



Biogeochemistry of soil inorganic and organic phosphorus: A compositional analysis with balances



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ABSTRACT

Present soil P cycling models and the related ecosystem services such as P retention deny the special properties of compositional data resulting from closure and leading to methodological inconsistencies and pathological behavior. Our objective was to elaborate and interpret a hierarchy of supervised balances between components of the soil P cycle computed as isometric log ratios (*ilr*) to avoid biases when assessing P risk in managed terrestrial ecosystems. Forty-one acid sandy soils, 56 acid to neutral loamy to clayey soils and 41 acid organic soils (10 fibric or hemic peat and 31 sapric 'moorsh' or 'muck' materials) were collected in Québec, Canada, analyzed for resin-P, NaHCO₃-P, NaOH-P and residual P fractions, arranged into a hierarchy of sound balances and classified for the risk of P leaching. Pathological behavior was shown by spurious correlations between P fractions varying in magnitude, sign and significance depending on what was considered as the "whole" (e.g. total P or soil dry matter). Discriminant analysis across unbiased balances showed that the balances related to soil genesis dominated P biogeochemistry. Soils at low or high P risk for P leaching showed contrasting degrees of P_o and P_i loads. Low- and high-P risk loamy and clayey soils differed significantly for balances relating labile to slowly available P_i, as well as those relating P_i to P_o. Low- and high-P risk sandy soils were contrasted by the balance relating oxalate-extractable Al and Fe. Peat and 'moorsh' materials differed in most *ilrs*. As soils loose ecosystem services provided by soil P geochemical and biological pools in terms of P-fractions balances, terrestrial ecosystems become less sustainable.

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1. Introduction

The soil phosphorus cycle is related to soil genesis (Tiessen et al., 1984; Turner et al., 2007) and land management (Messiga et al., 2010). Ecosystem services provided by the soil P cycle are (1) plant nutrition and productivity (Epstein and Bloom, 2005), (2) incorporation of P_i from the geochemical pool into the biological (P_o) pool by soil biota (Stevenson, 1986), and (3) P sorption by Al and Fe oxo-hydroxides (van der Zee et al., 1987).

Phosphorus cycling is influenced by the nature of solid phases, soil pH and biological activity (Pierzynski et al., 2005). While the oxalate-extractable P, Fe and Al estimate P accumulation from fast and slow reactions of phosphate ions with oxo-hydroxides (Lookman et al., 1995), soil pH determines the distribution of P_i species (Kovar and Claassen, 2005). The joint P turnover in soils also depends on autotrophic and heterotrophic activities as related to C and N cycling (Stewart and Tiessen, 1987) and climatic conditions (Delgado-Baquerizo et al., 2013). Soil inorganic P (P_i) and organic P (P_o) transformations are interrelated, because P_i is the main source for P uptake by plants and soil organisms and P_o can replenish solution P_i through hydrolysis (Stewart and Sharpley, 1987).

Hedley et al. (1982) proposed a sequential soil P extraction procedure to assess soil inorganic and organic P fractions. The Hedley method was found to be useful to classify terrestrial ecosystems (Cross and Schlesinger, 1995; Litaor et al., 2004) and monitor long-term P changes in response to land management (Frossard et al., 2000). Because the method may overestimate P_o and does not provide P speciation into different P fractions, information about P forms should be interpreted with caution as 'operational P fractions' (Negassa and Leinweber, 2009) if not supplemented by other techniques (Condon et al., 2005).

On the other hand, the P fractions are compositional data, i.e. strictly positive data that convey relative information and have special properties related to closure between 0 and the unit of measurement (Aitchison, 1986). Because redundancy, subcompositional incoherence and inherent non-normal distribution lead to methodological bias in the statistical analysis of compositional data (Filzmoser et al., 2009), statistical models using raw concentration data have pathological behavior. Parent et al. (1992, 2000) and Duguet et al. (2006) thus applied compositional data analysis (CoDa) to C, N and P transformations in soils using the centered log ratio transformation of Aitchison (1986). Parent et al. (2009) and Abdi et al. (2011) suggested using the isometric log ratio (*ilr*) transformation of Egozcue et al. (2003) and Egozcue and Pawłowski-Glahn (2005, 2006) for the statistical analysis of soil P fractions as balances.

