Controlling risks of P water pollution by sorption on soils, pyritic material, granitic material, and different by-products: Effects of pH and incubation time

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Abstract

Batch experiments were used to test P sorbent potential of soil samples, pyritic and granitic materials, mussel shell, mussel shell ash, sawdust and slate waste fines for different pH and incubation times. Maximum P sorption varied in a wide range of pH: <4 for pyritic material, 4-6 for forest soil, >5 for slate fines, >6 for shell ash, and pH 6-8 for mussel shell. P sorption was rapid (<24 h) for forest soil, shell ash, pyritic material and fine shell. On the opposite side, it was clearly slower for vineyard soil, granitic material, slate fines, pine sawdust and coarse shell, with increased P sorption even 1 month later. For any incubation time, P sorption was >90% in shell ash, whereas forest soil, pyritic material, and fine shell showed sorption rates approaching 100% within 24 hours of incubation. These results could be useful to manage and/or recycle the sorbents tested when focusing on P immobilization or removal, in circumstances where pH changes and where contact time may vary from hours to days, thus aiding to diminish P pollution and subsequent eutrophication risks, promoting conservation and sustainability.

Keywords: By-products; Forest and vineyard soils; Phosphorus; Recycling; Sorption; Water pollution

1 Introduction

As indicated in Chowdhury et al. (2017), P fertilizers are of great importance to improve crop productivity, and global food production could be highly affected by scarcity of the global P resource, but application of excessive dosses of P fertilizers may be of environmental concern, especially affecting water courses (Kyllingsbæk and Hansen 2007), where it can trigger eutrophication processes (Shinohara et al. 2016). Specifically, certain agricultural and industrial activities can cause excessive P concentrations in the environment, which may produce alarming growth of algae and aquatic plants (Garnier et al. 2005; Tian et al. 2016). In soils with low P retention potential, P can be easily released into the soil solution, then reaching aquatic environments (Allen and Mallarino 2006). Moreover, long-term application of P fertilizers may decrease P sorption, also increasing P concentrations, transport and availability (Mejías et al. 2013; Wang et al. 2015), causing a significant environmental risk, especially in lakes.

As commented by Pierzynski et al. (2005), P dynamics in soils is mainly dependent on mineralogy and organic matter constituents and characteristics, as well as on ionic strength and pH of the soil solution. However, the effect of pH on P sorption is somehow controversial. As an example, Sato and Comerford (2005) and Yin et al. (2016) indicate that P sorption decreases with increasing pH, whereas Pereira and De-Faria (1998), and Agbenin (1995), found that P sorption increased with increasing pH. In addition, studying P removal in biochars, Liu et al. (2016) found that electrostatic attraction was more important to adsorb P at a lower pH, while precipitation and surface deposition took over at higher pH.

Regarding the effect of contact time, previous studies indicate that P-sorption reactions are initially rapid and exothermic, replacing an OH^- or a water molecule by the phosphate anion, forming a surface phosphate-complex (Torrent et al. 1992; Brady and Weil 2002); in a second step, reaction mechanisms are slower and can subsequently lead phosphorus into the solid matrix of sorbents, resulting in less available compounds (McGechan and Lewis 2002).

Although numerous studies have been carried out dealing with P sorption on different materials (Mengel 1985; Maguire et al. 2001; Arias et al. 2006; Rahnemaie et al. 2007; Buckingham et al. 2010; Devau et al. 2010; Vega and Riemsdijk 2011; Lürling et al. 2016), many other sorbents could be investigated, especially those that are low-cost and that can facilitate the recycling of by-products and residues (Arshadi et al. 2015). In this way, we have recently studied P sorption on different soil samples and by-products (Romar-Gasalla et al. 2016), although the effects of changing pH and incubation time were not considered.

A relevant fact is that recycling waste and by-products as low-cost sorbents could aid to increase sustainability in the productive sectors affected (Núñez-Delgado et al. 2015; Grace et al. 2016), as recently shown for wastes from the mussel shell industry (Quintáns-Fondo et al. 2016).

Taking all that into account, we study the effects of pH and incubation time on P sorption for different soil samples, by-products and waste materials: a forest and a vineyard soil, a pyritic and a granitic material, fine and coarse mussel shell, shell ash, sawdust and slate waste fines. The results could aid to carry out a correct management of the soils and by-products tested, in situations where P levels should be controlled in solid or liquid media.

2 Material and methods

2.1 Materials

The materials investigated were two kinds of soils (forest and vineyard soil samples), pyritic material, granitic material, fine and coarsely crushed mussel shell, shell ash, pine sawdust and fines from slate processing. These materials have been previously characterized in detail (Fernández-Pazos et al. 2013; Seco-Reigosa et al. 2013, 2015; Otero et al. 2015; Rivas-Pérez et al. 2015). Recently, Romar-Gasalla et al. (2016) studied different aspects of P sorption on these soil samples and by-products, although the effects of changing pH and incubation time were not considered. More information regarding all these sorbents is presented in Supplementary Material.

