



Article

Physico-Chemical Properties and Phosphorus Solubilization of Organomineral Fertilizers Derived from Sewage Sludge

Andre Luiz de Freitas Espinoza ^{1,*}, Henrique Rasera Raniro ², Camille Nunes Leite ¹
and Paulo Sergio Pavinato ¹

¹ Department of Soil Science, Luiz de Queiroz College of Agriculture (Esalq), University of São Paulo (USP), Pádua Dias Avenue, 11, Piracicaba 13418-900, SP, Brazil; cnl@plen.ku.dk (C.N.L.); pavinato@usp.br (P.S.P.)

² Department of Crop Sciences, University of Natural Resources and Life Sciences, Vienna (BOKU), Konrad-Lorenz-Strasse 24, 3430 Tulln, Austria; henrique.rasera-ranirro@boku.ac.at

* Correspondence: andreluizesp@usp.br

Abstract: Sewage sludge (SS) is an organic waste that may potentially be used as a slow-release source of phosphorus (P), despite the necessity for pre-treatment and its lower P content compared to soluble mineral fertilizers. For these reasons, composted sewage sludge was used to manufacture pelletized organomineral fertilizers, by mixing it with the inorganic sources monoammonium phosphate (MAP) and AshDec[®] (ASD) (thermochemically incinerated SS). The fertilizers were physiochemically characterized and evaluated for their P solubilization dynamics and lability in the soil. The sources tested were as follows: organic compost of sewage sludge powder (SSC) and its pelletized form (SCP), pelletized organomineral SSC + MAP (S + MAP) and SSC + ASD (S + ASD), ASD alone, compared conventional MAP and a control (nil-P). These fertilizers were applied to columns containing 50 g of soil at the dose of 100 mg P column⁻¹ and were leached daily with 30 mL of water or 2% citric acid for 30 days. We analyzed the leachates for pH and P content. Pelletizing process resulted in denser products and promoted more gradual P release. The organomineral S + MAP was the most water-soluble recycled source, solubilizing about 70% of the total P, while the others presented much lower solubilization (<20%). In contrast, all fertilizers showed high solubility in 2% citric acid (except for S + ASD). After leaching, soil P fractionation disclosed that the P leftover in the soil remained mostly in the labile and moderately labile pools. Composting and the ASD process produced materials with slow P solubilization, being favored in acidic soils and in plant's rhizosphere. In turn, S + MAP resulted in a promising product with intermediate P solubility, better synchronized with crop demand, potentially increasing P-use efficiency. Our results shed light in the physico-chemical properties and on the solubilization dynamics of novel organomineral products in tropical soil conditions.



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1. Introduction

Phosphate fertilizer use is critical in most production areas and aims to ensure a sufficient phosphorus (P) availability for crops, to achieve high productivities [1]. Most phosphate fertilizers used in modern agriculture come from the mining of phosphate rock (PR), which is acidulated to increase the water solubility of P. However, PR deposits are unevenly distributed worldwide, generating dependency on imports, increasing production costs and making agricultural activity vulnerable to price fluctuations in the international market [2,3].

Although P fertilization is a widespread practice, P use efficiency is very low, being on average 50% in highly weathered soils, which are predominant in tropical regions such as Brazil [4]. This occurs because conventional P fertilizers are highly soluble in water, leading to the rapid P release into the soil solution. If not immediately absorbed by plant roots, the soluble P can be immobilized by the soil colloidal fraction, which is composed of

clay minerals and Fe and Al oxyhydroxides, abundant in highly weathered tropical soils. Over time, the energy of these P fixing bonds increases, becoming virtually irreversible and greatly reducing P desorption to the soil solution for plant uptake [5–8].

An alternative way to increase P use efficiency in tropical soils is to utilize slow- or controlled-release fertilizers with the intent to allow for a gradual P solubilization, more synchronized with plant demand. Phosphate fertilizers derived from sewage sludge (SS) are among these slow/controlled release products [9–12], and besides helping to reduce dependency on fertilizer imports, are an option for reusing and recycling nutrients from local waste that would otherwise be mostly discarded in landfills, potentially polluting the environment.

The SS is the main residue generated in wastewater treatment plants (WWTPs), and is rich in organic matter, macro and micronutrients. However, its P content (5.8 and 30 g kg⁻¹) is considerably lower than that industrialized mineral fertilizers (80 to 240 g kg⁻¹) [3,13,14], requiring the application of huge volumes to meet the crop demands. Furthermore, to enable its agricultural use, SS must undergo pre-treatments to eliminate pathogens. Among these treatments, composting can be highlighted as an economical and efficient method to SS sanitization [15]. However, it does not solve the low nutrient content problem, while SS's bulky nature makes it difficult to transport, store, handle and apply.

The use of SS as a matrix for organomineral fertilizers can increase the nutrient concentration [16], while pelletization densifies the material, reducing its volume and delaying P solubilization, therefore avoiding losses to the environment [17,18]. Another alternative for P recovery is via thermochemical treatments of incinerated sewage sludge ash (SSA), such as the AshDec[®] process, which also reduce SS volume while concentrating the P and sanitizing the material [12,19–21]. The AshDec[®] process can increase P's solubility in citric acid from 40 to 90% [19], with potential agricultural uses, considering that the P₂O₅ concentration in SSA (15–25%) is comparable to commercially exploited phosphate rocks (25–36%) [22]. The whole treatment, processing and P recovery from SS aim to improve the agronomic characteristics of the final product, creating more efficient alternatives for P use compared to conventional fertilizers.

In this research we investigated an organic sewage sludge compost (SSC) collected from a Brazilian WWTP. Besides the raw SSC, two new organomineral phosphate sources were tailored by mixing and pelletizing the SSC with mineral phosphate sources, either monoammonium phosphate (MAP) or the SSA derived AshDec[®] product (ASD). By performing an experiment in leaching columns, we aimed to (1) physically and chemically characterize the novel recycled fertilizers and (2) evaluate the P solubilization dynamics and changes in leachate pH and soil P fractions, in comparison to conventional MAP.

