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RESEARCH ARTICLE

Waste-derived fertilizers can increase phosphorus uptake by sugarcane and availability in a tropical soil#

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Abstract

Background: The use of highly water-soluble phosphorus (P) fertilizers can lead to P fixation in the soil, reducing fertilization efficiency. Waste-derived, low water-solubility sources can potentially increase sugarcane's P uptake compared to triple superphosphate by reducing adsorption to the soil.

Aims: We aimed to test struvite, hazenite, and AshDec® for their agronomic potential as recycled fertilizers for sugarcane production in a typical tropical soil. We hypothesize that these sources can reduce P fixation in the soil, increasing its availability and sugarcane's absorption.

Methods: In a greenhouse pot experiment, two consecutive sugarcane cycles, 90 days each, were conducted in a Ferralsol. The recovered sources struvite, hazenite, AshDec®, and the conventional triple superphosphate were mixed in the soil in three P doses (30, 60, and 90 mg kg^{-1}), aside a control (nil-P). At both harvests, sugarcane number of sprouts, plant height, stem diameter, dry mass yield, shoot phosphorus, and soil P fractionation were investigated.

Results: At 90 days, struvite and hazenite performed better for dry mass yield (70.7 and 68.3 g pot^{-1}, respectively) than AshDec® and triple superphosphate (59.8 and 57.4 g pot^{-1}, respectively) and for shoot P, with 98.1, 91.6, 75.6, and 66.3 mg pot^{-1}, respectively. At 180 days, struvite outperformed all treatments for dry mass yield (95.3 g pot^{-1}) and AshDec® (75.5 mg pot^{-1}) for shoot P. Struvite was 38% and hazenite 21% more efficient than triple superphosphate in P uptake, while AshDec® was 6% less efficient. Soil had higher labile P under struvite, hazenite, and AshDec® than triple superphosphate by the end of the first cycle, while only the later increased nonlabile P by the end of the experiment (180 days).

Conclusions: Waste-derived P sources were more efficient in supplying P for sugarcane and delivering labile P in 180 days than triple superphosphate.

KEYWORDS
AshDec®, hazenite, P efficiency, recycled sources, struvite

1 | INTRODUCTION

Sugarcane (*Saccharum* spp.) is a strategical crop due to its flexibility in producing sugar and ethanol according to market prices and demand. Brazil is the largest sugarcane producer worldwide, responsible for the production of 642 million tons of sugarcane in 2019–2020 (Companhia nacional de Abastecimento, 2019) and has been growing at an accelerated pace, more than doubling its planted area from 2001 to 2009 (MAPA, 2009) and increasing 14.3% from 2009 to 2019 (Miranda & Martinho, 2021).

One of the most important factors restraining Brazilian sugarcane productivity is the low phosphorus (P) availability and the high P-fixation capacity of most soils in the country, reducing fertilization efficiency (Soltangheisi et al., 2019). To overcome these issues and supply plants demand, large amounts of mineral P fertilizers are used annually, leaving behind a large P footprint (Roy, et al. 2016; Withers et al., 2018). This scenario is concerning, since the general P scarcity, phosphate rock prices instability, and a depletion of less polluted phosphate rock reserves could ultimately endanger Brazilian sugarcane production by making it unsustainable and/or financially impracticable (Mew, 2016).

The P fertilizers currently used in Brazilian sugarcane production are mostly highly water-soluble and usually applied at crop establishment, while cover applications are often reduced or overlooked due to its believed low plant response (Rossetto et al., 2002; Soltangheisi et al., 2019; Vitti & Mazza, 2002). Nevertheless, alternative slow-release fertilizers could be used as P sources to potentially increase crop P uptake (Soltangheisi et al., 2019). Among those, the waste-recycled struvite ($MgNH_4PO_4 \cdot 6H_2O$) has shown great potential in multiple studies with different crops (Gell et al., 2011; Katanda et al., 2016; Rech et al., 2019), although specific studies for sugarcane in tropical soil conditions are scarce.

Hazenite [$KNaMg_2(PO_4)_2 \cdot 14H_2O$], in turn, is a newly discovered struvite-type mineral also recycled through chemical precipitation from municipal or industrial wastewaters and could serve as an alternative to conventional fertilizers (Watson et al. 2020; Yang et al., 2011). Besides being environmentally friendly (Kraus et al., 2019), they present low water solubility, reducing P fixation in the colloids and thus being potentially more effective in agricultural systems than conventional fertilizers (Cabeza et al., 2011; Watson et al., 2020; Yang et al., 2011).

Moreover, the incineration of sewage sludge into ashes (SSA) and its thermochemical posttreatment with alkaline compounds at 900°C can also recover P by transforming the predominant poorly soluble Ca-P and Al-P forms present in SSA (Peplinski et al., 2013) into bioavailable $Ca(Na,K)PO_4$ (Herzel et al., 2016; Stemann et al., 2015). At the same time, toxic trace elements such as As, Pb, Cd, and Hg are evaporated and removed from the fertilizer product. The AshDec® fertilizer contains citrate-soluble $CaNaPO_4$ in concentrated form that can be directly applied onto the fields (Hermann & Schaaf, 2019).

Our hypothesis is that struvite, hazenite, and AshDec® can be more efficient phosphate fertilizers in a tropical soil condition, reducing P adsorption and improving sugarcane P absorption efficiency compared to the conventional highly water-soluble triple superphosphate (TSP). Our objectives were to evaluate the efficiency and residual effect of these three recycled sources in short-term sugarcane P supply compared to TSP, assessing their forms in the soil in two 90 days' consecutive cycles, while understanding how it affected sugarcane's growth and development, along with soil chemical parameters.

