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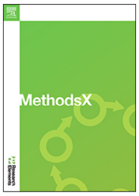

Novel microcosm design to test phosphorus desorption from static soil samples using iron oxide-impregnated filter papers[☆,☆☆]

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ABSTRACT

Iron oxide-impregnated filter papers (FeO) facilitate the study of soil phosphorus (P) desorption, availability and dynamics. However, many studies use homogenized and saturated shaking FeO soil systems, hindering opportunities to resolve environmentally relevant experimental objectives. This study created and evaluated a static microcosm to study soil P dynamics with FeO papers. The microcosm design is simple: silicone rubber is glued to the bottom of a piece of polyvinyl chloride (PVC) pipe. The soil is packed to a known bulk density into the PVC pipe. Finally, the top of the PVC pipe is sealed with a fine nylon mesh, allowing water exchange to the overlaying filter paper. To validate the systems, we created three proof-of-concept tests. The first determined whether FeO filter papers remove statistically significant P compared to a control. The second test examined the effect of soil water content before finally testing the influence of amendment sources on the amount of P desorbed. Results show that FeO papers paired with the microcosms desorb soil P, while the soil water content and amendment type impact total soil P released. Consequently, the soil microcosm design allows users to test and examine soil P processes with FeO papers under new experimental conditions.

- Phosphorus desorption studies using iron oxide-impregnated filter papers are limited by homogenized and saturated extractions. Consequently, a static microcosm design was created to allow for manipulation of experimental factors.
- Results from three proof-of-concept tests confirm that using iron oxide-impregnated filter papers with static microcosms promotes phosphorus desorption, allowing for adjustable experimental designs.

Specifications table

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Background

Agricultural yields rely on sufficient soil concentrations of the essential plant macronutrient phosphorus (P) [1]. While single-step chemical extractions estimate the amount of readily available P, the tests are incapable of measuring desorbable P [2]. In comparison, ‘infinite’ P sinks iron (hydr)oxide-coated filter papers (FeO) estimate the release rates of solid-phase soil P, thereby promoting estimations into P lability and leaching risks [3].

The mechanisms of FeO papers to examine desorbable soil P are well documented. The papers are a selective P sink, are appropriate for all soil types, and correlate well to plant uptake studies [4]. After application, the filter papers rapidly scavenge for solution P [5]. Reducing the concentration of dissolved P forces a diffusive effect that causes soil phase P to replenish the depleted pool [6]. As the FeO papers continuously promote the transfer of soil to solution P means the approach constitutes an infinite P sink [6,7].

The most common FeO paper sample preparation places the papers in a soil solution and new papers are supplied at known intervals [5]. However, this design has a few limitations. For example, the composition of P species differs by fertilizer, promoting variations in spatial and temporal releases [8,9]. Constant shaking increases the soil surface area in contact with the FeO paper, deviating from environmentally representative conditions [10]. Furthermore, soil particles often adhere to FeO paper, a potential source of error when quantifying desorbable P [11]. Consequently, these shortcomings offer opportunities to improve and modify the methodology to advance soil P dynamics investigations.

This article presents and validates a static microcosm design for FeO filter papers. Some noteworthy object features include the mesh overlaying the soil and silicone rubber as the microcosm base. The fine mesh negates soil loss, thereby limiting overestimations of desorbable P [12]. The silicone rubber acts as a gas-permeable, waterproof, and inert microcosm base [13]. While this manuscript presents a single design, the systems are entirely customizable regarding PVC height, diameter, soil quantity, and water content.

The microcosm provides numerous experimental prospects to control environmental factors that affect P dynamics. For example, agronomic soils are generally partially saturated with water, significantly contrasting from the saturated FeO spinning design. Consequently, a FeO microcosm design that allows flexible soil water contents will enhance P desorption trials. Elevated soil temperatures caused by global warming alter soil P-cycling processes [14]. The systems are space-efficient, which means less burden on incubator space and more flexibility to examine temperature effects on P desorption. Finally, as the microcosm height is customizable, users may use the microcosm to combine P desorption with diffusive processes at multiple distances. Therefore, these soil microcosms provide numerous opportunities to examine P soil dynamics.