2. Materials and Methods

2.1. Raw Materials

The research was undertaken using an organic compost generated in Brazil, provided by the Bela Vista WWTP, Piracicaba, São Paulo, Brazil. The AshDec[®] fertilizer (ASD) was provided by the German Federal Institute of Materials Research and Testing (BAM) and was obtained via the thermochemical treatment of German SSA, according to the method described in Stemann et al. [23], generating a fertilizer with the chemical formula CaNaPO₄.

To generate the SSC, the SS was mixed in windrows with tree pruning and grass clippings, as sources of structuring material. The compost pile was monitored by the São Paulo Agribusiness Technology Agency (APTA), which analyzed stability and maturity parameters. The SSC final product presented the following characteristics: water content = 38.1%; total, volatile and fixed solids = 59.6, 41.9 and 17.7%, respectively; electrical conductivity: 3020 µS cm⁻¹; C = 36.2%, N = 2.6%, C/N = 13.9; CTC = 1130.50 mmolc kg⁻¹; E. coli = 3.4 × 10² MPN g⁻¹ TS (most probable number per gram of total solids) and the absence of *Salmonella* sp. The observed parameters fit the Brazilian national criteria for application in soils established by the National Council Environment, Resolution 498/2020 [24].

The SSC was sieved to 4 mm and ground in a mill with a vertical rotor (Biotech—BT 608). To improve the physical-chemical characteristics of the product to be used as fertilizer, the pelletization and organomineral mixing of the SSC was performed.

2.2. Organomineral Fertilizers (OMFs) Production

By using the SSC as the organic matrix, we produced two pelletized OMFs. The mineral sources used in the composition of each fertilizer were ASD and MAP, both in powder form. The proportions of the OMFs' compositions were 70% organic compound and 30% mineral source. In addition, the pure SSC was pelletized for comparison. The solid components were mixed for 15 min in an adapted rotary cylindrical mixer with a 45° inclination, and then moistened with water until they presented an adequate consistency for pellet formation. The amount of water added per kg of dry mass corresponded to 0.45 L kg⁻¹ for S + ASD, 0.35 L kg⁻¹ S + MAP and 0.95 L kg⁻¹ for pure SSC.

The moistened matrix was pressed through the pelletizer and eventually passed through 3.4 mm openings to form the pellets. The pellet strands obtained were manually shaken, to break them into smaller pellets. These were placed in a forced circulation oven at 40 °C for 2 h. Afterwards, the pellets were air dried for 48 h and stored.

2.3. Physical Characterization

The physical properties of the recycled P sources and the granulated MAP, like density, humidity and resistance to compression (except for the powdered fertilizers), were evaluated in triplicates according to IFDC [25]. Bulk density (g cm⁻³) was obtained by weighing fertilizer samples in a box of known volume and dividing the weight of the sample by the volume of the container. Humidity (%) was determined by the difference between the weight of the sample before and after drying in an oven (FANEM 330) at 65 °C, until a constant weight was reached. The compression resistance test was performed in the Texture analyzer equipment (Brookfield CT3) by applying a load force equal to 25 kg to the fertilizers. Twenty-five pellets of similar size and granules were selected; each MAP granule and pelletized organomineral was placed individually under the platform and were pressed by a metal rod with a flattened tip, until the fertilizer was fractured. The force required (recorded in Newtons) was then converted to kilograms. In addition, the length and diameter of the fertilizers were sized with a digital caliper (Digital Caliper 150 mm).

2.4. Fertilizer P Solubilization Dynamics

To evaluate the P solubilization dynamics of the fertilizers in soil, a leaching column experiment was conducted under lab-controlled conditions for 30 days. The soil used was collected from the 0–20 cm layer of a Latossolo Vermelho-Amarelo, according to the Brazilian Soil Classification System, corresponding to an Oxisol, according to Soil Taxonomy [26], with loamy sandy texture and low P content, according to Cantarella et al. [27], located in a pasture area in Piracicaba, São Paulo, Brazil. The soil was air dried, sieved through a 2 mm sieve and analyzed for its chemical and textural parameters (Table 1).

Table 1. Soil chemical and textural parameters prior to experiment start.

pH	O.M.	P resin	S	K ⁺	Ca ²⁺	Mg ²⁺	Al ³⁺	H ⁺ Al	SB	CEC	V	m
CaCl ₂ 4.6	g kg ⁻¹ 14.0	mg kg ⁻¹ <6	9	1	4	3	2	mmol _c kg ⁻¹ 22	8	30	27	% 20
					Sand	Silt	Clay					
					g kg ⁻¹ 740	g kg ⁻¹ 90	g kg ⁻¹ 170					

O.M. = organic matter; SB = sum of bases; CEC = cation exchange capacity; V = base saturation; m = aluminum saturation.

For the experimental setup, acrylic columns with a 2.1 cm internal diameter and a 25 cm height, had their bottoms covered with nylon fabric and were fitted to a circular plastic base with an attached hose, to conduct the percolated leachate to the collecting jars (80 mL capacity) positioned below. The columns were placed in vertical positions on a medium density fiberboard support and filled with 50 g of soil (10 cm of soil). The soil was saturated with deionized water and then the treatments were positioned on top of the soil, with the P dose of 100 mg column⁻¹ for all sources. To avoid clogging and the disturbance of the soil surface by water droplets, 10 g of glass beads at the bottom and 5 g at the top were placed in the columns.

The experimental design was completely randomized, with three replicates, composed of the following treatments/sources: (1) powdered compost (SSC); (2) pelletized compost (SCP); (3) SSC + MAP pelletized (S + MAP); (4) SSC + AshDec[®] pelletized (S + ASD); (5) AshDec[®] powdered (ASD); (6) granulated monoammonium phosphate (MAP); and (7) control, nil-P (CTR). From a total of 42 soil columns, 21 were leached with deionized water and 21 with 2% citric acid.

2.5. Sampling and Analyses

A solution dosing system (300 mL) was attached on the top of each column with the drip adjusted, so as not to form a water sheet on the soil surface. The columns were watered every day for 30 consecutive days with 30 mL of deionized water or 2% citric acid solution. Leachates were collected 24 h after every application and weighed, and their pH was determined with an electronic pH meter. The leachates were stored and refrigerated in test tubes until the moment of the analysis. The P content was determined colorimetrically using the blue-molybdate method [28].

