



Phosphorus retention on forest and vineyard soil samples, mussel shell, pine-sawdust, and on pyritic, granitic and waste materials



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ABSTRACT

Focusing on P, we used batch-type experiments to study P retention on various materials: a forest soil sample, a vineyard soil sample, finely and coarsely ground mussel shell, pine-sawdust, pyritic material, granitic material, mussel shell calcination ash, slate processing fines, and three different mixtures that included three components: sewage sludge, mussel shell ash, and calcined mussel shell or pine wood ash. The pyritic material and the mussel shell ash showed the highest P retention capacity, reaching >95%. The lowest P retention (<60%) corresponded to the pine-sawdust and slate processing fines. Data fitted satisfactorily to the Freundlich model, unless in the case of pine-sawdust and slate fines; however, only forest and vineyard soil samples can be satisfactorily adjusted to the Langmuir model. These results could be useful to program appropriate management practices, especially when focusing on controlling P concentrations in different environmental compartments, as well as to correctly recycle the by-products and waste materials assayed.

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1. Introduction

The increasing application of phosphorus (P) fertilizers in recent years has significantly improved soil P status in most regions of the world (Mejías et al., 2013; Rubæk et al., 2013), but has caused P accumulation in some soils (Maguire et al., 2009; Wang et al., 2015). Nonetheless, in most cases, P accumulation is not high enough to cause environmental issues, especially in alkaline soils. However, P can be leached in organic forms (Hua et al., 2016), transported throughout the soil (Haygarth et al., 2005; Heckrath et al., 1995) or conveyed by runoff (Hart et al., 2004), then reaching aquatic environments (Allen et al., 2006). In fact, different cases of excessive spreading of P in soils, and overall in the environment, have favored P pollution episodes (Filippelli, 2008; Shen et al., 2011), with especial relevance in eutrophication processes (Buczko and Kuchenbuch, 2007; Correll, 1998; Lampert and Sommer, 1997; Reynolds, 1984).

Among the various technologies that can be used for P immobilization, sorption is considered an excellent alternative due to its effectiveness, low cost, easy of design and simplicity in use and maintenance (Bhatnagar and Sillanpää, 2010). Some agricultural and forest by-products and waste materials can be appropriate as bio-sorbents, many of them being effective, sustainable and low-cost materials (Ahmedna et al., 2000). Some of the

bio-sorbents that have been previously studied (although not specifically focusing on P retention) are nut shells (Toles et al., 1998; Wafwoyo et al., 1999), wastes from fresh fruits (Soleimani and Kaghazchi, 2008), and wastes from cereal crops (Elizalde-Gonzalez et al., 2008; Khalil, 1996). However, many of these and other sorbent materials have not yet been studied as regards its P-retention capacity.

In addition, a growing interest on the recycling of by-products as bio-sorbents has been shown in recent years (Núñez-Delgado et al., 2015). In this regard, we have previously studied different sorbent and bio-sorbent materials as regards their retention and/or removal potential, focusing on various pollutants in anionic form. Specifically, Fernández-Pazos et al. (2013) studied Cr(VI) retention on forest and vineyard soil samples, as well as on pyritic material, slate processing fines, and coarse and fine mussel shells, finding high Cr(VI) immobilization on the pyritic material, followed by the forest soil and fine shell, with higher Cr(VI) retention on the forest than in the vineyard soil samples, as well as higher Cr(VI) sorption on the fine than on the coarse shell. Seco-Reigosa et al. (2013a) studied As(V) retention on forest and vineyard soil samples, on slate fines, mussel shell and sawdust, finding higher As(V) immobilization on fine than on coarse shell, low retention on the vineyard soil samples and slate fines, high retention on the forest soil sample, and the lowest retention on sawdust samples. Seco-Reigosa et al. (2014) studied three waste mixtures, finding 90–96% sorption for anionic As. Otero et al. (2015) found low Cr(VI) retention on granitic material, while Seco-Reigosa et al. (2015) found up to 99%

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As(V) adsorption on shell-amended granitic material. In addition, Rivas-Pérez et al. (2015) studied forest and vineyard soil samples, pyritic material, granitic material, mussel shell, shell ash, pine sawdust and slate processing fines, focusing on competitive As(V) and P sorption.

Taking into account all this previous background, the objective of this work was to study P-retention potential of forest and vineyard soil samples, and of the following by-products and wastes: a pyritic material, a granitic material, fine and coarse mussel shell, mussel shell calcination ash, pine-sawdust and slate processing fines, as well as three different mixtures that included sewage sludge, mussel shell ash, and calcined mussel shell or pine wood ash. Specifically, we focused on the study of P retention as a function of increasing P concentrations added, as well as on the fitting of data to the Langmuir and Freundlich models, which were not carried out in previous works dealing with these sorbent materials. The results of the investigation could aid to correctly manage soils and appropriately recycle the tested waste materials, especially when focusing on solid and liquid media needing increased control in P retention and/or removal, respectively.

2. Material and methods

2.1. Materials

The materials used were: samples from a forest soil and a vineyard soil, finely (<1 mm) and coarsely (0.5–3 mm) ground mussel shell, pine-sawdust, pyritic material, granitic material, mussel shell calcination ash, slate processing fines, and three different mixtures that included three components: sewage sludge, mussel shell ash, and calcined mussel shell or pine wood ash.

