



# Soil phosphorus forms and fertilizer use efficiency are affected by tillage and soil acidity management

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## ABSTRACT

Liming combined with no-tillage (NT) can increase the efficiency of P use by accumulating soil P in more available forms and improving root exploration due to the absence of toxic  $Al^{3+}$ . This study aimed to evaluate the long-term effect of different combinations of soil tillage managements and liming in a subtropical Oxisol on the accumulation of P forms, legacy P index, and P use efficiency. A long-term experiment was used, established in 1987 testing (i) conventional tillage with lime (lime-CT) and control (control-CT), (ii) NT with lime (lime-NT) and control (control-NT), and (iii) occasional tillage with lime (lime-OT). The NPK fertilization was the same for all treatments. The accumulated grain yield and P use efficiency of 56 crops between 1987 and 2019 were evaluated. In 2019, soil samples were collected in the 0–5, 5–10, and 10–20 cm layers and submitted to a sequential chemical fractionation of P, P indexes, and soil acidity parameters. The results reveal that is necessary to correct acidity to increase crop yields and P use efficiency, regardless of the soil management used. This increase is enhanced when soil acidity correction by liming is combined with the use of conservation systems such as NT. Furthermore, after liming with incorporation, liming can be done on the soil surface, without the need to repeat the operation of lime incorporation, ensuring high efficiency of P use even after more than four decades.

## 1. Introduction

Phosphorus (P) is one of the nutrients that regulate global agricultural production (Rodrigues et al., 2016; Du et al., 2020; Oliveira et al., 2022). Although the natural content of total P in soils can reach values as high as 1800 mg kg<sup>-1</sup> in tropical and subtropical soils (Pavinato et al., 2021), only a very small amount of this P is readily available for plants due to strong P adsorption of 1:1 clay minerals (e.g., kaolinite) and iron (Fe) and aluminum (Al) oxides (Fink et al. 2016). In these soils, the only way to meet the crop's demand is with the addition of phosphate fertilizers (Mumbach et al., 2021).

The global shortage of P fertilizers has already gained considerable attention on the international agenda in recent years, in part due to the 800% price increase in phosphate rock in 2008 (Gilbert, 2009) and more

recently in 2021–2022 due to the combined effect of several factors, in particular, conflicts between fertilizer producing countries and high energy demand, affecting the fertilizer supply chain and threatening the global food supplies (Lang and McKee, 2022). Actual estimates of phosphate depletion or peak phosphate fertilizer production vary widely in the literature (El Bamiki et al., 2021), but following the 2008 price spike, there has been a growing awareness of P shortages, which together with the environmental impact of P pollution from overuse of the nutrient, have spurred research around the world to better explore how P is used by plants and lost from agroecosystems (Osmond et al., 2019; Assis et al., 2022; Mubvumba and DeLaune, 2023).

In a scenario of an imminent shortage of phosphate fertilizer reserves, the estimation of the legacy of P – the amount of P accumulated in the soil – has received special attention in recent years (Doydora et al.,

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2020; Gatiboni and Condron, 2021). Analyzing possible future scenarios for P management in agriculture, Withers et al. (2018) concluded that the most appropriate alternative to better use environmental resources and, at the same time, maintain or increase food production would be to take advantage of the “P legacy” in the soil. In Brazil, the legacy of P in the soil is estimated to reach 33.4 Tg due to the residual P resulting from the excessive application as compared to crop export over the years (Pavinato et al., 2020). However, to better use P reserves in the soil, management strategies are needed that allow the plants access to the P present in the soil (Gatiboni et al., 2021a; Pavinato et al., 2022). In this case, proper soil management through soil acidity correction and conservation systems without soil disturbance can increase the efficiency of nutrient use, both P from fertilizers and P accumulated in the soil, and result in phosphate fertilizer savings on a global scale (Pavinato et al., 2020).

Most subtropical soils are very acidic and have high levels of exchangeable aluminum (Al), which is toxic to most crops (Rabel et al., 2018). Neutralization of soil acidity and elimination of toxic  $Al^{3+}$  is essential to increase the efficiency of P fertilizers, which is present in low concentration in the soil solution and supplied to plants by diffusion (Morel et al., 2021). Due to its low mobility in the soil, it is necessary to increase the plant root development to facilitate access to P. Liming benefits root growth by eliminating toxic  $Al^{3+}$  when soil pH in water is higher than 5.5 and by increasing the saturation of Ca and Mg in the soil (Alves et al., 2021; Bossolani et al., 2022). This practice is strategic and cannot be a limiting factor for countries like Brazil, which has an abundance of limestone ore throughout its territory (Ruiz et al., 2020). Conversely, Brazil has only low solubility phosphate rock reserves, therefore, depending largely on imported phosphate fertilizers supply.