At the end of the experiment, the soil was collected and sectioned using the depths of 0–1, 1–2, 2–6 and 6–10 cm. Soil samples were dried in an oven at 40 °C until they reached a constant weight and were stored to undergo P fractionation as proposed by Hedley et al. [29], with modifications by Gatiboni et al. [30]. In short, 0.5 g soil samples were submitted to sequential inorganic (Pi) and organic P (Po) extractions in the following order: anion-exchange resin (Pi), NaHCO₃ 0.5 mol L⁻¹ (Pi and Po), NaOH 0.1 mol L⁻¹ (Pi and Po), HCl 0.1 mol L⁻¹ (Pi) and NaOH 0.5 mol L⁻¹ (Pi and Po). Every extraction was performed by shaking the samples in a Wagner Shaker (Tecnal TE-160) at 33 rpm for 16 h, being centrifuged at 3000 rpm for 15 min. The amount of inorganic P in each extract was determined using the molybdenum blue method [28]. Organic P was calculated with the difference between the total P (obtained by digesting the alkaline extracts) and the inorganic P of the alkaline extracts. After all the extractions, the remaining soil was dried in an oven at 40 °C until a constant weight was reached. We then weighed out 0.1 g of dry soil and submitted it to H₂SO₄ + H₂O₂ digestion, to determine residual P.

The P determined in each extraction was grouped according to the lability that correspond to the levels of P availability to plants; resin + NaHCO₃ were labile fraction, NaOH 0.1 + HCl were moderately labile and NaOH 0.5 + residual P were non-labile.

The recovered P from fertilizers was calculated according to Equation (1), considering the total P to be the amount of P added via fertilizers (100 mg column⁻¹) added to the total P of the control (no P application).

$$P \text{ recovery (\%)} = (P_{\text{leach}} + P_{\text{soil}}/P_{\text{ctr}} + P_{\text{fert}}) \times 100 \quad (1)$$

where P_{leach} corresponds to the total P leached out of the soil column for each source of P, P_{soil} is the soil total P determined by fractionation, P_{ctr} is the P leached out of the soil column plus the soil total P (determined by fractionation) of the control treatment and, finally, P_{fert} corresponds to the P added via fertilizers.

2.6. Statistical Analysis

The data were submitted to normality and homogeneity tests to verify if they met the model assumptions. An analysis of variance (ANOVA) was also performed and the means

that presented a significant difference at 5% probability were compared by the Scott–Knott test. The analyses were performed in R statistical software, using the ExpDes.pt package.

3. Results and Discussion

3.1. Physical-Chemical Characterization

The chemical composition of the fertilizers used to compose each treatment is presented in Table 2. Although only 16% of the total P (0.40% of 2.46%) in SSC is water-soluble, almost 90% is soluble in 2% citric acid (HCi), which has been shown to correlate well to plant-available P by mimicking the rhizospheric environment [31,32]. By incorporating mineral sources with higher P content (MAP and ASD) in the SSC, it was possible to increase the total P content in the organomineral sources S + MAP and S + ASD in 86% and 64%, respectively.

Table 2. Nutrient composition of the fertilizers used as treatment sources.

Fertilizer	N	K ₂ O	Ca	Mg	Na	P ₂ O ₅ Total	P ₂ O ₅ H ₂ O	P ₂ O ₅ HCi	P ₂ O ₅ NaC
	----- % -----								
SSC	3.73	1.10	3.08	0.63	0.09	2.46	0.4	2.20	2.30
SCP	3.73	1.10	3.08	0.63	0.09	2.46	0.4	2.20	2.30
S + MAP	5.91	0.77	2.16	0.44	0.06	17.34	13.48	-	17.21
S + ASD	2.61	0.64	4.36	0.76	3.34	6.75	0.49	6.22	-
ASD	-	0.33	9.50	1.50	11.00	16.60	0.70	15.60	-
MAP	11.00	-	-	-	-	52.00	44.00	-	52.00

P₂O₅ soluble in: HCi = citric acid, NAC = neutral ammonium citrate; SSC = organic sewage sludge compost; SCP = pelletized organic sewage sludge compost; SSC + MAP = organic sewage sludge compost + MAP; SSC + ASD = organic sewage sludge compost + AshDec[®]; ASD = AshDec[®]; MAP = monoammonium phosphate.

The total P present in recycled products is poorly soluble in water, but almost totally soluble in 2% citric acid, and the presence of other elements can also be highlighted. Due to the mixture of SS and plant material in composting, it was possible to increase the levels of K (0.19–1.10%) and Ca (0.82–3.08%) of the SSC (Table S1). In the composition of ASD, there were considerable quantities of the basic cations Ca and Na, which come from the addition of CaCl₂ and Na₂SO₄ in the thermochemical treatment of SSA. Their purposes are, respectively, to remove heavy metals by evaporation and to form alkaline phosphate compounds with a higher P bioavailability [22]. Despite its importance for plant nutrition, high Ca concentrations can promote the formation of Ca–P precipitates of lower bioavailability [21,33]. Meanwhile, the high content of Na is worrying, as it can salinize the rhizosphere and inhibit plant root development [34]. Therefore, part of the Na can be replaced by K in the AshDec process [22].

The physical parameters of the fertilizers are presented in Table 3. Pelletization increased the bulk density of the pure SSC by 38% (SCP), and of the organominerals S + MAP and S + ASD by 42% and 61%, respectively. These results corroborate several authors, including Nikiema et al. [35], where pelletization increased the density of products from fecal sludge by 20–50% compared to the powder form, and also the study by Hettiarachchi et al. [36], in which the pelletization of organic compost increased the density by 33–45%. Because of the mechanical pressure exerted on the pelletizing process, it is possible to increase the density of composted solid wastes and thus facilitate the use of SSC. Due to its bulky nature, its low density is one of the impediments that decrease the demand for the product, because it complicates handling, generates dust, occupies more storage space and makes transportation costly [36,37].

Table 3. Physical parameters of the waste-derived fertilizers, organomineral fertilizers and granulated MAP. Means are followed by the standard deviation.