2 | MATERIALS AND METHODS

The experiment consisted of a greenhouse pot trial to compare the effects of TSP to three distinct sources recycled from wastewater treatment plants (WWTPs) located in Germany, namely, struvite, hazenite (both precipitated from wastewaters), and AshDec®, obtained from the thermochemical treatment of SSA.

The chosen sugarcane (*Saccharum* spp.) variety was the RB96-6928, once it is vastly cultivated in Brazil and has a vigorous root system with great capacity to explore the whole soil volume, besides its high response to P fertilization reported in a previous study (Arruda et al., 2016). Establishment was done by transplanting 4-week-old pre-sprouted seedlings from a substrate (rice straw) to PVC pots filled with 6 kg of dried, sieved, and fertilized soil (distinct treatments). Before transplanting, the seedling roots were washed with tap water. The pots were placed inside a greenhouse located in the city of Piracicaba-SP (Southeast Brazil).

2.1 | Soil characterization and fertilization

Soil was collected from the surface layer (0–20 cm) of a grassland area in southeast Brazil, classified as a sandy clay loam Ferralsol (FAO, 2014), with very low P availability (Raij et al., 1997; Tiessen et al., 1983). The soil was air-dried and sieved using a 2-mm mesh sieve. Its chemical and textural characterization is shown in Table 1.

As the soil pH was acidic and Ca and Mg levels were also below the adequate limits for sugarcane, $CaCO_3$ and $MgCO_3$ were incorporated homogeneously and incubated for 15 days prior to sugarcane establishment to elevate the Ca level to 20 $mmol_c\ kg^{-1}$ and Mg level to 8 $mmol_c\ kg^{-1}$, incorporated homogeneously, according to regional recommendations. After the incubation, the soil pH_{CaCl_2} was increased to 5.6 ± 0.3. Potassium was also homogeneously added to the soil via KCl (50 mg kg^{-1}), to supply the initial sugarcane demand. Moreover, complementary K and S were supplied via K_2SO_4 surface fertilization 50 days after establishment, when 50 mg kg^{-1} of K and 16 mg kg^{-1} of S were applied. In the first cycle, nitrogen was added via NH_4NO_3, in the dose of 150 mg kg^{-1} divided in three similar applications, at 25, 50, and 75 days after establishment.

Phosphorus was only applied once at trial establishment, with varying sources and doses (treatments). The composition of the fertilizers used for each treatment is shown in Table 2. The N content of struvite and K content of hazenite were discounted to maintain equity in total nutrients applied.

TABLE 1 Soil chemical and textural parameters before experimental setup

pH ($CaCl_2$)	O.M.	P_{resin}	S	K^+	Ca^{2+}	Mg^{2+}	Al^{3+}	H + Al	SB	CEC	V	m
–	(g kg^{-1})	(mg kg^{-1})		(mmol$_c$ kg^{-1})							(%)	
4.9	16	5	6	<0.9	13	5	2	16	18.8	34.8	54	10
	Clay					Silt				Sand		
						(g kg^{-1})						
	201					11				788		

Abbreviations: CEC, cation exchange capacity; m, Al saturation; O.M., organic matter; SB, sum of bases; V, base saturation.

TABLE 2 Nutrient content of fertilizer sources composing the treatments for pot trials

	K	Ca	Mg	N	Na	P total	P water	P HCi
Fertilizers					(%)			
Struvite	–	–	8.3	6.0	–	12.5	0.65	12.4
Hazenite	7.1	–	7.3	–	–	10.7	1.44	10.6
AshDec®	0.8	9.5	1.5	–	11.0	7.24	0.30	6.80
TSP	–	10.0	–	–	–	19.6	17.6	19.6

Abbreviations: HCi, soluble in citric acid 2%; Water, water-soluble.

After the first harvest, 50 mg kg^{-1} of both N and K and 16 mg kg^{-1} of S were immediately applied to all pots. After 30 days, yet another 50 mg kg^{-1} of N was added, using the same previously mentioned source. For micronutrients, boron, zinc, and copper were added via H_3BO_3 (0.8 mg kg^{-1}), $ZnSO_4$ (4 mg kg^{-1}), and $CuSO_4$ (1.3 mg kg^{-1}), respectively, dissolved in 100 mL of water and applied at 25 days after plant establishment. All fertilizer doses were defined according to the recommendation of de Sousa and Lobato (2004).

Plants were watered daily, maintaining 60%–70% of the soil's maximum water holding capacity throughout the whole duration of the experiment. The greenhouse temperature was kept within the range of 30°C during the day and 18°C at night.

2.2 | Treatments

The tested sources were struvite, hazenite, AshDec®, and TSP. For the four sources, three P doses were administered (30, 60, and 90 mg kg^{-1}) based on their total P content. One control treatment (nil-P) was added for comparison, resulting in a total (4 × 3) + 1 design. Pots were distributed in randomized blocks with four replicates. The three recycled sources as well as TSP were finely ground and applied at establishment, the latter being used as a positive control.

2.3 | Sampling procedures and measurements

Sugarcane development was evaluated in two cycles of 90 days each (90 and 180 days after establishment). After the first cycle, the aboveground biomass was harvested for dry mass yield (DMY) and nutrient tissue content determination. Plants' sprouts were left for regrowth for another 90 days and harvested again for the same analyses. Right before the first and second harvest, biometric measurements were taken, including cane diameter, plant height, and number of sprouts.

The shoot DMY of both harvests was obtained after drying in an oven at 60°C. Shoot material was milled at 1 mm prior to sulfuric digestion for nutrient content determination (Tedesco et al., 1995). Once digested, the material was tested for its P content by the phosphomolybdate blue method (Murphy & Riley, 1962). Nitrogen content was determined by distillation in alkaline solution by micro-Kjeldahl method, collected in boric acid 4%, and then titration by sulfuric acid solution. Accumulated P in tissue (mg pot^{-1}) was estimated by considering P concentration in each harvest, multiplied by each shoot dry mass.

