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Plant-availability of phosphorus recycled from pig manures and dairy effluents as assessed by isotopic labeling techniques

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ABSTRACT

Deposits of phosphate rocks are non-renewable and the only fossil resource for the production of phosphate fertilizers. The presence of phosphorus (P) in animal and domestic wastes provides an alternative opportunity to recycle P for use as P fertilizer. Hence, the objective of the present study was to assess the plant availability of recycled P products derived from dairy effluents (one product, hereafter named as "RPDE") and pig manures (four products, "RPPM"), through bio- and chemical precipitation processes, respectively. The RPDE product is composed of Ca–P (partly as hydroxyapatite, HA) and RPPM products contain recovered struvite (ST) and Ca–P. Plant-availability of recycled P was compared to that of commercial triple superphosphate (TSP), reference HA, and reference ST. To this end, pot and soil incubation experiments were used. A pot experiment with a mixture of ryegrass and fescue was carried out using a P-deficient and slightly acidic (pH = 6.49) soil, at 50 mg P kg^{-1} application rates of the different products. The ^{32}P-labeling of soil P was used to determine the L-value (i.e. plant-available soil P) and to accurately quantify the P taken up by plants from the different P sources. Shoot and root biomass productions, plant P nutrition and L-value increased owing to application of P products. There were no significant differences between RPDE and RPPM products with regard to plant P nutrition. All recycled products were as effective as TSP and reference ST. By contrast, the P-equivalence of HA was only 22% of TSP in the slightly acidic soil. Thus, plant availability of P in RPDE product was higher than that of well-crystallized HA (synthetic product). Product application to incubated soils induced an increase in the amounts of phosphate ions in soil solution and isotopically exchangeable P (E-values), which were overall correlated to the L-values and plant P uptake. Plant-availability of recycled P may thus be inferred appropriately from simple soil incubations. In conclusion, this study shows that it is possible to substitute commercial fertilizers (such as TSP) by P recycled from pig manures and dairy effluents.

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1. Introduction

Phosphorus (P), an essential macronutrient, is a major constraint of crop growth and food production in many parts of the world (Elser et al., 2007). As a consequence, P fertilizers are commonly applied on agricultural lands. Commercial P fertilizers are manufactured from phosphate rock deposits, a non-renewable resource. The economically exploitable reserves of phosphate rocks could be depleted within the next century at the present pace (170 million tons year^{-1}; Cordell et al., 2009; Gilbert, 2009). Besides its role in crop production, P is also one of the essential elements for animal nutrition (Saito, 2001). The daily P requirement of dairy and beef cattle has been reported up to 95 and 40 g day^{-1}, respectively (CEEP, 2003). Animals can only utilize a small portion of the ingested nutrient, while the rest is excreted as manure or urine (Dhakal, 2008). Besides livestock wastes, urban and agro-industrial effluents also possess high concentration of P that can be partly lost in surface waters and contribute to eutrophication. It is therefore necessary to recycle P from human and animal wastes; moreover these can be used as alternative P fertilizers (Cordell et al., 2009).

Phosphorus can be extracted from wastes by various processes. Among them, chemical crystallization enables the precipitation of dissolved P in liquid effluents as calcium–phosphates (Ca–P) or struvite (ST; magnesium–ammonium–phosphate) (Antonini et al., 2012;

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Cabeza et al., 2011; Moerman et al., 2009; Ryu et al., 2012). Recovered ST was reported to be generally as effective as commercial mineral P fertilizers (e.g. single superphosphate or triple superphosphate (TSP)), while the effectiveness of Ca–P products depends on soil properties such as pH (e.g. Cabeza et al., 2011). The ST precipitation process has been developed to extract dissolved P from urban and agro-industrial effluents, but it had rarely been adapted for animal wastes (e.g. swine wastewaters) due to a high proportion of P in particulate forms and the necessity of a preliminary step of P dissolution (Capdevielle et al., 2013; Daumer et al., 2013). To recycle P from effluents produced by the agro-food industry (e.g. dairy effluents), ST precipitation is feasible following digestion and nitrification/denitrification steps (Mañas et al., 2012). However, such ST precipitation required the use of reagents and a biological P removal process can alternatively be adopted through a microbially induced P precipitation (Mañas et al., 2012; De Kreuk et al., 2005).

The Brittany region in France is specialized in intensive pig farming. P in swine wastewaters has to be recycled to avoid massive applications of P to agricultural soils and negative impacts on surface waters. In addition to animal wastes, the agro-food industry also represents a significant source of P-rich effluents that need to be recycled. P recycling processes of pig manures and dairy effluents have been recently developed in France through laboratory pilots and are currently adapted at an industrial scale. A first pilot was developed to recycle P from pig manures through chemical precipitation. The procedure includes preliminary steps, i.e. acidification of pig manure to enable P dissolution and separation of residual particulate organic matter. Thereafter, a crystallization reagent (i.e. magnesia (MgO)) is added to promote ST crystallization conditions ($[Mg^{2+}]:[NH_4^+]:[PO_4^{3-}]$ ratio of 1:1:1; pH of 7–11). The last step consists of crystal filtration (Capdevielle et al., 2013; Hutnik et al., 2011; Moerman et al., 2009). The final product contains ST crystals in variable proportions (ranging from 12 to 71% of the solid) but Ca–P phases also co-precipitate (9–40%). In addition, the final products also include MgO (16–57%; Daumer et al., 2013; Santellani et al., 2013). Product composition depends on the processing conditions (such as pH during the crystallization step, temperature, Mg:Ca ratio or N:P ratio) and particle size (Capdevielle et al., 2013). According to RAMAN spectroscopy analyses, particles >100 μm and particles between 25 and 100 μm contain more ST crystals than particles <25 μm (Capdevielle et al., 2013). A second pilot was developed to recycle P from dairy effluents through a biological process. It mainly consists in the formation of bacterial granules and microbially induced P precipitation in these aggregates (Mañas et al., 2012). Crystals are mainly hydroxyapatite (HA), as revealed by RAMAN spectroscopy, Energy Dispersive X-ray (EDX) and X-ray Diffraction (XRD) technics and the Ca:P molar ratio of the granules (Ca:P molar ratio of the granules (1.63) close to that of HA (1.67)) (Mañas et al., 2011, 2012).

