



Contents lists available at ScienceDirect

Analytica Chimica Acta

journal homepage: www.elsevier.com/locate/aca

Alternative approach to the standard, measurements and testing programme used to establish phosphorus fractionation in soils

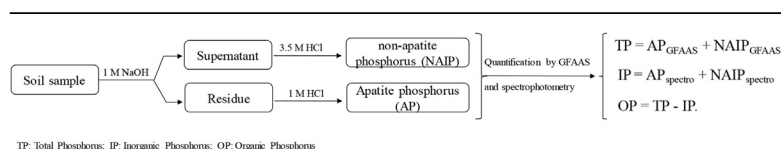
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HIGHLIGHTS

- An alternative method for the determination of phosphorus (P) forms is proposed.
- Total, inorganic and organic P are easily assessed by calculation.
- P forms depended on non-apatite and apatite P measured by GFAAS and UV-visible.

GRAPHICAL ABSTRACT



ARTICLE INFO

Article history:

Received 9 August 2017
 Received in revised form
 15 November 2017
 Accepted 19 November 2017
 Available online xxx

Keywords:

Phosphorus
 Fractionation
 Soil
 Absorption spectrometry
 Spectrophotometry

ABSTRACT

The fractionation of phosphorus in 9 soils was established according to the standards, measurements and testing (SMT) programme. Five fractions were so defined and phosphorus was analysed by spectrophotometry. In parallel, the phosphorus extracted in each fraction was determined using graphite furnace atomic absorption spectrometry (GFAAS) after validation of this technique using certified reference materials. Phosphorus in soils was named total phosphorus, inorganic and organic phosphorus, apatite and non-apatite inorganic phosphorus, depending on extractants. The use of both analytical techniques revealed differences between the concentration of phosphorus in soluble extracts and highlighted the fact that spectrophotometry was a selective analytical technique. In view of the resource- and time-consuming of the SMT procedure and the results obtained in the present study, an alternative method was proposed to estimate the fractionation of phosphorus in soil in order to precise the potential effects of phosphorus on plant nutrition when plant biomass is produced as part of metal-contaminated soil management.

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1. Introduction

Phosphorus (P) is an essential element for animals and terrestrial/aquatic plants. However, eutrophication of fresh water can take place when the concentration of P is so high. This explains why eutrophication is recognized as a pollution problem in North American and European lakes. Phosphorus is also one of the main nutrient and is so used as phosphate fertilizer in order to improve the growth of plants and their biomass in non-contaminated and contaminated soils by metals. Among the in situ metal

immobilisation technique of metals in contaminated soils, phosphate amendments were particularly studied do to their low solubility at acidic pH [1–3] and the formation of stable minerals in Pb and Zn contaminated soils with or without plant cover [4–7].

Determination of bioavailable P was a great concern few years ago because it involves in metabolic processes of living organisms, affects the production of energy in the photosynthesis processes and is important for plant nutrition. Since P cycles in terrestrial ecosystems and soils have been established, physico-chemical processes (e.g. precipitation, complexation sorption, oxidative and reductive reactions) were highlighted between P and the various target phases (e.g. iron- and aluminium-oxides and oxyhydroxides, organic matter, clays) [8]. Wuensche et al. (2015) recently reported

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<https://doi.org/10.1016/j.aca.2017.11.059>

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a comparison of 14 extraction methods used to extract P from 50 agricultural soils [9]. Depending on the extraction time and the soil-to-solution ratio, the extracting power of extractants was obtained according to the following order: HCl 1M after calcination > ammonium oxalate > 0.5 M HCl > 1 M HCl > dithionite > Bray II > Mehlich 3 > cation and anion exchange membrane > calcium-acetate-lactate > Olsen > Fe-oxide > LiCl > CaCl₂ > H₂O. The authors showed that (i) P extractability depended on the pH, carbonate content, iron oxide and texture, (ii) the extracted methods were in relation with different pools of P like soluble, organic or mineral P [9].

An interesting approach consists in the determination of the fractionation of P in sediments or soils. Indeed, the P fractionation can have a key role to estimate the available P for crops. In this way, various sequential extraction procedures were proposed [10–23] and one of them was harmonised and validated in the frame of the standards, measurements and testing (SMT) programme of the European Commission. This P sequential extraction procedure was based on the Burrus method [13], which results in an improvement of the William method [24], which was well described in the literature [18,25–28]. Five fractions were defined in the SMT procedure and for each step, the determination of P is carried out by spectrophotometry using the molybdenum blue method. Being time- and resource-consuming, it would be useful to have a simpler procedure (extraction steps and analytical method). In this perspective, the P content in each fraction of the sequential extraction was determined using UV-Vis spectrophotometry and graphite furnace atomic absorption spectrometry. Results obtained by both analytical techniques were compared, discussed and simplified method, resulting from a combination of concentration of P in the fractions defined by the SMT protocol, was proposed. Using both analytical methods, the P fractionation in soils should give information on the management of metal-contaminated soils via plant biomass production.