2.2 Methods

2.2.1 Characterization of the materials used

All materials were analyzed as described in Romar-Gasalla et al. (2016). More details and full characterization information is included in Supplementary Material.

2.2.2 P sorption as a function of pH

Triplicate 1-g samples of each of the various soils and sorbent materials were added with 10 mL solutions containing 100 mg P L^{-1} and different concentrations of HNO_3 (0.0025 M, 0.0038 M, 0.005 M, 0.0075 M) or NaOH (0.0025 M, 0.0038 M, 0.005 M, 0.0075 M), also including $NaNO_3$ 0.01 M as background electrolyte. All these chemicals were from Panreac (Spain). Different control samples were constituted by the various materials (without HNO_3 or NaOH), which were added with 10 mL of solutions containing

$NaNO_3$ 0.01M and 100 mg P L^{-1}. All these samples were shaken (24 h), centrifuged (15 min at 4000 rpm, which means 6167 ×*g*), and filtered (using acid-washed paper with 2.5 µm of pore size). The resulting liquid was analyzed for P using UV-visible spectroscopy (UV-1201, Shimadzu, Japan). Sorbed P was calculated as the difference between added-P concentration and P concentration in the equilibrium solution.

2.2.3 P sorption for different incubation times

Triplicate 10-g samples corresponding to the soils and sorbent materials were added with 100 mL of a 0.01 M $NaNO_3$ solution containing 100 mg P L^{-1} (1:10 solid:solution ratio), maintaining the contact during 1 month (720 hours). Aliquots (5 mL each) were taken at different incubation times: 0.5 h, 2 h, 4 h, 8 h, 12 h, 24 h, 168 h (1 week) and 720 h (1 month), centrifuged, filtered and analyzed for P, as above.

2.2.4 Statistical analysis

SPSS 21 (IBM, USA) was used to calculate coefficients of variation from mean and standard deviation values for replicate samples (descriptive statistics).

3 Results and discussion

3.1 P sorption as a function of pH

Fig. 1 shows that the influence of pH on P sorption was generally low and very variable. Arias and Fernández (2001) found similar behavior in previous studies that focused on soils amended with limestone and/or gypsum. Other authors obtained results showing that P sorption may decrease (Sato and Comerford 2005; Yin et al. 2016) or increase (Agbenin 1995; Pereira and De Faria 1998) with increasing pH.

In the present study, the highest P sorption results varied with pH for the different materials. Specifically, it was as follows: in the forest soil samples (with pH corresponding to the point of zero charge $pH_{pzc} = 5.53$, Table S1, Supplementary Material) maximum P sorption was obtained at pH between 4 and 6 (Fig. 1a); in the pyritic material (with $pH_{pzc} = 3.46$) maximum sorption occurred at pH values <4 (Fig. 1b); in slate fines (with $pH_{pzc} = 9.47$) it took place within the pH range 5 to 12 (Fig. 1b); in both kinds of mussel shell (with pH_{pzc} 9.72-9.94) maximum P sorption occurred in the pH range 6 to 9 (Fig. 1c); and in shell ash (with pH_{pzc} 12.87) it took place within the pH range 6 to 11 (Fig. 1d). In the other materials (vineyard soil sample, granitic material and pine sawdust) the influence of pH was even less clear. Similarly to that obtained in the forest soil samples here studied, some authors found maximum P sorption in soils at pH values close to

5 (Anjos and Rowell 1987; Das et al. 2006). Shen et al. (2015) studied phosphate removal using other sorbent materials, specifically they studied different tetraethylenepentamine-functionalized core-shell structured nano magnetic Fe_3O_4 polymers, finding that the adsorption processes were highly pH dependent (as well as related to the content of Fe_3O_4 magnetic core in the adsorbents), and that optimized pH value was at 3.0, although it was at 2.5 when Fe_3O_4 magnetic core was absent. Zach-Maor et al. (2011a) studied phosphate removal using a synthesized homogeneous layer of nano-sized magnetite particles, finding that at pH 6.3, approximately 50% and 70% of phosphate were removed (from initial concentration of 500 mg L^{-1}) within the first 1 and 3 h, respectively. Zach-Maor et al. (2011b) studied, in a detailed manner, adsorption mechanisms for this sorbent material, finding that it was initially carried by the active sites on the magnetite surface followed by a slow diffusion into the nano-magnetite layer, and that, following diffusion, some of the outer magnetite binding sites become available for further phosphate uptake.