Method details

Microcosm design

A schematic design of the microcosm is provided in Fig. 1. The microcosm includes a mesh material, a polyvinyl chloride (PVC) pipe, and silicone rubber. An automated table saw at the University of Copenhagen workshop cut the inert PVC pipes (inner diameter 7.5 cm, Lemvigh-Müller, Denmark) to a height of 3 mm. The mesh material comprised a 45 µM nylon mesh (NY-0202, Labopolis, Spain) cut with scissors to the size of the outer diameter of the PVC pipe. Silicone rubber (transparent silicone rubber, 1 mm thickness, RubberProff, Denmark) was cut to the outside diameter of the PVC pipe section using scissors. The glue used to connect the mesh to the PVC pipe and the PVC pipe to the silicone rubber was a hardware store super glue, as the product quickly bonds most plastics together. The users should avoid gluing any part of the soil while attaching the mesh to the PVC pipe. For ease of use, pieces of plastic created a barrier between the FeO paper and the underlying surface. These pieces of plastic were replaced with every FeO paper change.

With the conserved PVC diameter and height and a constant bulk density of 1.4 g cm^{-3}, the total soil mass used was 18.55 g dry weight. The systems are incubated upside down, promoting direct contact between the soil and FeO paper (with mesh in between) via water adhesion. To create FeO-impregnated filter papers, Whatman 5 (Cytiva, Marlborough, MA, USA) papers are cut to the area of the microcosm and synthesized according to [15].

Proof-of concept experimental design and characterization of the soil used

To validate that FeO P desorption studies are possible with this microcosm, we used three proof-of-concept tests to determine whether the microcosm design could support P desorption experiments. Table 1 provides the characteristics of the soils used to complete the trials. Soil texture was determined via the sieving and sedimentation procedure and classified based on the proportion of sand (2.0 – 0.06 mm), silt (0.06 – 0.002 mm) and clay (<0.002 mm) [16]. The water-holding capacity was measured by saturating the sample using a cylindrical column with a perforated base before oven-drying (105 °C) the soil and calculating the difference [17]. The soil pH was measured using a pH electrode in 0.01 M $CaCl_2$ with a soil dry weight: solution in mL ratio of 1:5 [18]. The rapid titration method measured the carbonate equivalent [19]. To estimate plant available P, the soil tests Olsen P at 1: 30 as grams of soil dry weight: mL solution as 0.5 M bicarbonate at pH 8.5 [20] and water-extractable P at 1:60 as grams of soil dry weight: mL solution as Milli-Q water [21] were used. Total soil P was measured using inductively coupled plasma optical emission spectroscopy (ICP-OES, Agilent 5100, Agilent Technologies, Santa Clara, CA, USA) following a microwave digestion with nitric acid, hydrogen peroxide and hydrofluoric acid (EPA 3051A) [22].