Moreover, the relative P efficiency (RPE) of the recycled sources was estimated by comparing the accumulated (sum of both harvests) shoot P to that of TSP, as done by Vogel et al. (2015). The equation for RPE is expressed below:

$$\mathrm{RPE} = \frac{\textit{Shoot P promoted by recycled source}}{\text{P uptake promoted by TSP}} \times 100. \qquad (1)$$

Soil was sampled right after each harvest (90 and 180 days) to determine the nutrient availability (P, K, Ca, and Mg) according to Raij et al. (2001) (Table S1). Furthermore, subsamples were submitted to P fractionation as described by Hedley et al. (1982), modified by Santos (2000), in which the 0.1 molar NaOH + sonication is substituted by a 0.5 molar NaOH extraction. The data were grouped in labile P [extracted by anion-exchange resin (membrane) and $NaHCO_3$ 0.5 mol L^{-1})], moderately-labile P (extracted by NaOH 0.1 mol L^{-1} and HCl 1 mol L^{-1}), and non-labile P (NaOH 0.5 mol L^{-1} and residual P) for a more comprehensive discussion.

Optimal P doses were calculated by using the values obtained for each curve ($y = ax^2 + bx + c$) according to the Equation (2) expressed below:

$$\text{Optimal P dose} = \frac{-b}{2a}. \tag{2}$$

2.4 | Statistical analysis

All data were tested for homogeneity of variance and normality of the residuals by the Shapiro–Wilk and Levene tests, respectively. Subsequently, analysis of variance (two-way ANOVA) was used to compare means at the same harvest and a three-way ANOVA to compare means between harvests. Means were compared through Tukey test at 5% error probability. Regression analysis was also performed to describe the effects of P dose and the interaction dose × source for the variables analyzed. We evaluated linear and quadratic models and selected the best one for each variable based on the coefficient of determination (R^2) and model significance ($P > F$). All the analyses were performed in the R statistical software considering $p \leq 0.05$ as cutoff for statistical significance.

3 | RESULTS

3.1 | Sugarcane biometric parameters

The sugarcane number of sprouts, plant height, and stem diameter at both harvests are available in Table S2, and will not be discussed since only minor differences were observed among fertilized treatments and no interaction of source × dose was present. While P source affected most variables (except number of sprouts at 180 days) in comparison to control, only minor differences were observed between the fertilized treatments for the same harvest, with no interaction between source and dose. Sugarcane DMY was also not affected by the interaction P source × dose in both harvest periods, showing only effect of each factor individually (Figure 1A,B). A positive correlation between doses and DMY was seen at both harvests for all sources (Figure 1A). The optimal P doses for DMY found in our experiment were 84.6 and 79.0 mg kg^{-1} (169.2 and 158 kg ha^{-1} of P correspondent into the 0–20 cm layer with soil density of 1.0 kg dm^{-3}) at 90 and 180 days, respectively.

Significant differences in DMY were observed between sources (Figure 1B). At 90 days, struvite (70.7 g pot^{-1}) and hazenite (68.3 g pot^{-1}) outperformed AshDec® (59.8 g pot^{-1}) and TSP (57.4 g pot^{-1}), all being significantly higher than control (21.5 g pot^{-1}). At 180 days, struvite's DMY (95.3 g pot^{-1}) was significantly higher than all treatments. Values were considerably higher at the second harvest for all treatments (except control), being on average 30.9% higher than observed in the first harvest.

3.2 | Sugarcane accumulated shoot P

The shoot P accumulated by sugarcane was affected by P dose and source at both 90 and 180 days evaluations (Figure 2A,B), but not by their interaction, similar to what was observed for DMY. The shoot accumulated P under struvite (100.3 mg pot^{-1}) and hazenite (91.6 mg pot^{-1}) was higher than those under TSP (65.4 mg pot^{-1}) and AshDec® (76.8 mg pot^{-1}) at 90 days (Figure 2B). At 180 days, most plants were capable of absorbing more P in comparison to 90 days, possibly as consequence of a better plant growth. Only struvite (110.0 mg pot^{-1}) was more efficient than AshDec® (76.39 mg pot^{-1}), while all treatments performed significantly better than control on both harvests.

The relative P efficiency (RPE) of the recycled sources in relation to TSP, considering here the sum of both periods (90 and 180 days), is presented in Table 3. Differences in efficiency between sources were apparent at the lowest dose (30 mg kg^{-1} P), in which struvite and hazenite were significantly better than AshDec® and TSP. At 60 mg kg^{-1} P, only struvite differed from all, and no differences were observed under 90 mg kg^{-1}. On average of doses, struvite was 38% more efficient than TSP in accumulating sugarcane shoot P over the course of the experiment. Hazenite was 21.3% more efficient and AshDec® was 6.4% less efficient than TSP, but the latter not statistically significant in any dose.

3.3 | Soil P fractions and lability

For better comprehension, soil P fractions were grouped here by their lability pools. The complete data of soil P fractionation discerned by each extractor (and organic or inorganic forms) are available in Table S3. The labile P pool was affected by the interaction source × dose at 90 days (Figure 3), but only by source (Figure 4A) and dose (Figure 4B) individually at 180 days.

In the first cycle (90 days), TSP was responsible for the lowest labile P levels compared to the other sources in the same doses, while struvite tended to outperform both TSP and hazenite in this regard at higher doses, being comparable to AshDec® (Figure 3). In the second cycle (180 days), there was a general depletion of labile P in relation to 90 days, explained by plant uptake. At this time, all sources showed comparable levels of labile P (40.4 mg kg^{-1} on average) directly correlated with dose, only differing from control (25.1 mg kg^{-1}) (Figure 4A,B).