2. Materials and methods

2.1. Standard solutions and reagents

All solutions were prepared from analytical-grade reagents unless otherwise specified. Doubly distilled water (Carlo Erba, Val de Reuil, France) was used to prepare all aqueous solutions and to make dilutions. Concentrated hydrochloric acid (HCl, 37%, $d = 1.19$) was obtained from J.T. Baker (Deventer, Netherlands). Standard stock solution of 40 mg P L⁻¹ was prepared from anhydrous potassium hydrogen phosphate (KH₂PO₄, Suprapur[®], Merck).

The reagents used for the spectrophotometric determination of phosphorus were sulphuric acid (H₂SO₄, 96%, Panreac, Lyon, France), L(+)-ascorbic (C₆H₈O₅, Panreac), ammonium molybdate tetrahydrate ((NH₄)₆Mo₇O₂₄·4H₂O, SDS FRANCE, France), potassium antimonyl tartrate trihydrate (K(SbO)C₄H₈O₆·1/2H₂O, Alfa aesar, Schiltigheim, France).

All glassware and polypropylene materials were cleaned by soaking in 0.5 M nitric acid overnight (J.T. Baker for metal trace analysis) followed by rinsing with doubly distilled water.

2.2. Instrumentation

A double-beam UV-1800 spectrophotometer (Shimadzu, Tokyo, Japan) was used for UV-Vis spectrophotometric measurement of phosphate.

An electrothermal atomic absorption spectrometer (Shimadzu AA-6800, Tokyo, Japan) with an ASC-6100 auto-sampler (Shimadzu) was used for phosphorus determination. The AA-6800 was fitted with a digital control technology graphite furnace atomizer

(GFA-EX7) and a pyrocoated tube with pre-inserted platform. Argon was used to provide a protective atmosphere.

2.3. Soil sampling and pretreatment procedure

Soils, used for the production of homegrown vegetables, were sampled from ploughed layer samples (0–25 cm) on contaminated areas highly affected by the past atmospheric emissions of two lead and zinc smelters located in urban area in the north of France. One soil, located far enough from any industrial development, was selected as an agricultural soil non-contaminated by metals. Each composite sample, constituted of 8 sub-samples taken at random, was air-dried at a temperature below 40 °C, crushed to pass through a 2-mm stainless steel sieve, and finally passed to less than 250- μ m sieve with an ultra-centrifugal mill (ZM 200, Retsch, Hann, Germany).

2.4. Procedures

2.4.1. Determination of soil characteristics

Particle-size distribution was obtained through sedimentation and sieving (NF X31-107). Soil pH was measured in a water suspension (NF ISO 10309) and organic matter contents were obtained by the NF ISO 10694 standard. Total carbonate contents were obtained by measuring the volume of CO₂ released after a reaction with HCl (NF ISO 10693). The cation exchange capacity (CEC) was determined after percolation of 1.0 M ammonium acetate solution at pH 7 according to the NF X 31-130 standard [29]. Exchangeable cations (Ca²⁺, K⁺, Mg²⁺, and Na⁺) were extracted with a CH₃COONH₄ (0.1 M, pH = 7) solution, and measured according to the French standard NF X 31-108. The soil's physicochemical parameters are given in Table 1.

2.4.2. Total phosphorus (P_T) determination

The tested soil phosphorus extraction method was based on the procedure described by Wuenscher et al. (2015) with slight modifications [30]. Briefly, soil samples (200 mg) were placed in a porcelain crucible and calcined in a furnace (Nabertherm P330, Lilienthal, Germany) at 450 °C (3 h). Phosphorus was then extracted at room temperature from ashes using 3.5 M HCl (20 ml, 16 h). The supernatant was removed from the residue after centrifugation for 20 min at 4530 p.m. (Rotanta 460, Hettich, Tuttlingen, Germany). Two reference materials were used to check the quality of the analytical procedures: CRM BCR-141R (calcareous loam soil from Pellegrino, Italy) and NIST 2710a (soil from the flood plain of the Silver Bow Creek, Montana).

2.4.3. Phosphorus fractionation extraction procedure

The detailed protocol for obtaining the different fraction of phosphorus was summarized in Fig. 1. In the first step, the total particulate phosphorus was fractionated chemically into Non Apatite Inorganic Phosphorus (NAIP; phosphorus bound to aluminium, iron and manganese oxyhydroxides) and Apatite Phosphorus (AP; phosphorus bound to calcium). An extraction was performed on 200 mg of soil samples using 1 M NaOH (20 ml, 16 h) to remove the extractable phosphorus. After centrifugation for 20 min at 4530 rpm, the supernatant (10 ml) was treated with 3.5 M HCl (4 ml, 16 h) in order to assess the concentration of NAIP, the forms of P associated with oxides and hydroxides of Fe, Mn and Al. The residue from the first extraction was rinsed with 20 ml of distilled water, after which time the supernatant was discarded and the residue was mixed with 1 M HCl (20 ml, 16 h) to assess the concentration of AP, the forms of P associated with carbonates. The second step consisted in determining the concentration of inorganic and organic phosphorus contents (Fig. 2). Soil sample

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