Fertilizer	Diameter (mm)	Length (mm)	Humidity (%)	Density (g cm ⁻³)	Resistance (kg)
SSC	-	-	6.40	0.44	-
SCP	3.78 ± 0.14	15.50 ± 2.02	5.19	0.61	10.24 ± 2.12
S + MAP	4.16 ± 0.23	16.63 ± 2.22	3.98	0.67	8.08 ± 1.38
S + ASD	3.90 ± 0.12	19.12 ± 2.30	4.09	0.71	6.00 ± 1.96
ASD	-	-	0.00	1.17	-
MAP	3.80 ± 0.48	-	1.87	1.13	6.90 ± 1.19

SSC = organic sewage sludge compost; SCP = pelletized organic sewage sludge compost; S + MAP = organic sewage sludge compost + MAP; S + ASD = organic sewage sludge compost + AshDec[®]; ASD = AshDec[®]; MAP = monoammonium phosphate.

The importance of the pellet presenting a certain resistance to crushing is related to the maintenance of its physical integrity during transport, handling and storage [38]; therefore, a compression resistance test was performed on the pelletized fertilizers and the results were compared to granulated MAP (Table 3). The SCP pellets were the most resistant, followed by S + MAP and S + ASD, which were similar to granular MAP, signaling appropriate resistance of the pellets. These results indicate that the pelletization of these raw materials using only water could be a simple and cheap enhancement method to be performed during the manufacturing process. Pellet formation occurs through the inter-particle bonding between solid particles in high pressure and temperature conditions [39], and substances present in SS, especially lignin and protein, act as natural bindings that favor the structuring of pellets [40,41]. Although the connection mechanisms between organic components in pellets are known, investigations into the connecting mechanisms between organic and mineral compounds are lacking; this is what motivated our study.

3.2. Leaching Column Experiment

3.2.1. Leachate pH Values

The pH values of the leachates were monitored to relate the variations with possible reactions caused by the dissolution of fertilizers into the soil solution (Figure 1). The changes in pH values when deionized water was used were strongly influenced by the amount of water added daily to the columns, which saturated the soil pores with water for a considerable period of time and may have caused changes in the redox conditions of the soil. The control treatment pH was close to 5.0 at the beginning, increasing gradually until day 8 (6.8), and presented variations, until the end of the incubation, between 6.1 and 7.4. This same trend was also seen for all treatments, although with different amplitudes (Figure 1A).

Much of the increase in the pH of the leachates, including in the control, can be explained by the reduction in Fe III present in the Fe oxides of weathered soils such as Oxisols. When submerged in water, anoxic conditions may favor the reduction to Fe II, in a process in which H⁺ protons are consumed and soil pH increases, as observed by Lu et al. [42] in eight different acidic soils whose pH increased rapidly in 5 days under the conditions of low redox potential.

The pH variation observed initially in the treatment with S + MAP and MAP may have also occurred due to the solubilization of the MAP (NH₄H₂PO₄) and the formation of ammonium bicarbonate, a salt with high pH in solution [43]. However, it was expected that pH would decrease over time because of the dissolution of ammoniacal phosphate fertilizers, which would lead to the nitrification process of NH₄⁺ that generates acidity [44]. However, oxygen limitations in the environment due to the high water volume applied may have disfavored nitrification. Nevertheless, the average pH value of MAP (6.4) and S + MAP (6.6) were the only ones lower than the control (6.8).

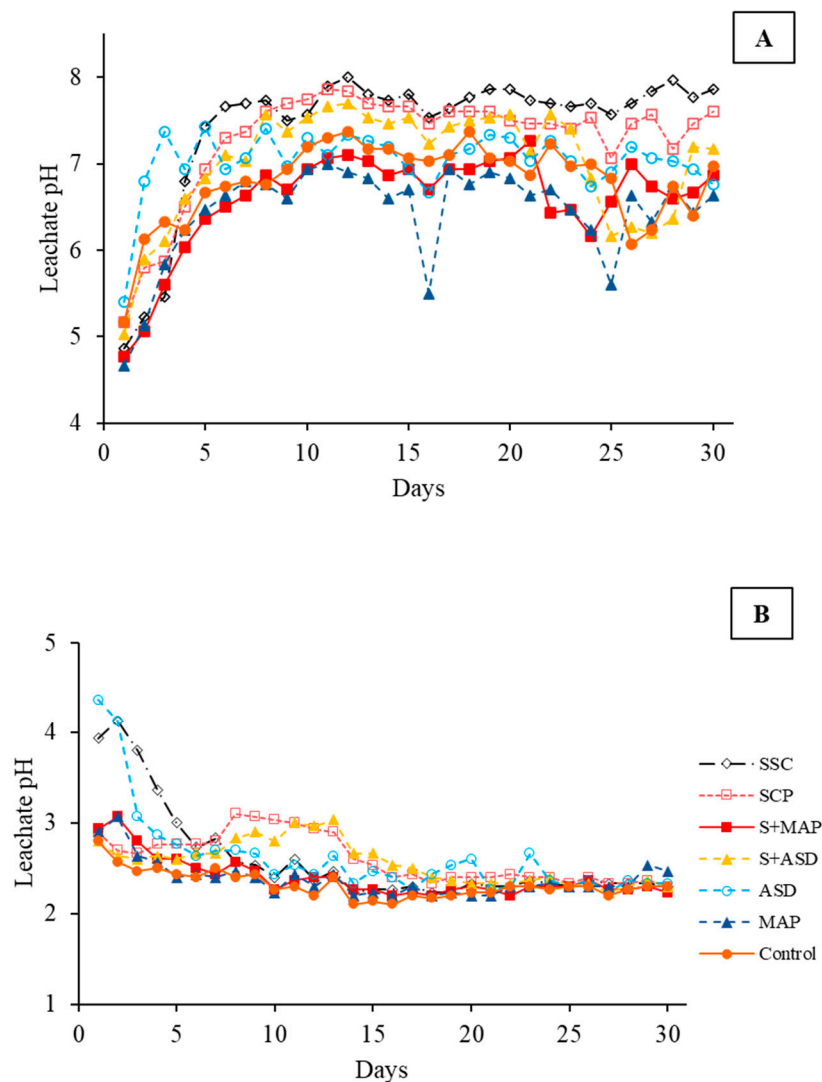


Figure 1. Daily average leachate pH under deionized water (A) and citric acid (B). SSC = organic sewage sludge compost; SCP = pelletized sludge compost; S + MAP = compost + MAP; S + ASD = compost + AshDec®; ASD = AshDec®; MAP = monoammonium phosphate.