Moderately labile P was affected by the interaction source × dose at 90 days, and by dose only at 180 days (Figure 5). At 180 days, the moderately labile P diminished considerably for all sources, meaning that this fraction has contributed to P dissolution and thus to plant uptake. AshDec® presented a particularly greater reduction in this P pool (on average >22% depletion), no longer differing from other treatments.

The non-labile P was the pool that presented the highest values among all labilities throughout all treatments, representing ≈45% of the total soil P in the fertilized treatments and ≈50% for control in both harvests. This fraction was the less affected by the treatments at

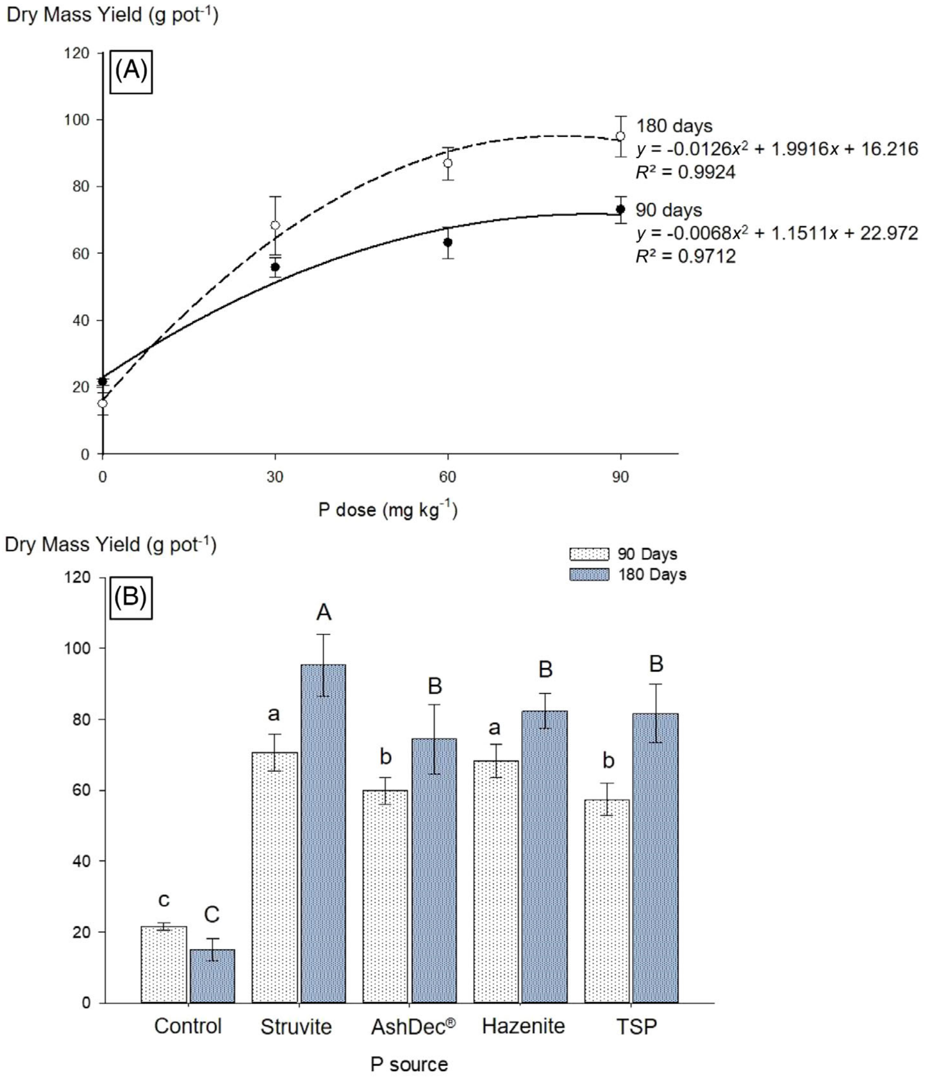

FIGURE 1 Sugarcane mean dry mass yield (g pot^{-1}) as affected by phosphate doses (A) and sources (B) of recycled phosphate sources at 90 and 180 days after seedlings establishment ($n = 4$). Means followed by the same letter in the same harvest do not differ by 5% error probability in Tukey test. Error bars represent standard error

90 days, where no effect of source nor dose was observed (data not presented). However, the source × dose interaction influenced the soil nonlabile P at 180 days (Figure 6), when TSP diverged significantly from other treatments.

4 | DISCUSSIONS

The discussion section will be organized by focusing on the effects of the fertilizer in the soil and plant and their agronomic potential, not considering current prices or market availability scenarios. This is the case since it is difficult to have an accurate production cost estimation of recycled sources due to major differences in the characteristics of WWTPs worldwide (and even within a same country), wastewater and sewage sludge element content, fluctuation of reagent prices, and so forth.

However, authors such as Shu et al. (2006), Egle et al. (2016), and Kok et al. (2018) have stated that the P recovery from waste is economically viable in multiple scenarios, even decreasing the WTTPs operational costs in some cases. This is especially the case in more developed regions such as Europe, where new P recycling policies are currently being implemented like the Green Deal "Farm-to-Fork" strategy that will pressure countries to recycle their waste and reduce fertilizer input, which will ultimately help recycled sources to be competitive in the market (European Commission, 2019).