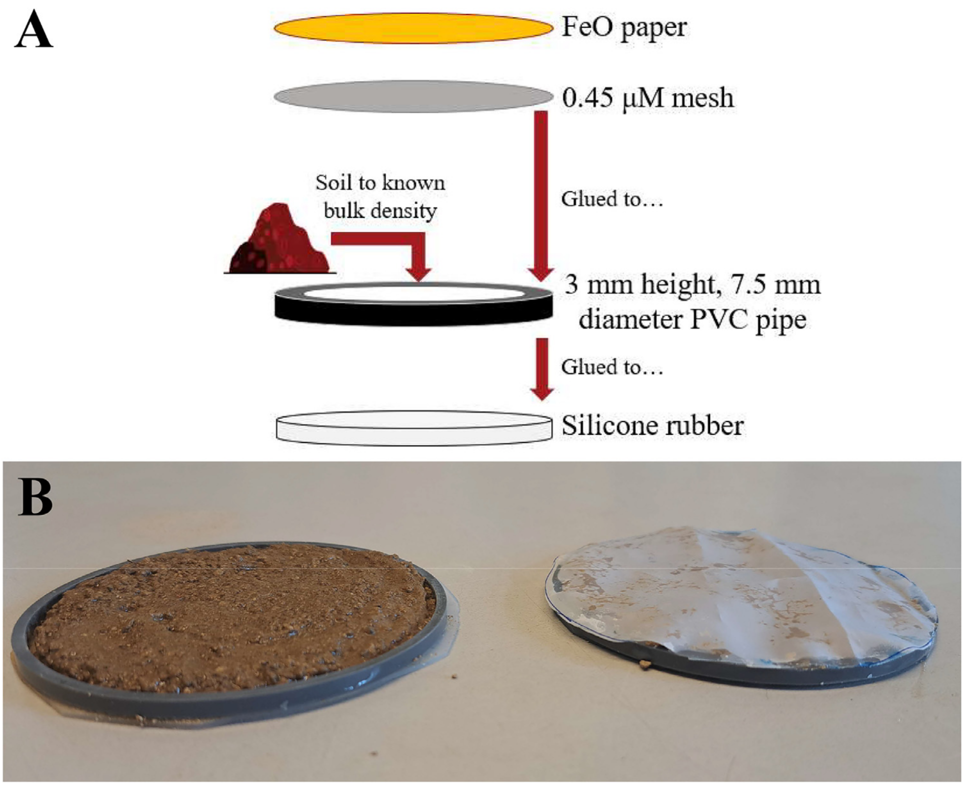


Fig. 1. (A) Graphical schematic of the microcosm design and (B) a picture of the system. FeO = iron-oxide impregnated filter paper; PVC = polyvinyl chloride.

Table 1
Characteristics of soils used in the study.

Soil order [a]	Calcisol	Stagnic cambisol- bottom of catena	Stagnic cambisol - top of catena
Site of Origin	Spain	Germany	Germany
Clay (g^{-1} dry matter) [b]	46.0	12.5	6.8
Silt (g^{-1} dry matter) [b]	28.0	48.7	31.9
Sand (g^{-1} dry matter) [b]	26.0	38.8	61.4
Water-holding capacity (g H_2O 100 g^{-1} dry matter) [c]	65.3	34.6	49.1
Soil pH [d]	7.82	7.57	7.31
Carbonates ($CaCO_3$ equ.,%) [e]	70.1	< 0.1	< 0.1
Olsen P (mg kg^{-1} soil)	7.92	33.9	9.96
Water-extractable P (mg kg^{-1} soil)	>DL	11.7	4.9
Total P (g kg^{-1} soil) [h]	0.25	1.40	0.98

[a] FAO soil order (Food and Agriculture Organization of the United Nations, 2014);.
[b] Soil texture via sieving and sedimentation (Blume et al., 2011);.
[c] Water holding capacity: cylindrical column with perforated base (Wilke, 2005);.
[d] 1 g soil dry weight:5 mL 0.01 M calcium chloride (FAO, 2021);.
[e] rapid titration method (Rayment & Lyons, 2011).
[h] Total P: microwave method and quantified via ICP-OES. >DL = below the detection limit.

Test 1: Investigating whether the microcosms promote soil phosphorus desorption

The first trial examined whether the design promoted statistically significant desorption of soil P. To accomplish this trial, the bottom and topsoil of a *catena* of a Stagnic Cambisol was used. The soil is part of a long-term agricultural field and was collected in 2022 from the plow layer (top 25 cm). The soil was received dry and was sieved to 2 mm before use. The soil was rewet before packing into the microcosms (n = 3 microcosms per treatment). Half of the microcosms were incubated with FeO papers, while the other half did not receive FeO papers. All systems were kept on a lab bench at room temperature, and the FeO papers were replaced daily (n = 10). The soil was weighed daily to replace evaporated water. While microcosms were left in an ambient environment, the soil never dried out (if evaporated water was replaced daily). The systems were incubated upside down, allowing evaporation primarily through the silicone rubber. This trial did not measure the amount of P on the FeO filter papers. Instead, after 10 FeO paper changes, the soils were allowed to dry out at room temperature before grinding using a mortar and pestle and sieving to < 2 mm. The soil tests Olsen P and water-extractable P were used to determine changes in soil P, where phosphate concentrations were measured colourimetrically using the malachite-green plate methodology [23]. As the systems are not homogenized, results are presented in mg L^{-1}.

Test 2: Amount of phosphorus desorbed by soil water-holding content in the microcosms

The second trial focused on determining whether soil water content alters the amount of soil P desorbed. Generally, high water content is required to transfer soil solution P to external systems. For example, in similar infinite-sink systems (like the ferrihydrite-filled DGT gels), the soil water content needs to be high (80 – 100 %) to ensure that the phosphate ions can diffuse to the binding sites [24]. The Calcisol was used for this trial (see Table 1). Three different moisture contents were explored, including 60 %, 80 % and 100 % of soil water holding capacity. Soil microcosms were constructed and incubated similarly to Trial 1. The FeO filter papers were replaced daily ($n = 5$ microcosms per treatment), and weight was monitored daily to replace evaporated water. Once replaced, P was desorbed from filter papers using 40 mL of 0.1 M H_2SO_4 in 50 mL centrifuge tubes [7]. The samples were shaken for 1 h before being filtered into new centrifuge tubes using Whatman 5 filter papers. The malachite plate methodology measured phosphate released from FeO papers [23]. Blanks included clean FeO filter papers. Summed standard deviations were calculated by summing the total squared standard deviation of previous filter paper concentrations before taking the square root to find the new standard deviation. The standard error was then measured by dividing the new standard deviation by the square root of the number of samples involved.

