 2
–The atomic ratios in pollen samples.

Tree Species	C:N	C:P	N:P
C. sempervirens IL	40	466	12
B. Pendula IL	70	770	11
P. Halapinesis IL	19	252	13

Atmospheric particles were collected during flowering season (5–13 of April 2017) and during the summer (background) (11–18 of June 2015) at a sampling station, near a pine grove at the roof top of the Earth Science institute at the Hebrew University of Jerusalem. The samples were collected on pre-weighted quartz fiber filters from Whatman (QM-A), placed on a high volume air sampler (Tisch Environmental, Cleves OH), operating with a volumetric flow rate of 1.13 m^3 $minute^{-1}$. The sampled particles were divided to fine < 0.5 μm and coarse > 0.5 μm fractions and only the fine fraction was analyzed. After sampling, the filters were weighed and temporarily stored in sealed plastic bags at 4 °C until analysis.

2.2. P concentrations

The concentrations of the P pools were measured in the pollen samples. The resin P pool represents labile inorganic P lightly adsorbed to outer surfaces of particles (Myers et al., 2005). This fraction was extracted by shaking between 0.5 and 1 g of pollen with anion exchange resin membranes (BDH- 55164) in 5 L of deionized water for 24 h. The HCl P pool was extracted by shaking between 0.5 and 1 g in 100 mL of 1 M HCl for 16 h. The concentration of total P was determined by the ignition method (Saunders and Williams, 1955; Walker and Adams, 1958), where duplicates of each pollen sample were ignited at 550 °C. The organic P fraction was defined as the difference between the total P and HCl-P. The P concentrations were determined colorimetrically by the method of Murphy and Riley (1962), with an average difference of 1% between duplicates.

2.3. Elemental analysis

The determination of C, and N was performed using the Thermo Flash 2000 CHN-O Elemental Analyzer. Weight percentages are accurate and reproducible to within ± 0.3%.

Several elements (Al, Fe, Ca, Mg, Mn, Na, K, Ba) were analyzed by ICP-MS (Agilent 7500cx). A series of ICP multi-element standard solutions VI–Merck Millipore (with concentrations ranging from 1 ppt–100 ng/ml)) and standards of major metals (with concentrations ranging from 300 ng/ml—3 μg/ml) was used for calibration. Internal standards (50 ng/ml Sc, 5 ng/ml Re and Rh) were added to every standard and sample for drift correction.

2.4. Isotopic analysis

The $\delta^{18}O_P$ values of the pollen resin P were determined as described

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