4.1 | Sugarcane biometric parameters

Overall, all three recycled sources were comparable to or outperformed TSP. Although the experiment was carried out in similar conditions for all treatments, pot size may have limited plant growth that

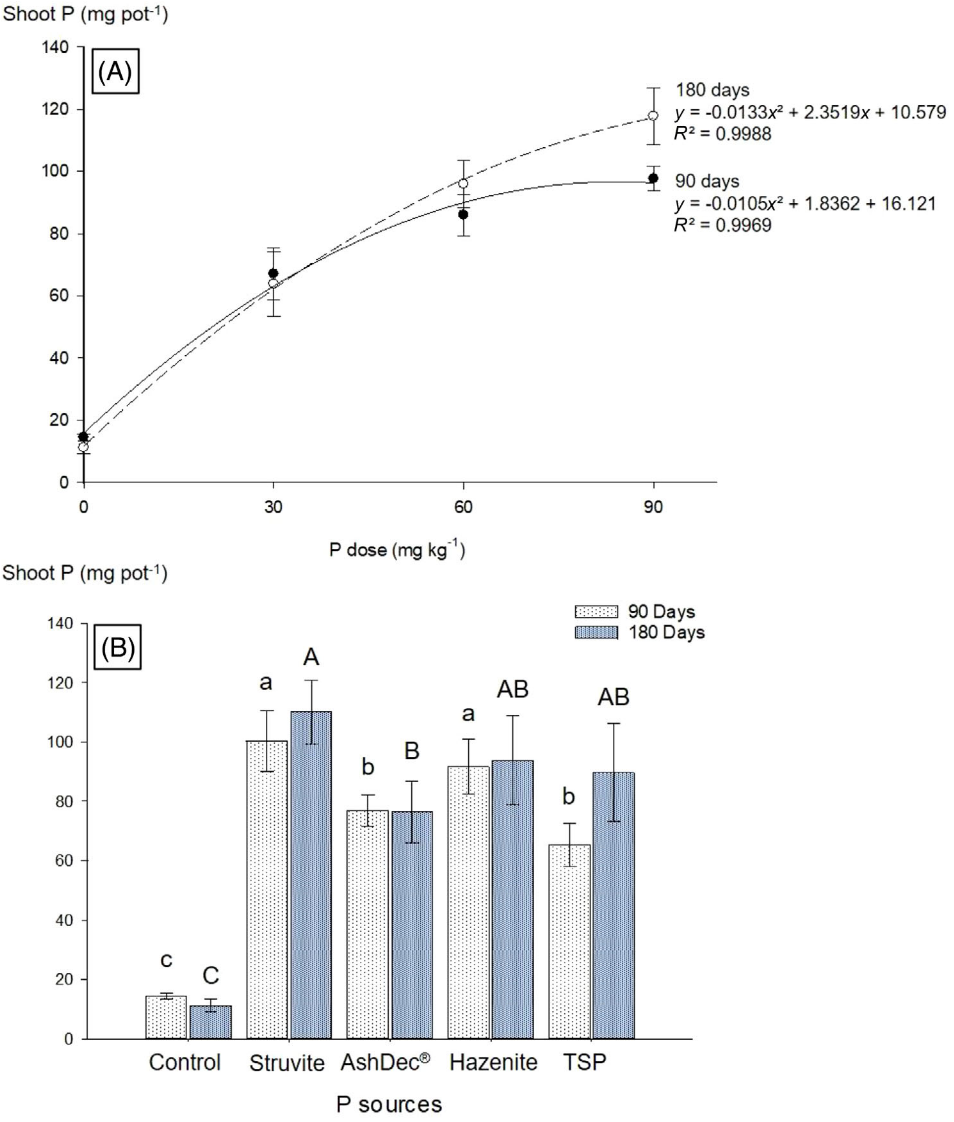

FIGURE 2 Sugarcane mean shoot P (mg pot^{-1}) as affected by phosphate doses (A) and sources (B) of recycled phosphate sources at 90 and 180 days after seedlings establishment ($n = 4$). Means followed by the same letter in each harvest do not differ by 5% error probability in Tukey test. Error bars represent standard error

could have contributed to mask certain treatment effects (Kiær et al., 2013), "homogenizing" the biometric results. Moreover, sugarcane is well known for its great capacity of overcoming stressful scenarios, which might have also contributed for a similar growth and development irrespective of doses and fertilizers (Azevedo et al., 2011; Grivet & Arruda, 2002).

For DMY, higher yields were observed in the second cycle compared to the first, and a possible explanation is that the root activity and residual organic acids exuded from the first cycle could promote a higher solubilization of phosphates, which were readily absorbed by the already established root system at sprouting, as suggested by Talboys et al. (2016) and by Hermann and Schaaf (2019), to be an important factor for both struvite and AshDec® solubilization.

In Brazil, phosphate fertilizers are usually added into the furrow during sugarcane planting, and doses can be as high as 78.5 kg ha^{-1} P under adequate soil P level (Rein et al., 2015). Moreover, in cases of low soil available P level, an additional fertilizer P correction is also recommended before planting, varying from 35 to 131 kg ha^{-1} P, normally added onto the soil surface and incorporated in full soil volume (0–20 cm) (de Sousa & Korndörfer, 2011). The optimal doses for DMY promotion found in our study are consistent with the P correction and fertilization rates described in the literature by authors such as Rein et al. (2015), Mendonça et al. (2015), among others.

At 180 days, struvite outperformed all other sources in DMY, while others only differed from control. Higher DMY production and the effectiveness of struvite compared to water-soluble P sources were also reported by Katanda et al. (2016) on canola and by Barak and Stafford (2006) and Gell et al. (2011) on maize, while Massey et al. (2009) and Rech et al. (2019) reported a similar performance by

TABLE 3 Mean relative P efficiency (RPE) of recycled fertilizer sources and doses related to TSP in sugarcane's shoot P during two consecutive cycles of 90 days

	P dose (mg kg^{-1})			
	30	60	90	
Fertilizer		%		Mean
Struvite	178 ± 8.1^{a}	127 ± 16.4^{a}	109 ± 11.0^{ns}	138.0
Hazenite	166 ± 14.9^{a}	97 ± 6.5^{b}	101 ± 8.0	121.3
AshDec®	96 ± 7.2^{b}	91 ± 11.7^{b}	94 ± 11.0	93.6
TSP	100^{b}	100^{b}	100	100
Mean	135.0	103.7	101.0	

Note: Means followed by the same letter do not differ by 5% error probability in Tukey test; ns = non-significant ($n = 4$). Errors represent standard error.