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Table 1

Sequential binary partitions of ($D = 11$) parts made of C, N, Al and Fe oxo-hydroxides, inorganic P (P_i) and organic P (P_o) pools for the formulation of balances as isometric log ratio ($n^+ =$ number of components at numerator; $n^- =$ number of components at denominator; $g(c^+) =$ geometric mean of components at numerator; $g(c^-) =$ geometric mean of components at denominator).

ilr	C	N	Al _{ox}	Fe _{ox}	Resin-P _i	NaHCO ₃ -P _i	NaOH-P _i	NaHCO ₃ -P _o	NaOH-P _o	Residual P	F _v	n ⁺	n ⁻
ilr 1	1	1	1	1	1	1	1	1	1	1	-1	10	1
ilr 2	1	1	-1	-1	-1	-1	-1	-1	-1	-1	0	2	8
ilr 3	1	-1	0	0	0	0	0	0	0	0	0	1	1
ilr 4	0	0	1	1	-1	-1	-1	-1	-1	-1	0	2	6
ilr 5	0	0	1	-1	0	0	0	0	0	0	0	1	1
ilr 6	0	0	0	0	1	1	1	1	1	-1	0	5	1
ilr 7	0	0	0	0	1	1	1	-1	-1	0	0	3	2
ilr 8	0	0	0	0	1	1	-1	0	0	0	0	2	1
ilr 9	0	0	0	0	1	-1	0	0	0	0	0	1	1
ilr 10	0	0	0	0	0	0	0	1	-1	0	0	1	1

Our goals were to (1) demonstrate pathological phenomena in the classical way to conduct statistical analyses on soil P biogeochemical compositions, (2) compare the P distribution in soils with contrasting genesis, and (3) present a metaphoric pan balance model for diagnosing soil P biogeochemical systems.

2. Material and methods

2.1. Soil collection

Mineral soils were sampled in the arable layer (0–15 to 0–30 cm) in the Province of Quebec, Canada, and classified as clayey, loamy, and sandy. The 41 acid sandy soils under cranberry or potato production were classified (Soil Survey Staff, 1999) as sandy to loamy, coarse-loamy and coarse-silty Haplaquods, sandy to coarse-loamy, loamy, Haplaquepts, loamy Dystricrepts, and loamy Endoaquents. The 56 acid loamy to clayey soils under maize and soybean cropping systems were classified as Haplaquepts (Soil Survey Staff, 1999). Organic soils were Haplohemists and Haplofibrists (Soil Survey Staff, 1999): 10 peat materials were collected in natural mires (peatlands) and classified as fibric or hemic (Soil Survey Staff, 1999); 31 'moorsh' ('muck') materials (Okruszko and Ilnicki, 2003) were collected in the arable layer (0–30 cm). 'Moorshing' (Okruszko and Ilnicki, 2003) is a soil transformation process occurring in the aerated profile of organic soils following drainage and cultivation.

2.2. Soil analyses

Mineral soil materials were dried at 50 °C for 24 h then sieved to <2 mm. The pH of mineral soils was measured in 0.01 M CaCl₂ using a soil: solution ratio of 1:1 (v/v). Organic soil materials were air-dried to

Table 2

Properties of organic and mineral soils used in this study.