The forest soil sample was from the A horizon of a soil developed on granitic rocks in the vicinity of the Alcoa aluminum factory (San Cibrao, Lugo province, Spain), with *Eucalyptus globulus* as the dominant tree species. The vineyard soil sample was from the A horizon of a soil developed on schists, in Sober (Lugo province, Spain). The mineralogical composition of Galician soils developed over granitic substrates is characterized by variable amounts of quartz, K-feldspar, muscovite, biotite and plagioclase (Eimil-Fraga et al., 2015; IGME, 1981), whereas the most common minerals present in Galician soils developed over schist are phyllosilicates 1:1 (mostly as kaolinite) and illite, with the appearance of inter-stratified illite-vermiculite and traces of Fe oxy-hydroxides (Macías and Calvo, 1992; Silva-Hermo and Macías-Vázquez, 1984). The pyritic material was from a copper mine spoil (Touro, A Coruña province, Spain). The granitic material was a C horizon derived from a rocky substrate, nowadays exposed to the atmosphere after the elimination of the upper horizons, and was sampled in Santa Cristina (Ribadavia, Ourense province, Spain). The slate processing fines were from the slate-processing enterprise Europizarras S.L. (A Fonsagrada, Lugo province, Spain). Pine-sawdust was a commercial product from Vitakraft (Germany). The finely and coarsely crushed mussel shells were from the factory Abonomar S.L. (A Illa de Arousa, Pontevedra province, Spain). Mussel shell ash was from Calizamar S.L. (Boiro, A Coruña province, Spain). The three mixtures including waste materials were as follows (w/w, dry basis): Mixture M-32, 45% sewage sludge, 51% mussel shell ash, and 4% calcined mussel shell; Mixture M-58, 47% sewage sludge, 40% mussel shell ash, and 13% calcined mussel shell; and Mixture M-78, 47% sewage sludge, 48% mussel shell ash, and 5% wood ash.

The forest and vineyard soils, as well as the pyritic and granitic materials, were sampled and transported to the laboratory to be air dried and sieved through 2 mm. The chemical determinations were carried out on the <2 mm fraction.

2.2. Methods

2.2.1. Characterization of the materials used

C and N contents were measured on 5-g samples using an elemental Tru Spec CHNS auto-analyzer (LECO, USA) (Chatterjee et al., 2009). A

pH-meter (model 2001, Crison, Spain) was used to measure pH in water (10 g of solid sample, with solid:liquid relation 1:2.5), as well as to determine values corresponding to the point of zero charge (pH_{pzc}) measured as per Mimura et al. (2010). A NH_4Cl 1 M solution was used on 5-g samples to displace the exchangeable cations, then quantifying Ca, Mg and Al by atomic absorption spectroscopy, and Na and K by atomic emission spectroscopy (AAAnalyst 200, Perkin Elmer, USA); the effective cationic exchange capacity (eCEC) was calculated as the sum of all these cations. Available P was determined as per Olsen and Sommers (1982) using 5-g samples. Total concentration of P was determined by means of UV-visible spectroscopy (UV-1201, Shimadzu, Japan) after nitric acid (65%) microwave assisted digestion on 1-g samples (Tan, 1996). Total concentrations of Na, K, Ca, Mg, Al, Fe, Mn, As, Cd, Cr, Cu, Ni and Zn, were determined using ICP-mass (820-NS, Varian, USA), after nitric acid (65%) microwave assisted digestion on 1-g samples (Nóbrega et al., 2012). Ammonium oxalate solutions acidified to pH 3 with oxalic acid were used to obtain total non-crystalline Al and Fe (Al_o , Fe_o) from 1-g samples (Álvarez et al., 2012). All trials were performed by triplicate.

Table 1 shows the results corresponding to the chemical characterization of the soil samples and sorbent materials assayed. In addition, the particle-size distribution of forest and vineyard soil samples was determined by using the Robinson pipette procedure. Both soil samples have sandy loam texture. The particle size distribution results were as follows: forest soil sample 65% sand, 20% silt and 15% clay; vineyard soil sample 73% sand, 12% silt and 15% clay. Thus, the average clay content was coincident in both soil samples.

2.2.2. P retention study

The methodology of Arnesen and Krogstad (1998) was used to study P retention for different concentrations of P added to each of the soil samples and sorbent materials assayed (triplicate samples).

In the P retention experiment, 3 g of each solid sample was added with 30 mL of 0.01 M $NaNO_3$ containing 0.5, 5, 10, 25, 50 or 100 mg P L^{-1} prepared from analytical grade NaH_2PO_4 (Panreac, Spain). The resulting suspensions were shaken for 24 h, centrifuged at 4000 rpm ($6167 \times g$) for 15 min, and finally filtered using acid-washed paper (pore size 2.5 μm). In the equilibrium dissolutions, pH was measured using a glass electrode (Crison, Spain), dissolved organic carbon (DOC) and P were determined by means of UV-visible spectroscopy (UV-1201, Shimadzu, Japan) (Tan, 1996). Sorbed P was calculated as the difference between two terms: a) the sum of added P and P measured in the control sample, and b) P remaining in the equilibrium solution. P, DOC and pH were determined by triplicate in all samples.

2.2.3. P data fitting to sorption models

Data were fitted to the Freundlich (Eq. (1)) and Langmuir (Eq. (2)) models.

The Freundlich equation can be formulated as follows:

$$q_e = K_F \cdot C_e^n \quad (1)$$

where q_e is the P adsorbed per unit of mass of the sorbent, C_e is the equilibrium concentration of the dissolved P, K_F is a constant related to the sorption/retention capacity, and n is a constant related to the retention intensity.

The Langmuir equation is formulated as follows:

$$q_e = X_m \cdot K_L \cdot C_e / (1 + K_L \cdot C_e) \quad (2)$$

where X_m is the maximum sorption/retention capacity, and K_L is a constant related to the retention energy.

2.2.4. Statistical analysis

All determinations were performed in triplicate. The statistical package SPSS 19.0 (IBM, USA) was used to perform statistics (descriptive

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