In just a few months of reaction, liming with incorporation into the soil neutralizes the acidity in the topsoil, up to 20 cm deep, where most of the roots are concentrated (Miotto et al., 2019). However, soil acidity neutralization in the no-tillage system (NT) with the surface lime application is more challenging, as the lime reaction is usually restricted to the topsoil (~10 cm), and due to its low solubility, the neutralization of soil acidity in deeper layers is slow and less likely (Rheinheimer et al., 2018). The problem of subsurface acidity in no-till has been frequently reported in Brazil and Paraguay (Pias et al., 2020), South Africa (Liebenberg et al., 2020), and Australia (Li et al., 2019). For this reason, some authors recommend the occasional tillage of the soil to neutralize the acidity in deeper soil layers and eliminate compacted soil layers (Peixoto et al., 2020; Bellinaso et al., 2021).

In long-term NT, P accumulation also occurs in the topsoil, forming a strong gradient of P availability in depth (Tiecher et al., 2017). The accumulation of P in the first 5-to-10 cm of soil can compromise the absorption by plants, especially in situations of water deficit. The first centimeters of soil are the first to dry out due to the natural drainage and the evaporation process and, as a consequence, there is limited diffusion or plant uptake of P accumulated on the surface. The NT system also results in a greater amount of organic residues deposited on the soil surface, accumulating P and organic carbon on the soil surface. Many studies report that there may be competition for P sorption sites due to the greater amount of organic anions in the soil surface soil under NT (Zamuner et al., 2008; Fink et al., 2016) but this relationship is still controversial, and the increase in organic carbon in NT compared to conventional tillage (CT) does not always affect the maximum soil P adsorption capacity, although it can increase the P desorption (Fink et al., 2014).

In CT, the frequent soil tillage before each crop results in a uniform soil profile that, combined with liming, results in a better environment for root exploration in the plowing layer, with soil acidity correction and redistribution of nutrient contents in depth. However, it compromises the stability of aggregates and the structure of the soil, reduces the retention and availability of water, determines the partial loss of organic matter, and incorporates the straw present on the surface into the soil, in addition to exposing the phosphate to new sorption sites. In contrast,

long-term NT promotes an increase in soil organic matter and available P in the surface layers, but it can also result in a gradient of soil acidity and P availability in depth that is not ideal for crop development. Thus, an alternative would be the strategic or occasional tillage (OT) of the soil when necessary. An indicator for decision-making of this operation would be the need for liming or the occurrence of compaction, which usually occurs within a few years, depending on the cropping system and soil type. This operation can be advantageous by combining the positive effects of both soil tillage systems (NT and CT). However, information on this soil management is scarce, especially regarding its effects on P dynamics in the soil.

Therefore, our hypothesis is that the use of conservation systems associated with liming can increase the P use efficiency due to the accumulation of soil P in more available forms and better root exploration in the absence of toxic  $Al^{3+}$ . The objective of this study was to evaluate the long-term effect of different combinations of soil tillage and liming in a subtropical Oxisol on soil P forms, P use efficiency, and P legacy index. The study was based on a 32-year-old field experiment with historic crop yields and fertilization under conventional tillage, no-tillage, and occasional tillage, where plowing and disking were performed after each liming operation (five times in 32 years).

## 2. Material and methods

### 2.1. Site description and history of the experimental area

The experiment, installed in 1978, is located on an Oxisol at the Agrária Foundation for Agricultural Research (FAPA), of the Cooperativa Agrária Mista Entre Rios Ltda, in Guarapuava, Paraná state, southern Brazil (25°33'02" S, 51°29'24" W). The relief is gently sloped and the altitude is 1100 m above sea level. The climate is Cfb type according to the Köppen–Geiger classification (Alvares et al., 2014). The average annual rainfall from 1976 to 2022 was 1,886 mm per year. The monthly average of precipitation, average, maximum, and minimum temperature from 1976 to 2022 is presented in Fig. 1.