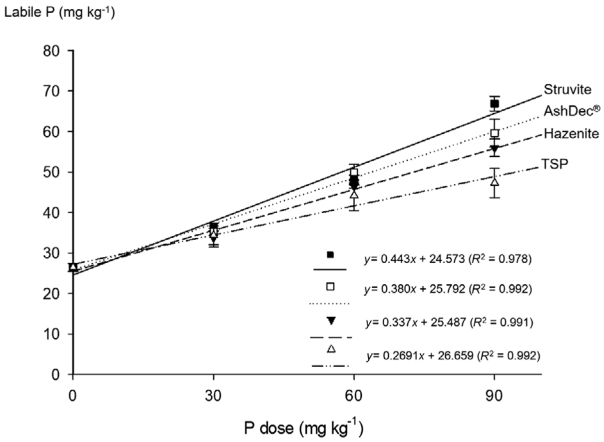

FIGURE 3 Soil labile P (mg kg^{-1}) as affected by the interaction phosphate source × dose at 90 days after establishment ($n = 4$). Means followed by the same letter do not differ by 5% error probability in Tukey test. Error bars represent standard error

different struvites compared to TSP on soybean and spring wheat, respectively.

In our trial, it is possible that the Mg content of struvite was a determining factor for its considerably higher DMY, since higher Mg uptake was observed for the struvite treatments (Figure S1), potentially allowing this source to outperform TSP and AshDec®, which performed similarly. Corroborating to our data, Severin et al. (2014) also reported similar yields for maize when treated with different types of treated sewage sludge ashes (SSAs) compared to TSP.

4.2 | Sugarcane accumulated tissue P

TSP performed significantly worse in tissue P than struvite and hazenite after the first cycle; however, it was comparable to all P sources in the second cycle. An explanation is that TSP solubilized its P very quickly after application, when plants were not yet fully established. This allows a premature adsorption of orthophosphates into the soil colloids, ultimately reducing their plant availability (Fink, Inda, Bavaresco, et al., 2016; Mng'ong'o, 2018). These phosphates, however, may not be too strongly adsorbed, and may be later accessed when roots were more developed (second cycle). Contrariwise, AshDec®'s solubilization might have been too slow, being a detrimental factor in its performance by delaying P availability for a longer period and ultimately reducing plant growth and shoot P content, compromising its efficiency in the second cycle.

4.3 | Relative P efficiency

There was an overall good response from the recycled P sources in terms of P efficiency, with struvite and hazenite being more efficient than TSP, and AshDec® being marginally lower. The same trend was reported by Vogel et al. (2017) for forage rye, who observed that in a field scenario struvite had an RPE 10% higher than TSP, while SSAs tested underperformed by 4%–9%, and by Vogel et al. (2015) in a greenhouse scenario using five different crop species, where struvite's RPE was up to 19% higher than TSP, while SSA-based recycling fertilizers were 1.3%–6.9% lower. Franz (2008) also reported that plants fertilized with SSA-derived sources showed similar P absorption compared to commercial superphosphates for three different plant species.

The significant differences between sources at lower doses observed in the trial are logical because when less fertilizer is added, a higher percentage of the total P applied will be adsorbed by the colloids, once the low P concentration cannot immediately saturate all soil P-fixing sites. Since highly water-soluble sources are promptly solubilized, soil particles can quickly adsorb its phosphates, reducing its relative efficiency at lower doses.

Although struvite, hazenite, and TSP allowed comparable shoot P in the second harvest, struvite's DMY was significantly higher than under hazenite (90 days) and TSP (both harvests). This is due to P being one of the plant macronutrients with the lowest use efficiency in terms of DMY production (Borges et al., 2019). Possibly, plants were capable of accumulating and storing P rather than metabolizing it, not being fully effective in its use (Marschner, 2012).

The same reasons behind struvite's higher DMYs when compared to the AshDec® could also help to explain the differences in shoot P. A further explanation could be that the citric acid-soluble P obtained for the AshDec® might have been overestimated for the environment in which the plants grew, thus presenting reduced availability and therefore lower P uptake. Another aspect could be the presence of ammonium in the struvite composition, which is released to the solution with fertilizer solubilization and can undergo nitrification via ammonium oxidizing bacteria, even in slightly acidic pH conditions (De Boer & Kowalchuk, 2001), generating nitrate (NO_3^-). This nitrate is then taken up by the plant, alkalinizing the rhizosphere, which can mobilize adsorbed P, possibly explaining the better performance of struvite in comparison to AshDec®.