Given the important issues of P recycling processes (e.g. production of alternative P fertilizers), it is required to assess plant availability of recycled P in order to manage their spreading to field crops. To achieve this purpose, preliminary studies involving pot experiments are commonly carried out to assess the response of plants (inferred with shoot and root biomass production and plant P uptake) to P application (e.g. Cabeza et al., 2011; Massey et al., 2009; Walker et al., 2012). However, it could be difficult to obtain accurate and reliable information, especially when soils are not P-deficient and there is little response of plants to applied P fertilizers. On the other hand, to obtain reliable and accurate information, it can be necessary to carry out pot experiments using an isotopic labeling technique. Soil P is labeled by introducing ^{32}P (½life = 14.3 days) or ^{33}P (½life = 25.3 days) as phosphate ions. This technique enables to differentiate P uptake from native soil P and from applied P sources such as rock phosphates (e.g. Morel and Fardeau, 1989; Zapata and Axmann, 1995), filter substrates derived from small-scale wastewater treatment systems (Kvarnström et al., 2004), urban sewage sludge (Frossard et al., 1996; Guivarch, 2001) or thermo-chemically sewage sludge ashes (Nanzer et al., 2014). In addition, it is possible to analyze the positive or negative effects of applied products on P uptake from native soil P (e.g. Morel and Fardeau, 1989, 1990a,b).

Soil incubation experiments without plants are also used to assess the ability of the products to increase the amounts of phosphate ions in soil solution and isotopically exchangeable phosphate ions, and this ability can be related to data obtained with pot experiments (e.g. plant P uptake; Cabeza et al., 2011; Guivarch, 2001; Nanzer et al., 2014; Sinaj et al., 2002). Moreover, comparisons between incubation and pot experiment data may be used to show that plant-availability of recycled P can be inferred appropriately from simple soil incubations. It can also be required to use soil incubations with products to assess the eventual effects of product application on soil microbial activity (Oehl et al., 2001). Indeed, products may induce a flush in microbial activity, microbial immobilization of P derived from the soil and/or the products and consequently competition between soil microorganisms and plants for available P (McLaughlin and Alston, 1986; Mclaughlin et al., 1988). Such stimulation in microbiological soil activity needs to be accounted for since it may prevent any improvement in plant P nutrition and enhancement in plant growth after product application (McLaughlin and Alston, 1986).

The objective of the present study was to assess plant-availability of P recycled from pig manure and dairy effluents (produced through chemical- and bio-precipitation, respectively), using pot experiments with ^{32}P-labeling of soil. We also carried out soil incubations to assess i) the ability of the products to increase the amounts of phosphate ions in soil solution and isotopically exchangeable phosphate ions and ii) the product effects on soil microbiological activity. The recycled P products were compared to reference products: a water-soluble fertilizer (TSP), HA and ST.

2. Material and methods

2.1. Main characteristics and P status of pure products

A first recycled P product (hereafter named as “RPDE”) was provided by the “Biosystems and Process Engineering Laboratory” (LISBP–INSA Toulouse, France). It was derived from a dairy effluent through a bioprecipitation process and is composed of Ca–P, mainly as HA (Mañas et al., 2012). Other recycled P products (RPPM-1 to RPPM-4) were provided by the IRSTEA institute (“Unité de gestion environnementale et traitement biologique des déchets”, Rennes, France). They were derived from pig manures through a chemical precipitation process (acidification, addition of polymer, draining table step, MgO addition, and filtration). They were obtained with variable processing conditions, which resulted in different final compositions (Table 1; Daumer et al., 2013). TSP was a commercial product ($Ca(H_2PO_4)_2$), while reference HA ($Ca_5(PO_4)_3OH$, M = 502.31 g mol^{-1}, Acros Organics) and reference ST ($MgNH_4PO_4$, M = 137.31 g mol^{-1}; Sigma-Aldrich) were synthetic products. RPPM products include the 25–100 and >100 μm fractions. All other products were grounded using a mixer mill Retsch MM400 (final particle size ≈ 50–100 μm).

Organic carbon (C) and total nitrogen (N) in recycled products were determined by dry combustion (NF ISO 10694 and 13878; AFNOR, 1999), and total potassium (K), calcium (Ca) and magnesium (Mg)

Table 1
Compositions of RPPM products.

	% of products			ST/Ca–P ratio
	Recovered ST	Ca–P	MgO	
RPPM-1	19 ± 4	24 ± 5	57 ± 8	0.79
RPPM-2	19 ± 3	27 ± 3	53 ± 3	0.70
RPPM-3	12 ± 6	35 ± 5	55 ± 7	0.34
RPPM-4	38 ± 6	15 ± 2	47 ± 7	2.53

Data from Daumer et al. (2013).

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