Before the experiment, the area was covered by native Atlantic Forest, which was cut and cleared in the 1920s. After that, spontaneous-grown pasture, mainly grasses, was utilized for grazing until the 1950s, when the pasture was plowed and replaced by wheat (*Triticum aestivum*) and rice (*Oryza sativa*) for a few years. Then, between 1962 and 1968, white clover and winter grasses were introduced as pasture for dairy cattle, and from 1968 to 1977 wheat and soybeans (*Glycine max*) were cultivated in the conventional tillage (CT) system.

When the experiment was set up in 1977, the soil in the 0–20 cm layer had a water pH of 5.5, 28 g kg<sup>-1</sup> of total organic carbon, 0.15 cmol<sub>c</sub> dm<sup>-3</sup> of exchangeable Al, 5.7 cmol<sub>c</sub> dm<sup>-3</sup> of exchangeable Ca + Mg, 0.25 cmol<sub>c</sub> dm<sup>-3</sup> of available K by Mehlich-1 solution, 5.9 mg dm<sup>-3</sup> of available P by Mehlich-1 solution, and 2.5 % of Al saturation. In this same soil layer, the clay, silt, and sand content were 570, 370, and 60 g kg<sup>-1</sup>, respectively (Jaster et al., 1993). The main minerals found in the clay fraction are kaolinite, iron oxides (goethite and hematite), and aluminum oxides (gibbsite). The content of Fe extracted with ammonium oxalate, related to Fe oxides with low crystallinity, is 1.43 g kg<sup>-1</sup>. For the same layer, the Fe content extracted with dithionite-citrate-bicarbonate, related to the pedogenic Fe oxides, is 80.8 g kg<sup>-1</sup>. The hematite/(hematite + goethite) ratio is 0.30, and the gibbsite/(gibbsite + kaolinite) ratio is 0.74.

### 2.2. Experimental design

The present experiment is divided into two phases, from 1978 to 1987, and from 1987 to present. In the first phase, from 1978 to 1987, the experiment consisted of only two soil management systems, namely: (i) conventional tillage with plowing and two disking in winter and summer, and (ii) no-tillage in winter and summer, where the sowing of the crops is performed without soil tillage. Before the implementation of

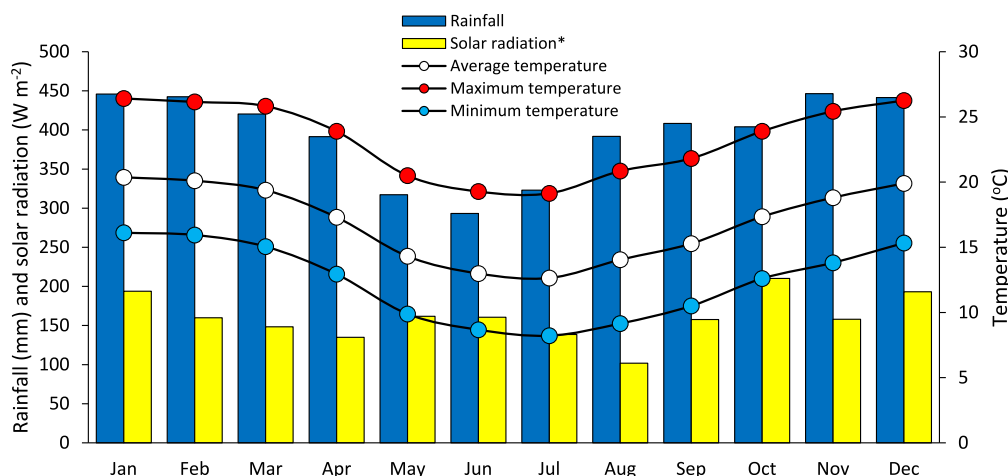


Fig. 1. Monthly average temperature and rainfall in the experimental area in the period from 1976 to 2022. \*Solar radiation data are from the 2002–2022 period.

the experiment in the summer of 1978, 1.5 Mg ha<sup>-1</sup> of limestone and 300 kg ha<sup>-1</sup> of a byproduct generated during iron ore processing, composed mainly of calcium silicophosphate (mixture of Ca<sub>3</sub>(PO<sub>4</sub>)<sub>2</sub> and CaSiO<sub>3</sub> - Thomas slag) were applied in the area in the winter of 1977.