The decrease in P sorption with increasing pH observed in the pyritic material and forest soil samples here studied could be due to different causes. One of them could be related to the increase in hydroxyl groups in the solution due to the higher pH, because of the competition between OH^- and phosphate for specific sorption sites of the mineral surfaces. Haynes (1982) indicates that the progressive increase in the negative surface charge that occurs with increasing pH causes an increased electrostatic repulsion, reducing P sorption. In the present study, the forest soil samples, granitic material, and especially the pyritic material, have variable-charge components, such as Fe and Al oxy-hydroxides, kaolinite-type clays and/or organic matter, that at acid pH have positive charge and favor P sorption; but when the pH of the medium increases, de-protonation processes take place, with the consequent appearance of negative charges on variable-charge components, favoring a decrease in P sorption. When such components are positively charged, their surfaces can retain $H_2PO_4^-$ and HPO_4^{2-} anions. Authors such as Torrent et al. (2007) consider that amorphous Fe and Al minerals are of great importance in P sorption (with special relevance in acid and loamy to coarse soils). Other authors, such as Paulter and Sims (2000) and Campos et al. (2016), have suggested that the high specific surface area and active sites of amorphous Al-hydroxides are one of the most important causes of P adsorption. Naidu et al. (1990) attribute the decrease in P sorption when pH increases to neutralization and precipitation of Al as Al hydroxide, decreasing P-sorption sites. Berkowitz et al. (2006) showed the influence of Al precipitation on P sorption, due to the fact that when Al concentration increases the degree of crystallinity of $Al(OH)_3$ also increases, thus diminishing surface area and P sorption.

P sorption on the pyritic material and forest soil samples here studied, at pH values >5 (which corresponds to a common pzc value for forest soils, as was the case of the forest soil sample here studied) can be related to the presence of Fe sulfates and oxy-hydroxides resulting from oxidation of pyrite (for the pyritic material), and to the presence of Fe and Al oxy-hydroxides and of organo-aluminum complexes (for the forest soil samples); all of them are variable-charge compounds and acquire negative charge at high pH, causing that sorption of P (present as HPO_4^{2-} at these pH values) occurs through a cationic bridge. Furthermore, it must be taken into account that organic matter can perform a dual role on P sorption: unfavorable due to the blocking of sorption sites by organic anions (Guppy et al. 2005), and favorable due to its negative charge, attracting cations that can induce P retention (Sanyal and De-Datta 1991; Torrent et al. 1992; Wang et al. 2009; Campos et al. 2016).

In the case of shell ash and mussel shells, high P sorption results were obtained at higher pH. This may be related to the presence of carbonates such as calcite, which favors P sorption, especially at pH above 6 (Zhang and Selim 2008; Yolcubal and Akyol 2008). In addition, some authors indicate that carbonates have a higher importance in P-sorption in alkaline soils (Hinsinger 2001; Carreira et al. 2006).

3.2 P sorption for different incubation times

Percentages of P retained for different incubation times are shown in Fig. 2, where two very different behaviors can be distinguished. On the one hand, the forest soil samples (Fig. 2a), pyritic material (Fig. 2b), fine shell (Fig. 2c) and shell ash (Fig. 2d), show rapid P sorption, reaching the maximum within 24 h in most cases, remaining fairly constant from that moment. On the other hand, the vineyard soil samples (Fig. 2a), granitic material (Fig. 2b), slate fines (Fig. 2c), coarse shell (Fig. 2c), and sawdust (Fig. 2d) have a clearly slower P sorption, showing increases even after one month (720 h). Torrent et al. (1992) and Hongshao and Sranforth (2001) investigated the influence of incubation time on P sorption, finding that it was initially rapid, and then a slower phase occurred. In the present study this behavior was observed in the forest soil samples, shell ash, pyritic material and in both kinds of mussel shell. Explanations to the existence of the slower phase include precipitation reactions (Jonasson et al. 1988), diffusion into the surface pores or into the adsorbent matrix (Strauss et al. 1997), or coagulation reactions (Anderson and Lindquist 1985).

For any incubation time, P sorption was >90% in shell ash, being 95% after just 4 hours of incubation, then increasing by 3% after a week of incubation (168 h). Also, the forest soil samples, pyritic material, and fine shell showed sorption rates approaching 100% within 24 hours of incubation. The increase in incubation time resulted in a significant increase in P sorption exclusively in the vineyard soil samples, slate fines, granitic material, sawdust, and coarse shell. This effect did not occur in the other materials, maybe due to having very accessible sorption sites, then facilitating saturation after a few hours of contact.

4 Conclusions

Batch-type studies were conducted to evaluate the effects of pH and incubation time on P sorption for different soil samples, by-products and waste materials. The results indicate that, in general, the influence of pH on P sorption was very dependent on the sorbent material, with optimal values for P sorption presenting a wide oscillation (pH between 4 and 12). Regarding the effect of incubation time, mussel shell, shell ash and the pyritic material showed very rapid P sorption, reaching maximum values within 24 h, which were maintained from that time; however, the granitic material, slate fines, vineyard soil samples and sawdust showed a much slower P sorption. These results could aid to program the correct use of the soils and by-products studied, in circumstances where P concentration should be controlled, and in which pH and contact time with sorbents may suffer substantial changes, aiding to control P pollution, facilitating water management and promoting sustainability.

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Figure captions

Fig. 1 P sorbed at different pH values for the various sorbent materials tested. Average values and error bars corresponding to 3 replicates, with coefficients of variation always <5%

Fig. 2 P sorbed at different incubation times on the various sorbent materials tested. Average values and error bars corresponding to 3 replicates, with coefficients of variation always <5%