Test 3: Concentration of phosphorus desorbed by amendment source

The final trial examined how different P sources influenced the amount desorbed from soils. The Calcisol was used to complete the experiment. The soil was collected from an agricultural field in 2022, comprised of a mixture of the Ap horizon topsoil to 25 cm depth. Four treatments were used, including three amendments (meat and bone meal (MBM; 36.4 g kg^{-1} P, 426 g kg^{-1}C and 101 g kg^{-1} N), iron-precipitated sewage sludge (sewage; 22.3 g kg^{-1} P, 323.5 g kg^{-1}C and 41.5 g kg^{-1} N and triple superphosphate (TSP 212.2 g kg^{-1} P) with a no amendment added control (control). More information about the recycled amendments MBM and sewage can be found in [25]. Soil was rewet briefly before adding amendments at 120 mg kg^{-1} P. Soils and amendments were mixed in a bowl for 5 min before creating microcosms as described in Trial 1. Systems were incubated at 28 °C and kept inside garbage bags with cups of water to reduce evapotranspiration. The filter papers were replaced weekly (i.e., every seven days) for 11 filter paper replacements. Systems were weighed, and lost water was replaced every 3 or 4 days. The extraction of P from filter paper mirrored the methodology outlined in test 2. Total phosphate extracted (mg L^{-1}) were cumulatively summed identically to the procedure outlined in test 2.

Statistical analysis for all proof-of-concept tests

To complete statistical analysis, R (version 4.2.1) via Rstudio (2023.06.0 + 421 "Mountain Hydrangea" Release) was used. Normality was determined using a combination of the Shapiro-Wilk test and histograms. For test 1, a *t*-test ($p > 0.05$) was used to determine statistical differences between the means before and after FeO incubation for Olsen and WEP. Test 2 used the lme4 [26] package to generate a linear mixed model to test the relationship between the amount of P desorbed and WHC, with the equation and diagnostic plots (created using DHARMa [27,28]) provided in Figure S1. The summed phosphate concentration was left untransformed, whereas fixed effects included WHC and the number of filter paper changes. The random effect was the microcosm replicate, as systems were repeatedly measured. Visual inspection of residual plots showed no issues regarding heteroskedasticity or problematic deviations from the QQ plot (Figure S1). To determine if WHC was significant to the model, a likelihood ratio test compared the whole model to a null model without WHC as a fixed factor. A post hoc multiple comparison between WHC treatments was completed using the package multcomp [29].

Normality was rejected for the dataset of test 3, as the Shapiro-Wilk test, histograms, Q-Q plots and Box-Cox results on the summed variables did not support the distribution. Therefore, to test whether the cumulative P concentration differed by amendment, a gamma mixed model using the glmmTMB package [30], with the equation and diagnostic plots (created using DHARMa [27]) provided in Figure S2. The dataset contained only a few zeroes (time 1 control for replicate 1 and 2, and time 1 MBM for replicate 1). These zeroes represent a methodological limitation of the malachite green method since the limit of detection is 0.05 ppm, and the probability of not recovering any phosphate from the tested soils is improbable. Therefore, values were replaced with 65 % of the lower limit of quantification to allow a gamma family model investigation (i.e., 0.65 * 0.05 ppm = 0.035 ppm [31]). Note that these new numbers were not part of the summed total value. Since microcosms were repeatedly measured, the replicate was added as a random effect, with treatment and filter paper changes as fixed effects (formula included in Figure S2). Dispersion factors included both fixed factors. While the QQ plot residuals look acceptable, the residual vs predicted show some misfit, but there is not enough to be problematic. Once more, post-hoc multiple comparison of means was tested using the package multcomp [29].