In the second phase of the experiment (after 1987), the lime factor was inserted in the experiment. After that, the conventional tillage was conducted (1) without lime and (2) with lime incorporation in the soil by plowing and disking. No-tillage had the following liming treatments: (3) without lime, (4) surface liming, and (5) occasional tillage for lime incorporation in the soil, which was performed five times in 32 years. In the second phase, the experimental design was a completely randomized block and split-plots, where tillage was in main plots (20 × 100 m) and lime in sub-plots (20 × 33 m). The dolomitic lime rates used based on Effective Neutralizing Power (ENP) of 100% were 4.5, 3.0, 4.0, 4.7, and 4.7 Mg ha<sup>-1</sup> respectively in 1987, 1995, 2001, 2007, and 2018, totaling 20.9 Mg ha<sup>-1</sup>.

### 2.3. Fertilization history and cumulative crop yields

The same crop rotation was carried out in all treatments, with wheat (8 seasons), barley (*Hordeum vulgare*, 8 seasons), and black oats (*Avena sativa*, 7 seasons) as commercial crops in winter, and radish (*Raphanus sativus*, 4 seasons) and vetch (*Vicia sativa*, 1 season) and a consortium of both species (3 seasons) as cover crops preceding corn. In summer, soybeans were grown in most seasons (24), in rotation with corn (*Zea mays*, 8 seasons). The sequence of winter and summer crops in crop rotation from 1987 to 2019 and the N, P, and K applied in each crop and the cumulative amount is presented in Table 1. The fertilizers used were N-P-K dry commercial formulas, containing urea as a N source, single or triple superphosphate as a P source, and potassium chloride as a K source. These fertilizers had range of different percentage of N-P<sub>2</sub>O<sub>5</sub>-K<sub>2</sub>O concentration, such as 00-20-20, 00-25-25, 00-28-20, 05-25-25, 08-28-20, 08-30-20, 09-25-24, 10-20-20, and 10-26-24. In the topdressing fertilization with N, urea was used as a source of N. The rates of N, P, and K added in each cultivation are listed in Table 1. The rates of N, P, and K were defined according to regional recommendations, which take into account not only the crop but also an annual adjustment based on yield expectation and previous soil analysis (Fontoura et al., 2015). The average rate of P applied was 38 ± 12 kg ha<sup>-1</sup> for barley, 39 ± 6 kg ha<sup>-1</sup> for corn, 28 ± 5 kg ha<sup>-1</sup> for wheat, 23 ± 5 kg ha<sup>-1</sup> for white oats, and 18 ± 7 kg ha<sup>-1</sup> for soybean (Table 1).

Cumulative grain crop yields in the whole period were calculated taking into account crop yields of each winter and summer season. The annual variation of crop yield by treatment and by the crop is not part of the objective of the present work and, therefore, only the cumulative crop yields for the different combinations of tillage and lime treatments

were considered. The no-tillage effect on crop yield was calculated as the increase in crop yield comparing the NT and CT treatments (Eq. 1), and the lime effect on crop yield was calculated as the increase in crop yield comparing the limed and the control treatments (Eq. 2).

$$\text{Notillage effect on crop yield} = \left[ \left( \frac{(\text{Control NT yield}) - (\text{Control CT yield})}{\text{Control CT yield}} \times 100 \right) + \left( \frac{(\text{Lime NT yield}) - (\text{Lime CT yield})}{\text{Lime CT yield}} \times 100 \right) \right] / 2 \quad (1)$$

$$\text{Lime effect on crop yield} = \left[ \left( \frac{(\text{Lime CT yield}) - (\text{Control CT yield})}{\text{Control CT yield}} \times 100 \right) + \left( \frac{(\text{Lime NT yield}) - (\text{Control NT yield})}{\text{Control NT yield}} \times 100 \right) \right] / 2 \quad (2)$$

The export of P via grains was estimated using the sum of the crop yield of each crop and the P content in the grains. For the crops of black oat, barley, and wheat, reference values obtained from a local database of the Agrária Foundation for Agricultural Research were used: 4.0 g kg<sup>-1</sup> of P for white oat, 3.0 g kg<sup>-1</sup> of P for barley, and 3.5 g kg<sup>-1</sup> of P for wheat. For corn, the average content of 2.2 g kg<sup>-1</sup> of P was used based on 36 observations from Sena (2010) and 41 observations from Duarte et al. (2019). For soybeans, the average content of 5.5 g kg<sup>-1</sup> of P obtained in a meta-analysis of nutrient content in soybeans in Brazil was used (Filippi et al., 2021).