For the other treatments the average pH stabilized after 7–8 days to just above the neutral value (SSC = 7.4; SCP = 7.3; S + ASD and ASD = 7.0). The SSC was the treatment with the greatest pH range (4.9–8.0), but a neutral/slightly alkaline pH prevailed for most of the observed time. Reports of pH increases in acidic soils due to the application of SSC are common in the literature, staying in the range from neutral to slightly alkaline, due to the ammonification process that consumes H^+ and generates OH^- ; however, subsequent ammonia nitrification is common, reducing it to nitrate and releasing H^+ , thus acidifying the soil soon after [45]. As can be observed, the pH values of the leachate did not decrease even at the end of the trial, which may be related to the low mineralization of the organic compost in the analyzed period. On the other hand, the constant leaching may have caused the removal of basic cations present in the compost, which would result in a higher pH value of the leachate.

Under 2% citric acid leaching test, there were smaller variations in pH values of the leachates over the time. This is because of the buffering capacity promoted by the percolated solution itself in the soil. The exception occurred in the first three days of leaching, where leachates from the SSC and ASD treatments were higher in the beginning, being 3.9 and 4.4, respectively, while the other treatments presented pHs close to the control, 2.8 (Figure 1B). This may be due to the rapid dissolution of these sources in the

citric acid solution, immediately increasing its pH. The pH (CaCl_2) of the SSC was 6.4 (Table S1), a result of the composting of SS with plant material, which starts with a pH close to neutral and decreases, due to the production of organic acids and CO_2 , until stabilizing due to the buffering action of humic substances formed [46,47]. The ASD, on the other hand, is a product of the mixture of phosphate with alkaline compounds (CaNaPO_4) with solubilization favored by the acidification of the medium [22]. However, from the sixth day and so on these two treatments stabilized the pH to values similar to other treatments (~2.5).

It is also noted that there were peaks in pH values of the SSC (3.1) and S + ASD (3.0) leachates between the 8th and the 13th day, in contrast to what occurred with the raw material of these powdered and non-pelletized sources, SSC and ASD. This observed trend reinforces the idea that the elements present in the pellet are protected and therefore solubilization is slower [16].

3.2.2. Fertilizer P Solubilization Dynamics

A higher P solubilization from recycled fertilizers was observed when leached with 2% citric acid than with water (Figure 2), which was expected, since most P in these sources is poorly water soluble and almost totally soluble in citric acid (Table 1). When leached by deionized water, treatments with MAP presented high P solubility, with similar behavior between MAP and S + MAP, since both solubilized more than 50% of the P in 5 days (Figure 2A). The total leached P (Figure 2C) was highest in MAP ($84 \text{ mg column}^{-1}$), followed by the organomineral S + MAP ($73 \text{ mg column}^{-1}$), while the other sources solubilized little during the incubation period, not exceeding 20% of the applied dose ($100 \text{ mg column}^{-1}$). This shows that the mixture of the low water solubility SSC with a highly soluble inorganic fertilizer resulted in a product that is able to release P into the soil within a short time after application.

Since MAP is an acidulated phosphate fertilizer with high water solubility, it is rapidly available in the soil solution [3]. The expected benefit of mixing such a fertilizer with a less soluble organic one is to better synchronize the availability of P with crop demand. The P from the mineral fertilizer is immediately available in the solution, supplying the demand in the early growth stages of the crop, while the P from the organic source is solubilized slowly, as the organic compounds are mineralized, meeting plants' needs in the later growth stages and potentially even being available for subsequent crops [16].

Most of the recycled sources evaluated here presented very low solubility in water (Figure 2C) but were highly soluble in citric acid (Figure 2D), where more than 85% of the applied P was leached, with the exception of the organomineral S + ASD, which solubilized only 42% of the applied P. If only the water solubility of recycled products were considered for their use as fertilizers, the agronomic potential of most would not be underestimated. For example, Raniro et al. [48] observed that the poorly water soluble ASD promoted the dry biomass production of sugarcane, equivalent to triple superphosphate, a high water soluble P source. For this reason, considering the solubility of fertilizers in citric acid (organic acid synthesized and exuded by plants' roots) may decrease the chance of misinterpretations related to the low water solubility of P and the availability of P to crops. Furthermore, to ensure a complete picture of the agronomic potential of alternative sources, this type of study should be related to plant experiments.

Although the total P leached in 2% citric acid was not significantly different between the recycled sources and MAP (except for S + ASD), the P solubilization rate was distinct (Figure S1). SCP and ASD showed less pronounced curves of solubilized P, indicating a slower and more gradual dissolution (Figure 2B). SSC, S + MAP and MAP had most of their P solubilized in the first days, while S + ASD solubilized P only from the 14th day and forward.