Method validation

Results from test 1 demonstrate that Olsen P soil levels decreased for both tested soils after ten filter paper changes. However, only the top of the Stagnic Cambisol *catena* for WEP recorded statistically significant differences (Fig. 2). The higher total soil P content in the bottom soil may require more FeO paper changes to cause identifiable soil P changes in the WEP pool. Nevertheless, the microcosms promote P desorption via FeO papers that alter soil phosphorus test concentrations.

While higher water contents promoted greater phosphate recovery ($p = <0.0001$ compared to the null model), the recovery trend between the treatments during test 2 appears similar (Fig. 3). The WHC used impacts phosphate recovery, as each WHC treatment

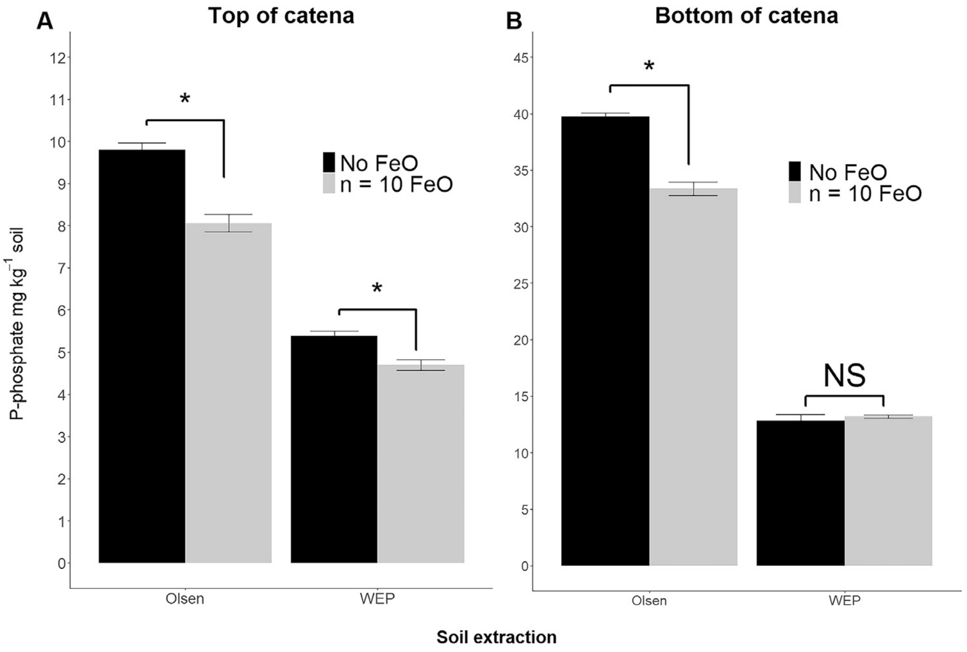


Fig. 2. Mean ± standard error of Olsen P [20] and water-extractable P (WEP; [21]) for microcosms incubated with ($n = 10$) and without iron-oxide-impregnated filter papers. The difference between means was determined using the t-test at $p > 0.05$.