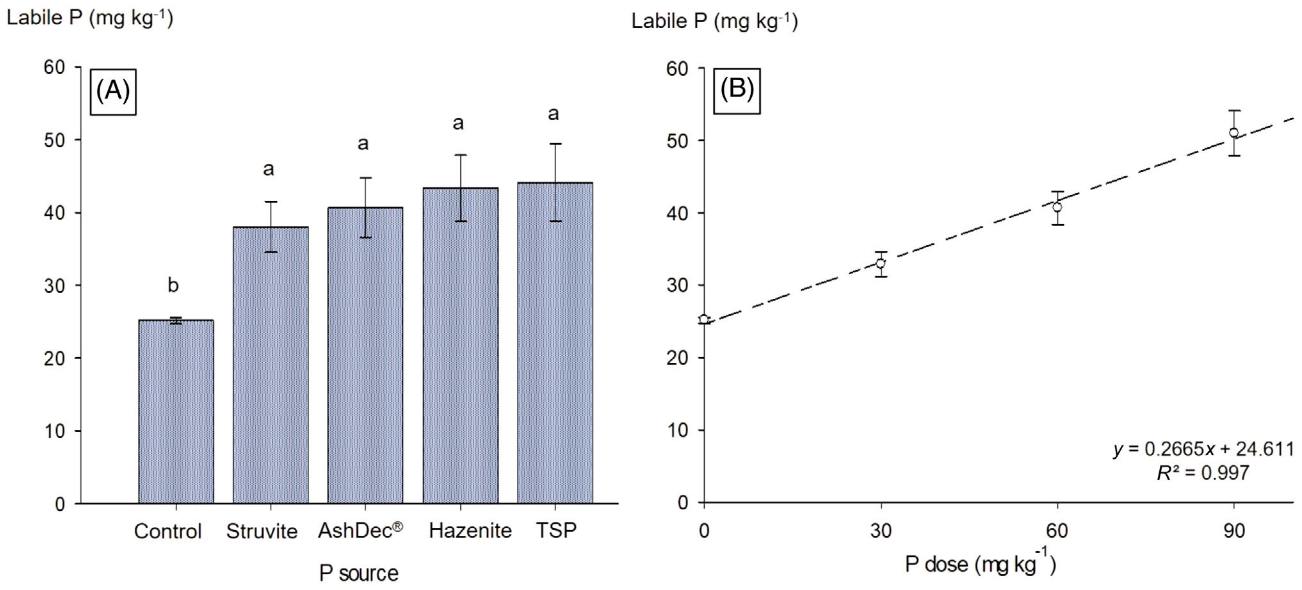

FIGURE 4 Soil labile P (mg kg^{-1}) as affected by P source (A) and dose (B) at 180 days after establishment (*n* = 4). Means followed by the same letter do not differ by 5% error probability in Tukey test. Error bars represent standard error

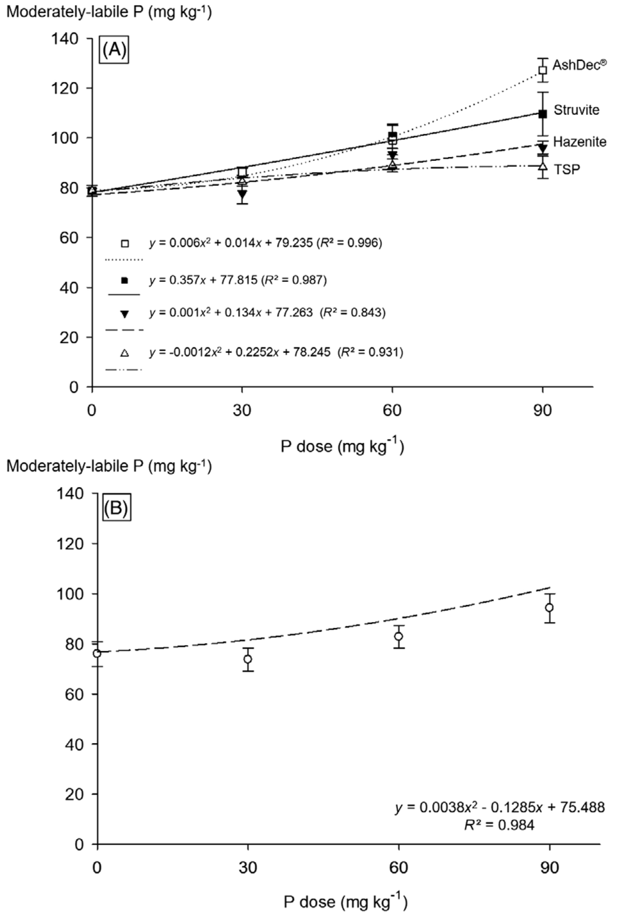

FIGURE 5 Soil moderately labile P (mg kg^{-1}) as affected by the interaction source × dose at 90 days (A) and by dose only at 180 days (B) after establishment (*n* = 4). Error bars represent standard error

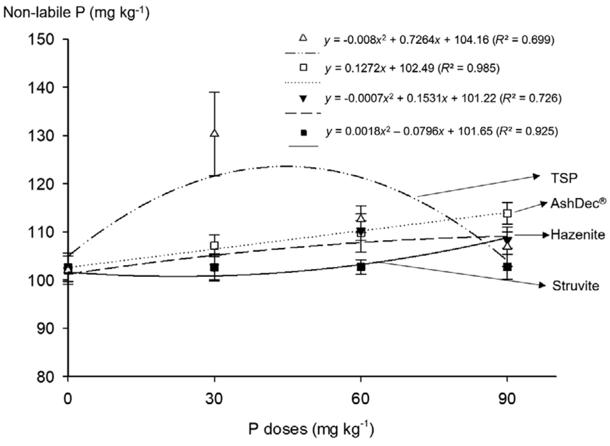

FIGURE 6 Soil non-labile P (mg kg^{-1}) at 180 days after establishment under phosphate sources and doses (*n* = 4). Error bars represent standard error

4.4 | Soil P pools

4.4.1 | Labile P pool

Good performances in terms of P bioavailability by recycled P sources were also found by Vogel et al. (2017) for soils cultivated with spring barley. As we do here, they also reported a positive linear correlation between P doses, sources (struvite, TSP, and two types of SSA), and soil labile P. In their study, however, significantly higher values of soil labile P were found for struvite in comparison to a Ca-SSA treatment, which was not the case for our AshDec® fertilizer.

Although the labile P is readily available for plant uptake, if not rapidly absorbed it will be adsorbed to the soil with time due to soil–fertilizer interactions, especially in soils with high Fe/Al oxi(hydroxides) content, ultimately decreasing P availability (Bortoluzzi et al., 2015; Fink, Inda, Tiecher, et al., 2016). This happens with the "aging" and the increase in the adsorption energy of the P bonds between these oxides, which ultimately ends up turning labile into moderately labile and those into nonlabile P if no contrary pressure is imposed upon the system, such as rhizosphere and microorganism's activity (Wang et al., 2013; Yoon et al., 2014).