	Organic soils		Coarse-textured mineral soils		Fine-textured mineral soils	
	Minimum	Maximum	Minimum	Maximum	Minimum	Maximum
pH(CaCl ₂)	2.7	6.4	3.9	5.7	5.1	7
C	147	576	6	55	14	39
N	7	26	0.1	3.3	0.6	3.2
Resin-P _i	9	611	13	152	18	170
NaHCO ₃ -P _i	0.1	524	16	225	12	122
NaOH-P _i	1	430	55	1091	24	305
NaHCO ₃ -P _o	11	184	5	88	3	561
NaOH-P _o	12	826	4	332	2	246
Residual P	15	1577	194	759	405	1274
PMIII	2	754	13	282	14	262
AlMIII	1	1592	638	1720	322	1940
FeMIII	56	999	54	373	160	593
Pox	40	2233	146	1747	245	1313
Alox	292	6889	1188	28371	1620	10480
Feox	340	7828	657	11161	1043	5772

() Detection limit.

constant weight and sieved to <1 mm. Soil pH was determined in 0.01 M CaCl₂ using a soil: solution weight: volumetric ratio of 5: 20 for 'moorsh' materials and 1.2: 20 to 40 for peat materials, depending on liquid sorption capacity.

Soil texture was determined according to Gee and Bauder (1986). Organic C and N were quantified using CNS-Leco2000. Oxalate-extractable P, Fe and Al were determined according to Ross and Wang (1993) as a measure of P sorption by oxo-hydroxides (van der Zee et al., 1987). The P, Al, and Fe were also determined according to Mehlich (1984), a method commonly used in North American routine laboratories to classify soils according to P fertility and the P risk of leaching (Guérin et al., 2007; Pellerin et al., 2006); approximately 10% of extracted P is P_o (Khiari et al., 2000). Elements were quantified by inductively coupled argon plasma (ICP-OES).

The Hedley sequential P fractionation was conducted using 0.5 g soil samples according to Tiessen and Moir (2008). Briefly, resin-P (P_i) was extracted with 25 mL water and anionic membranes, shaken for 16 h at 25°C, then centrifuged at 4°C for 15 min at 38,000 g; the P was recovered from membranes in 25 mL 0.5 M HCl. Thereafter NaHCO₃-P (P_i and P_o) was extracted using 25 mL 0.5 M NaHCO₃, pH 8.5, shaken for 16 h at 25°C, and centrifuged at 4°C for 15 min at 38,000 g. The NaOH-P (P_i and P_o) was obtained using 25 mL 0.1 M NaOH, shaken for 16 h at 25°C; centrifuge at 4°C for 15 min at 38,000 g. The HCl-P (P_i): 25 mL 1.0 M HCl, shaken for 16 h at 25°C; centrifuge at 4°C for 15 min at 38,000 g. Residual P and total P in P fractions were extracted following potassium persulphate digestion in an autoclave (103.4 kPa, 121 °C) for 1.5 h (Zheng et al., 2001). The concentration of P_i was determined by the molybdenum blue colorimetric method (Murphy and Riley, 1962). The NaHCO₃-P_o and NaOH-P_o were calculated by difference between P_i and total P.

The Hedley operational P pools were interpreted as follows (Cross and Schlesinger, 1995): (1) the resin-P fraction is a pool of easily exchangeable and solution P_i; (2) the NaHCO₃-P fraction is a labile pool of P_i or P_o including microbial P; (3) the NaOH-P fraction comprises P_i and P_o of lower plant-availability associated with Al and Fe oxides and soil organic matter; and (4) residual P that contains insoluble P_i associated with Ca and Mg minerals and a highly resistant pool of inorganic occluded P and P_o included in stable humus. Operational P pools are heterogeneous at molecular level (Turner et al., 2005).

2.3. Methodological limitations

Although the Hedley fractionation procedure is useful to investigate the fate of native and applied P in terrestrial ecosystems, its value in providing an insight of P biogeochemistry is limited (Hinsinger, 2001). The allocation of sequential soil P extracts to operational P pools in terms of environmental relevance and ecological functions has been criticized by Turner et al. (2005). To improve interpreting the Hedley fractions, phosphate molecules can be analyzed in chemical extracts using X-ray absorption near edge structure [XANES], nuclear magnetic resonance [NMR], Fourier transform infrared [FTIR] and Raman spectroscopy

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