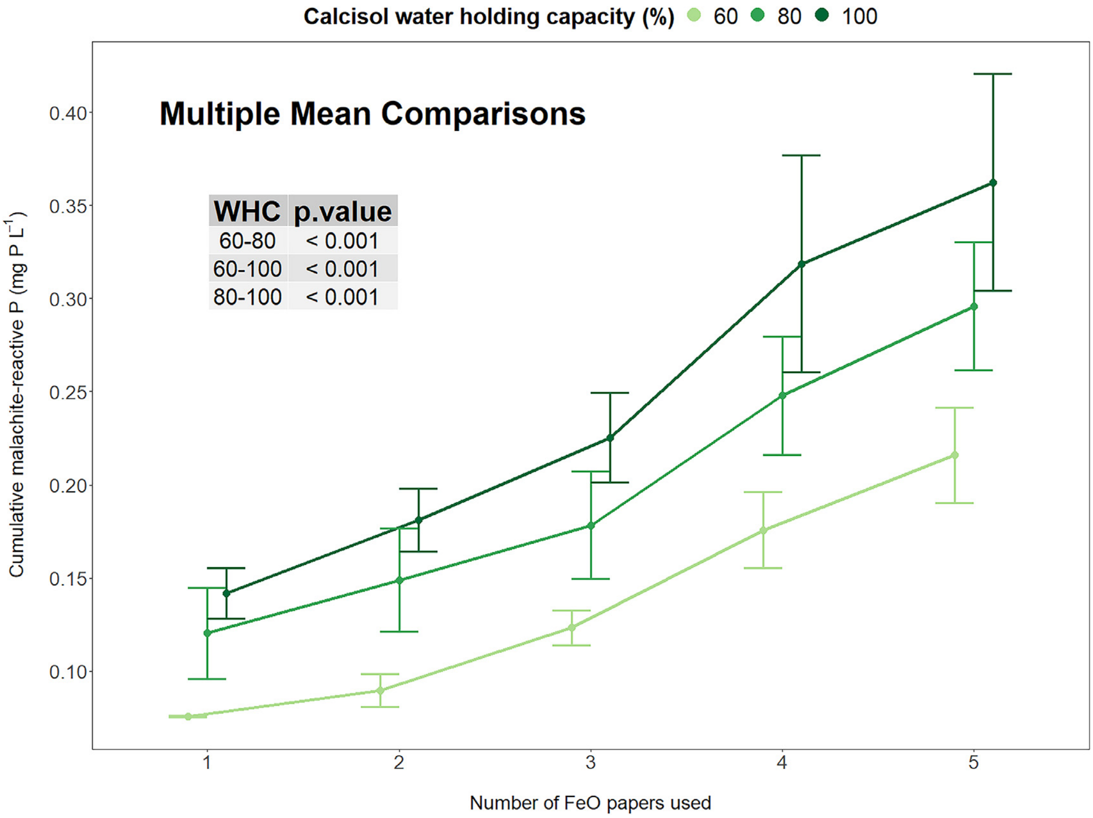
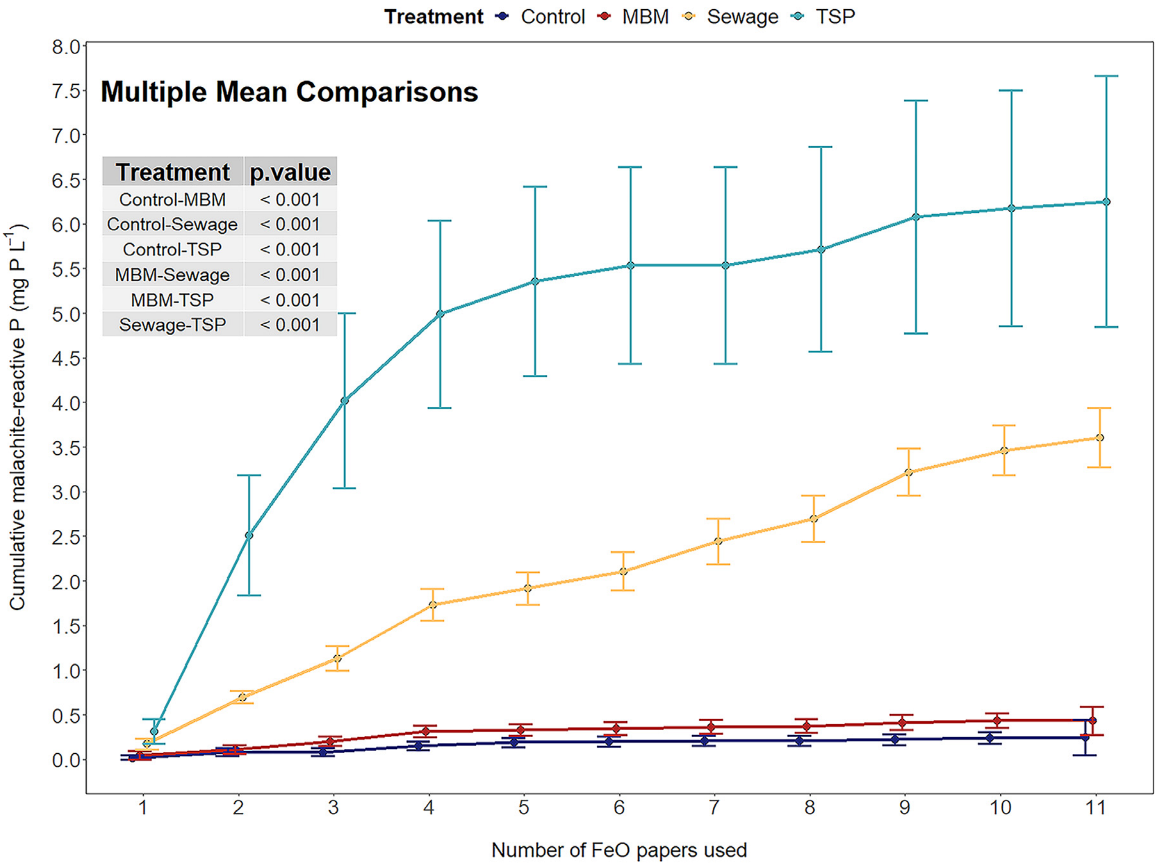