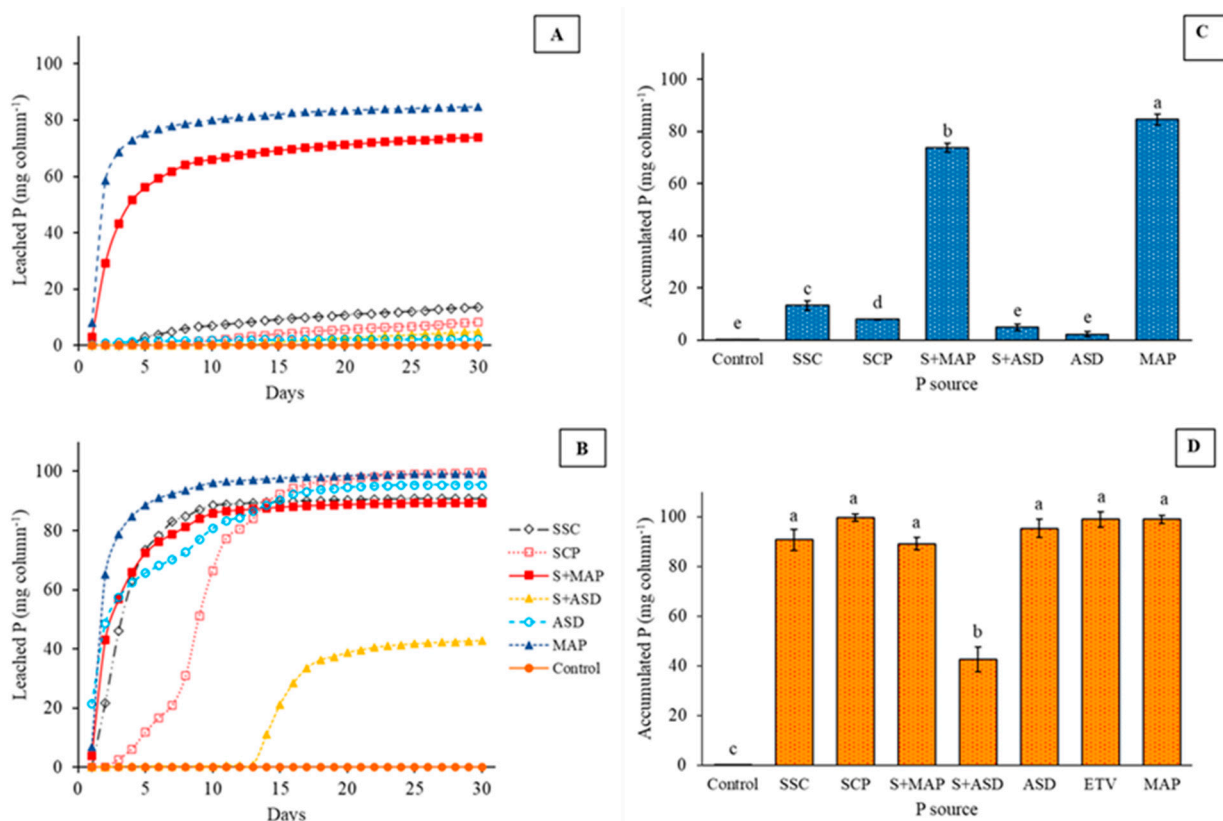


Figure 2. Accumulated daily P leached with water (A) and citric acid (B). Total P leached in water (C) and citric acid (D) in 30 days. Equal letters do not differ statistically according to the Scott–Knott test at a 5% significance level. Error bars represent standard error of the means ($n = 3$). SSC = organic sewage sludge compost; SCP = pelletized sludge compost; S + MAP = compost + MAP; S + ASD = compost + AshDec[®]; ASD = AshDec[®]; MAP = monoammonium phosphate.

The S + ASD treatment solubilized less P than its individual components (SCP and ASD), indicating that the pelletized mixture left the product more recalcitrant and impaired the release of P from the pellet. We also found that the physical form modified the dissolution dynamics of the fertilizer in soil, as SSC (powder) compared to its pelletized form (SCP), solubilized more P in water and had a faster initial solubilization in citric acid. This was expected due to the pellet having a lower specific surface area, which reduces the reactivity of the material and causes slower nutrient release [49].

Soluble phosphate fertilizers rapidly release P into the soil solution, which can be taken up by plants, shift to less labile forms through specific and nonspecific adsorption reactions with the surface of minerals, or precipitate with cations [50]. In weathered soils, where Fe and Al oxyhydroxides are predominant, sorption reactions are more rapid and intense, becoming practically irreversible as P remains in the soil [7,51]. Therefore, part of the P applied via fertilizer can be immobilized in the soil, decreasing the efficiency of fertilization by reducing P bioavailability in later stages of crop development.

In order to meet the P demand of the plant during its cycle, slow-release fertilizers are alternatives for supplying P in the long term, capable of making P available in a more synchronized manner with plant development, and thus avoiding environmental losses. In the initial phases, the plant does not yet have a well-developed root system, so if most of the P applied is made available immediately, little will be absorbed and most of it may be lost by surface runoff, leaching and/or erosion processes, or else reduce its lability in the soil and bioavailability at later plants stages [11,33].

3.2.3. Soil P Lability

Most of the remaining soil P in the columns leached with water was found in the labile and moderately labile pools in the 0–1 cm layer (Figure 3). This is indicative that these fertilizers' P solubilization and diffusion in the soil was low and/or slow. This allowed for the accumulation of P in the application site. The slow release of P from fertilizers is one way to avoid losses to the environment, either by fixation in the soil, making P less available to plants [44] or by leaching [52], all of which decrease fertilizer efficiency.

The ASD and S + ASD were the sources that increased the total soil surface P the most: 9778 and 1479 mg kg⁻¹, respectively, with expressive amounts in the labile fraction extracted with NaHCO₃ (Table S4). The labile P pool is the one in osmotic equilibrium with the soil solution; that is, it is more easily replaced either by the mineralization of organic P or the desorption of inorganic P [5,48]. This demonstrates that these fertilizers are capable of increasing available soil P as they solubilize over several days after application. Despite the high soil P concentration with S + ASD, only 18.3% of the applied P was recovered, indicating that most of it remained in the pellet (Table 4).

Most of the soil P with ASD was found in the moderately labile pool (7774.7 mg kg⁻¹) extracted by HCl (Table S2), usually associated with the desorption of Ca–P forms [53]. The formation of Ca–P under ASD may have been favored due to the high concentration of basic cations in this source. At high concentrations, P and Ca may precipitate, forming poorly water soluble minerals, which can solubilize in acidic conditions, such as that of the rhizosphere [54]. Furthermore, high moderately labile P values in the 0–1 cm layer under ASD could have partially come from fertilizer that was not completely dissolved, being sampled along with the soil, as supported by Nanzer et al. [55], who reported that the P in different thermochemically treated ashes were mostly in the forms of chlorapatite or hydroxyapatite, 90% of which could be extracted by HCl.

The moderately labile pool plays an important role as a source of P to the soil, as it keeps the solution P levels in equilibrium when the supplement of available P via fertilizer is insufficient to sustain plant growth. In fact, all less labile forms of P can function as a buffer in the soil when the P exported by crops exceeds the P content in solution. Intermediate labile P fractions can switch to more labile forms in order to maintain equilibrium; however, it may not solubilize as quickly as required by some crops [5,52,54].