From the accumulated grain yield data and P exports via grains, and the total P applied between 1987 and 2019, two nutrients use efficiency indexes commonly used as a long-term indicator of trends were calculated (Dobermann, 2007). The first is the Partial Factor Productivity (PFP), which estimates how productive this cropping system is compared to its nutrient input (Eq. 3).

$$\text{Partial Factor Productivity (kg kg}^{-1}\text{)} = \frac{\text{Cumulative grain yield (kg ha}^{-1}\text{)}}{\text{Cumulative rate of P applied (kg ha}^{-1}\text{)}} \quad (3)$$

The second was the Partial Phosphorus Balance (PPB) which estimates how much nutrient is being taken out of the system as compared to how much is applied (Eq. 4).

**Table 1**

History of winter and summer crops from 1987 to 2019 in the experimental area and amount of N, P and K applied in each crop.

Year	Crop	N (kg ha <sup>-1</sup> )	P (kg ha <sup>-1</sup> )	K (kg ha <sup>-1</sup> )
1987	Wheat	46	26	33
1987/88	Soybean	12	18	25
1988	Barley	53	46	58
1988/89	Soybean	12	18	25
1989	Wheat	46	26	33
1989/90	Soybean	12	18	25
1990	Barley	53	46	58
1990/91	Soybean	12	18	25
1991	Vetch	8	16	31
1991/92	Corn	32	39	63
1992	White oats	10	22	42
1992/93	Soybean	0	21	39
1993	Barley	14	31	58
1993/94	Soybean	0	21	39
1994	Wheat	46	26	33
1994/95	Soybean	0	21	39
1995	Radish	0	0	0
1995/96	Corn	91	39	50
1996	White oats	36	20	38
1996/97	Soybean	0	17	23
1997	Wheat	36	25	31
1997/98	Soybean	0	17	33
1998	Barley	43	26	33
1998/99	Soybean	0	22	42
1999	Radish	0	0	0
1999/00	Corn	170	33	42
2000	White oats	57	26	50
2000/01	Soybean	0	13	25
2001	Wheat	42	20	25
2001/02	Soybean	0	13	25
2002	Barley	53	16	31
2002/03	Soybean	0	14	26
2003	Radish	0	0	0
2003/04	Corn	188	33	63
2004	White oats	37	17	22
2004/05	Soybean	0	16	31
2005	Wheat	94	26	33
2005/06	Soybean	0	16	31
2006	Vetch + Radish	0	0	0
2006/07	Corn	206	35	67
2007	Barley	118	46	58
2007/08	Soybean	0	22	42
2008	White oats	46	26	33
2008/09	Soybean	0	26	50
2009	Wheat	65	33	42
2009/10	Soybean	0	26	50
2010	Vetch + Radish	0	0	0
2010/11	Corn	205	40	70
2011	Barley	68	46	58
2011/12	Soybean	0	26	50
2012	White oats	52	26	33
2012/13	Soybean	0	27	52
2013	Wheat	65	33	42
2013/14	Soybean	0	27	52
2014	Vetch + Radish	0	0	0
2014/15	Corn	210	45	80
2015	Barley	73	46	58
2015/16	Soybean	0	22	42
2016	White oats	18	22	40
2016/17	Soybean	0	0	0
2017	Wheat	74	35	64
2017/18	Soybean	0	0	0
2018	Radish	0	0	0
2018/19	Corn	214	50	63
Sub-totals	White oats (n = 7)	256 [36.6]	159 [22.7]	258 [36.9]
	Barley (n = 8)	475 [59.4]	302 [37.8]	415 [51.9]
	Wheat (n = 9)	514 [57.1]	250 [27.8]	337 [37.4]
	Vetch (n = 1)	8 [8.0]	16 [16.0]	31 [31.0]
	Radish (n = 4)	0 [0]	0 [0]	0 [0]
	Vetch + Radish (n = 3)	0 [0]	0 [0]	0 [0]
	Corn (n = 8)	1316 [164.5]	314 [39.8]	498 [62.3]