Fig. 3. Mean ± standard error of the cumulative P-phosphate desorbed from the Calcisol by water-holding capacity (WHC) after 5 iron-oxide-impregnated filter papers (FeO) filter paper changes. A linear mixed model was used to determine interactive effects between cumulative phosphate recovered by FeO papers and WHC, with the number of FeO paper changes being a random effect. A multiple mean comparison test identified differences amongst WHC groups.



Treatment	p.value
Control-MBM	< 0.001
Control-Sewage	< 0.001
Control-TSP	< 0.001
MBM-Sewage	< 0.001
MBM-TSP	< 0.001
Sewage-TSP	< 0.001

Fig. 4. Mean ± standard error of the cumulative P-phosphate desorbed from the Calcisol by fertilizer treatment after 11 iron-oxide-impregnated filter paper changes. A generalized linear mixed model using the gamma family was used to determine interactive effects, with multiple mean comparisons completed by treatment. MBM = meat and bone meal; Sewage = sewage sludge; TSP = triple superphosphate.

was statistically dissimilar from the other soil water content. The highest WHC promoted the greatest P desorption, as increased soil water volumes promote greater P solubility [32]. Yet, from user experience, handling microcosms was difficult at 100 % WHC over the other tested water content. While not statistically examined, phosphate desorption patterns appear similar between WHC treatments, revealing the flexibility in potential experimental designs for the microcosm.

Results from this trial demonstrate a treatment difference by P amendment (Chisq=<0.0001 to the null model, Fig. 4). Unsurprisingly, TSP-treated soil desorbed the greatest phosphate concentration to the filter paper, as the amendment possesses the highest proportion of water-soluble phosphate [33]. Over time, concentrations of P collected from TSP-fertilized soil appear to hit a plateau after paper change nine, due to P sorption reactions in the soil [34]. In comparison, the sewage sludge-treated soils promote P release throughout the 11 filter paper changes. Finally, the MBM-treated soil, while significantly different from the control, did not match the P desorption concentrations to the same intensity as the sewage sludge or TSP. This demonstrates that P fertilizer impacts subsequent phosphate release to FeO papers, where the microcosm design is suitable for treatment-effects investigations.

Additional information

A proper assessment of soil P release is not completed after measuring the amount of P sorbed by FeO papers. Briefly, soil P availability relies on the intensity factor, the quantity factor and the buffer capacity [35]. [3] determined that the FeO methodology, in combination with the [12] model, describes soil P of a similar size and availability for plant uptake to the isotope exchange kinetics method. Therefore, this new microcosm, if paired with a routine soil P test, could provide new information about the fate of soil P in environmentally-relevant conditions.

Limitations

- If the FeO paper is left in contact with the soil for too long, microorganisms will consume the paper. To avoid introducing an outside carbon source (i.e., cellulose) to the experiment, we recommend that the users check the structural integrity of the filter paper daily for the first few days of the experiment. The exact time before microorganisms colonize the filter paper is experiment-dependent. For example, the MBM-fertilized soil promoted rapid microbial growth due to substantial amounts of organic matter, nitrogen, phosphorus and calcium [36], but did not promote much P desorption (Fig. 4). Higher temperature incubations promote greater microbial growth [37]. Figure S3 shows before and after pictures of FeO filter papers incubated on soil amended with MBM for 30 days (high C and N promoted rapid microbial growth; see [25]) to allow the users to see the issue and to be informed of the phenomena.

Ethics statements

Not applicable.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

CRediT authorship contribution statement

Aimée Schryer: Conceptualization, Methodology, Validation, Formal analysis, Investigation, Writing – original draft, Visualization. **Pietro Sica:** Conceptualization, Investigation, Writing – review & editing. **Dorette Müller-Stöver:** Writing – review & editing, Supervision. **Jakob Magid:** Writing – review & editing, Supervision, Funding acquisition.

Data availability

Data will be made available on request.

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.mex.2024.103040.

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