Under low available P conditions, plants may adopt some strategies to access more stable forms in the soil, such as increasing the root system, interacting with microorganisms, the release of phosphatase enzymes, and increasing the exudation of organic acids in the rhizosphere [56]. Almeida et al. [57] found that tropical grass species, such as *Urochloa ruziziensis*, release more organic acids (citric, malic and lactic) in the rhizosphere under deficiency conditions than in P sufficiency. For this reason, plants that exude more organic acids are more likely to benefit from phosphate fertilization from slow-release sources. This was evident in the research of Talboys et al. [11], where the species that exuded more organic acids (*Fagopyrum esculentum*) was able to absorb more P than the species that exuded less (*Triticum aestivum*).

As for the recovery of P applied via fertilizers, the amount of P remaining in the soil in the treatments with S + MAP and MAP was smaller than the others, because much of the solubilized P was leached out of the soil columns, carried by water percolation. However, even so, the amount of P remaining in the soil was similar to the SSC and SCP sources. In the latter two, there was almost no dissolution of the fertilizers, as can be seen when the P balance is calculated (Table 4). Only 29% (SSC) and 20% (SCP) of the applied P was recovered in the leachate or in the soil. The non-recovered portion remained in the fertilizer pellets. Although much of the P in SSC is in inorganic forms, it does not mean that they are readily available to plants [9]. According to O'Connor et al. [58], high concentrations of Fe and Al (10–30 g kg⁻¹) in biosolids can reduce P bioavailability, as may have happened to SSC and SCP, and their low P solubilization is a consequence of the high Fe concentration (13 g kg⁻¹) found (Table S1).

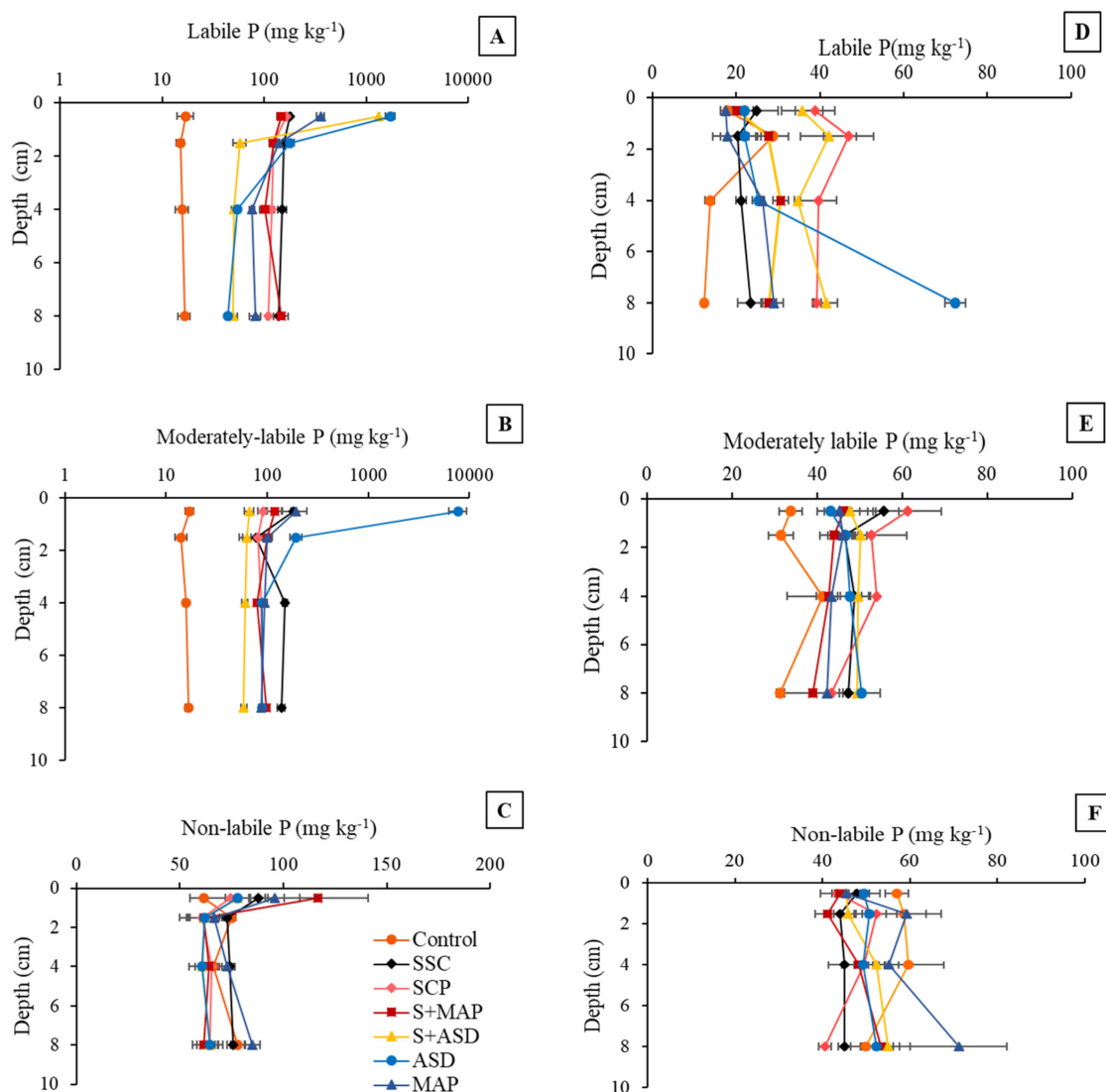


Figure 3. Soil P pools obtained by the soil fractionation method from columns leached with water (A–C) and citric acid (D–F), grouped by lability. Error bars represent standard error of the means ($n = 3$). SSC = organic sewage sludge compost; SCP = pelletized sludge compost; S + MAP = compost + MAP; S + ASD = compost + AshDec[®]; ASD = AshDec[®]; MAP = monoammonium phosphate.