The higher soil labile P values observed for the struvite compared to the AshDec® could be explained by the fact that the former presents (although very small) some solubility in water, and may be dissolved irrespective of plant activity after a certain time depending on environmental conditions, different from the AshDec®, which is completely insoluble in water as reported in a previous study with the same sources (Raniro et al., 2022). This could have given struvite-treated plants a head start that allowed faster root development, plant establishment, and further increase rhizosphere activity, ultimately resulting in higher yield.

4.4.2 | Moderately labile P pool

Higher values observed under AshDec® at 90 days might be explainable by its original material composition. SSA contains low P bioavailability depending on its characteristics (Smol et al., 2020). The AshDec® thermochemical process aims to increase the bioavailability of these P compounds by formation of $CaNaPO_4$ (Hermann & Schaaf, 2019). However, some phosphates present in SSA may remain in moderately labile forms even after thermochemical treatment, such as Ca-P, and thus the addition of a not fully reacted thermochemical product to the soil could impact this P pool.

Different authors have pointed out that even relatively low solubility P compounds can be mobilized in the soil through crop and microorganism's activity (Dakora & Phillips, 2002; Eichler-Löbermann et al., 2007; Requejo & Eichler-Löbermann, 2014), which can explain this moderately labile P reduction between crop cycles. This means that it managed to find its way into more labile fractions, possibly due to rhizosphere effect, which reinforces the proposition that this fertilizer is particularly affected by plant activity (Hermann & Schaaf, 2019).

4.4.3 | Non-labile P pool

Our P fractionation results showed that most of the soil P was in non-plant-available forms for every treatment. This is possibly due to the non-labile P being more related to the soil characteristics than fertilizer sources and/or doses (Gatiboni et al., 2007), and may be altered in long-term management. An increase in nonlabile P was observed at 180 days compared to 90 days for most doses under TSP.

In highly weathered and acidic tropical soils, phosphorus fixation is a major concern since P can interact with clay minerals (mostly kaolinite) and Fe/Al oxy(hydroxides) (goethite and gibbsite), generating strong bonds that hamper dissolution and reduce plant availability (Gérard, 2016; Hamid & Ahmad, 2012). The significant participation of Fe and Al components in the clay fraction in the soil here tested could have contributed significantly to adsorbing the readily available TSP-derived phosphates, reducing its efficiency in this soil.

At first, only a small percentage of the fertilizer bonded to the soil; however, as time progresses, more fertilizer-derived P became bonded with high energy, thus increasing the correlation between nonlabile P and the treatments. Along this line, it is reasonable to assume that the longer these distinct treatments are observed, the higher the correlation between them and the nonlabile P would become, due to P-binding reactions taking place in the soil.

Analyzing the bioavailability of soil P forms for successions of different crops, Gatiboni et al. (2007) observed that while the residual P did not suffer major alterations until the nineth cycle, great depletion was observed between the 12th and 15th cycles for different species. This shows that in situations of high P scarcity, all P fractions may solubilize to the soil solution, corroborating to our findings. Although treatment effect on the non-labile P pool was very subtle, it was possible to observe a pattern of reduction of this fraction between periods (Table S3), signalizing that some of these phosphates were released to the moderately labile fraction. The only exception was TSP, which showed a slight increase in non-labile P at 180 days when compared to the 90 days values. This was expected for TSP once its high water solubility and asynchrony with plants demand could ultimately cause P adsorption to the soil colloids, possibly becoming less available overtime.

The previously reported results demonstrate how the P sources studied hazenite and AshDec® can be beneficial for increasing P fertilization efficiency and sugarcane P supply, like it has been shown for other crops under struvite in distinct soils by multiple authors (Gell et al., 2011; Katanda et al., 2016; Rech et al., 2019). This is crucial for the understanding of such sources and their agronomic potential as P fertilizers for sugarcane production in tropical conditions, and serve as a starting point for the development of new research, potentially in field scenarios, aiming to disclose more sustainable options for P fertilization, ultimately contributing to waste recycling, smart use of resources, safety of agroecosystems, and crop production in tropical areas.

5 | CONCLUSIONS

Overall, all three recycled sources performed better or at least comparably to TSP. Struvite and hazenite allowed higher DMY by sugarcane than TSP and AshDec® at 90 days, which performed similarly. Struvite outperformed all sources at 180 days. Struvite and hazenite also performed significantly better than TSP and AshDec® in accumulated shoot P at 90 days, but only AshDec® was significantly worse than Struvite at 180 days.

Soils under struvite and AshDec® had more labile P after the first 90 days when compared to TSP and hazenite. No differences between fertilizers were found in the end of both cycles (180 days) in soil labile P, showing that the fertilizer dynamics influence diminished over time. AshDec® was the only fertilizer that impacted the moderately labile soil P fraction at 90 days, which matches the fertilizer late release characteristics. However, due to its low initial P release, AshDec® did not allow good plant initial development at the lowest dose, and therefore did not perform as well as the other recycled sources in terms of fertilizer efficiency.

Fertilizer source and dose interaction affected the non-labile P pool after 180 days, being significantly increased under TSP. This compartment presented an overall reduction between the first and the second harvest, meaning that in certain scenarios it might participate into increasing soil moderately labile and posteriorly labile P. Pot size and trial duration may have limited growth and development of plants, thus masking certain differences between fertilizers.

This study enlightened struvite, hazenite, and AshDec®'s potential to perform better or at least comparatively to TSP for sugarcane P supply and DMY production in a tropical soil, and future field trials could significantly add to the discussion and understanding of these sources. Likewise, microbiological and enzyme activity analyses could have a significant impact in P solubilization and may also contribute to the understanding of the mechanisms and conditions under which struvite, hazenite, and AshDec® excel.

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DATA AVAILABILITY STATEMENT

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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