Regarding the balance of P under 2% citric acid, most of the applied P leached out for all P sources (>85%), so there was minimal P remaining from fertilizer (<15%) in the soil (Table 4). Overall, the distribution of the soil P pools did not show large variations. The difference was small between the sources in the moderately labile and non-labile pools (Table S3 and S5). In the labile pool, however, ASD promoted more P in the 6–10 cm layer (Figure 3), indicating that there was movement of P in the soil profile when in contact with citric acid. The presence of P far from the application site indicates that there was P diffusion in the soil, but perhaps with a longer experimental duration, this labile P would move deeply in the soil profile until it completely leaves the column system.

Table 4. Percentage of P leached out, soil-accumulated labile, moderately labile and non-labile P pools, and recovered P relative to the total P originally in the soil + applied P dose.

Treatment	Leached P	Labile P	Moderately Labile P	Non-Labile P	Recovered P
----- (%) -----					
Leaching with deionized water					
Control	1.9	12.3	31.3	54.5	100.0
SSC	42.7	23.8	21.5	12.0	29.0
SCP	36.9	27.6	20.5	15.1	20.2
S + MAP	84.1	7.1	5.1	3.7	82.3
S + ASD	24.5	43.3	15.4	16.3	18.5
ASD	3.5	19.0	69.4	8.0	60.3
MAP	85.2	5.7	5.1	4.0	93.2
Leaching with citric acid					
Control	0.2	14.4	33.4	52.0	100.0
SSC	94.0	1.2	2.5	2.3	91.7
SCP	94.0	1.8	2.2	2.0	100.7
S + MAP	91.9	3.6	2.1	2.5	90.2
S + ASD	85.9	3.9	5.0	5.3	47.3
ASD	92.8	2.2	2.4	2.6	97.7
MAP	93.9	1.2	2.0	2.9	100.1

SSC = organic sewage sludge compost; SCP = pelletized organic sewage sludge compost; S + MAP = organic sewage sludge compost + MAP; S + ASD = organic sewage sludge compost + AshDec[®]; ASD = AshDec[®]; MAP = monoammonium phosphate.

The fact that the sources SCC, SCP, ASD and S + ASD solubilized P in citric acid, even though they are only slightly soluble in water, indicates that they could potentially supply P to plants in P-impooverished environments. Plants use some mechanisms to mobilize and absorb recalcitrant P, such as increasing the rooting system, excreting acid phosphatases, associating with mycorrhizal fungi and exuding organic acids, including citric acid [59]. For these reasons, in addition to fertilizer solubility, plant and soil types are important factors to consider in managing soil nutrients with recycled sources. Based on the results presented, it can be inferred that sources that are poorly soluble in water, such as SCC, SCP, ASD and C + ASD, would be more suitable for the cultivation of crops with a longer cycle, with the solubilization of ASD and S + ASD being favored in acidic environments, while SSC and SSP depend on OM mineralization. On the contrary, OMF formed by S + MAP, which mixes sources with high and low P solubility, has the potential to serve short-cycle crops, as it supplies P immediately and also gradually in the soil. Although these inferences can be made only after knowing the solubilization dynamics of the sources, their potential as a fertilizer must be further investigated in studies when plants are actively growing and extracting nutrients from soil solution.

4. Conclusions

Organic compost of sewage sludge proved to be in compliance with the norms for agricultural use according to the Brazilian normative instruction 61 [60]. Mixing and pelletizing organic sewage sludge compost with mineral sources was shown to have significant effects on the physico-chemical properties of the generated sources. Regarding the physical characteristics, the pelletization process, using only water, resulted in products with physical characteristics that are suitable for handling and storage, signaling the possibility of producing organomineral fertilizers through a relatively simple process without a need for synthetic binding substances. This represents one less input necessary to produce pellets, which can confer economic, technical and logistic advantages. However, this statement can only be confirmed through a study of the economic viability of producing these fertilizers. Furthermore, the pelletized form of the fertilizers promoted slower P release into the soil, relative to mineral granular and organic powdered forms.

From a chemical perspective, combining sewage sludge compost with monoammonium phosphate (70/30 *w/w*, respectively), generated an organomineral fertilizer (S + MAP) which exhibited a high P content and intermediate water solubility, therefore being potentially suitable for the cultivation of short-cycle crops. Meanwhile, combining the compost with a less water soluble source resulted in a product with lower solubility than its isolated raw materials (S + ASD). The fertilizers derived from sewage sludge presented low solubility in water, but they were highly soluble in citric acid, indicating that in acidic soil conditions, or through acidification by plant rhizosphere, this P may become available at a feasible time for crop utilization. Moreover, most of the P remaining in the soil from organic compost sources was found in the labile and moderately labile fractions, reinforcing the idea that these sources may have the ability to make P available in the medium to long term.

Future studies with the organomineral sources produced here will focus on the evaluation of their performance regarding plant nutrition and on the measurement of their residual effects in tropical soils for subsequent crops. The results obtained in this study can provide insights on the choice of phosphate sources to compose organomineral fertilizers according to the desired solubilization dynamics; while it serves as a basis for plant nutrition studies with organomineral sources, since they provides information about P release dynamics, as well as effects, in soil P pools.

Supplementary Materials: The following supporting information can be downloaded at: <https://www.mdpi.com/article/10.3390/soilsystems7040100/s1>; Figure S1: Daily leaching of P from the soil columns, with deionized water (A—left vertical axis for MAP and C+MAP, and right for the others) and citric acid (B). SSC = organic sewage sludge compost; SCP = pelletized organic sewage sludge compost; S+MAP = organic sewage sludge compost + MAP; S+ASD= organic sewage sludge compost + AshDec[®]; ASD = AshDec[®]; MAP = monoammonium phosphate; Table S1: Chemical analysis of sewage sludge and organic sewage sludge compost from WWT Bela Vista—Piracicaba, SP; Table S2: Soil P fractionation of columns leached with deionized water. Fractions separated by lability; Table S3: Soil P fractionation from columns leached with 2% citric acid. Fractions separated by lability; Table S4: Soil P fractionation of columns leached with deionized water. Fractions separated by extractors. Table S5: Soil P fractionation of the columns leached with 2% citric acid. Fractions separated by extractors.

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