**Table 1 (continued)**

Year	Crop	N (kg ha <sup>-1</sup> )	P (kg ha <sup>-1</sup> )	K (kg ha <sup>-1</sup> )
	Soybean (n = 24)	48 [2.0]	442 [18.4]	792 [33.0]
	Winter crops	1252	727	1040
	Summer crops	1364	757	1289
Total	–	2616	1484	2330

\*Values in the brackets indicate the average rate of nutrients applied for each crop.

$$\text{Partial Phosphorus Balance (\%)} = \frac{\text{Cumulative P removed by grain (kg ha}^{-1}\text{)}}{\text{Cumulative of P applied (kg ha}^{-1}\text{)}} \times 100 \quad (4)$$

#### 2.4. Soil sampling and analysis

In May 2019, soil samples were collected at 0–5, 5–10 and 10–20 cm depth layers in two trenches per plot. The samples were air-dried, ground, and subsequently sieved through a 2 mm mesh and stored for further analysis. Soil pH was determined in water suspension (1:1, v/v). Exchangeable K, Ca, Mg and Al were extracted with NH<sub>4</sub>Cl 1 mol L<sup>-1</sup>. Then, Al saturation was calculated as the proportion of Al as compared to the sum of K, Ca, Mg and Al.

#### 2.5. Phosphorus fractionation

Soil P forms were evaluated using the sequential chemical fractionation proposed by [Gatiboni and Condron \(2021\)](#). This procedure is performed in three steps, to estimate the P sorption capacity, the total P content, and the P forms.

In the first set, 0.500 g of soil sample was placed in a 15 mL falcon tube. Then, 10 mL of a stock solution containing 125 mg L<sup>-1</sup> of P were added and, subsequently, stirred at 30 rpm in an 'endless' type shaker for 60 min. Afterward, the tubes were centrifuged for 15 min at 3600 g. Then, the supernatant was collected for determination of the remaining P in the sample using the [Murphy and Riley \(1962\)](#) method. From this, the remaining P index was calculated using Eq. 5.

$$\text{Remaining P (\%)} = \left( \frac{125 - P_{\text{remaining}}}{125} \right) \times 100 \quad (5)$$

The total P content was estimated in another aliquot of soil sample using the method proposed by [Olsen and Sommers \(1982\)](#). For this, 0.100 g of soil was added to a 50 mL digestion tube, plus 2 mL of sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) and 2 mL of 37% hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) in the presence of saturated MgCl<sub>2</sub>. Subsequently, the tubes were placed in the digester block at 350 °C for 120 min. After the digestion process, distilled water was added to complete the volume of 50 mL. An aliquot was taken from this extract to determine the P content using the [Murphy and Riley \(1962\)](#) method.

Finally, in the third set, the sequential fractionation of P was performed. For this, 0.500 g of soil was added to falcon tubes and subjected to different extractors in sequential order: 0.01 mol L<sup>-1</sup> CaCl<sub>2</sub> (soluble-P), Mehlich-3 (available-P), 0.5 mol L<sup>-1</sup> NaOH (NaOH-P) and 1.0 mol L<sup>-1</sup> HCl (HCl-P). In the Mehlich-3 extract, P, Fe, and Al were measured by ICP-OES (Inductively Coupled Plasma – Optical Emission Spectrometry). In the 0.5 mol L<sup>-1</sup> NaOH extract, the inorganic P was measured by the [Dick and Tabatabai \(1977\)](#) method, and the total P was measured after acid digestion of these extracts ([Brookes and Powlson, 1981](#)). Organic P was then obtained by the difference between total P and inorganic P. The determination of P in the other extracts was performed using the [Murphy and Riley \(1962\)](#) method.

From the data obtained in the chemical fractionation of P in the soil, the occluded P (occluded-P), the degree of P saturation (saturation-P), and the legacy P index (legacy-P index) were calculated. The occluded-P



was considered as the difference between the total P and the sum of the fractions of inorganic and organic P extracted by 0.01 mol L<sup>-1</sup> CaCl<sub>2</sub>, Mehlich-3, 0.5 mol L<sup>-1</sup> NaOH, and 1.0 mol L<sup>-1</sup> HCl (Eq. 6).

$$\text{Occluded-P (mg kg}^{-1}\text{)} = \text{total-P} - (\text{soluble-P} + \text{available-P} + \text{NaOH-Pi} + \text{NaOH-Po} + \text{HCl-P}) \quad (6)$$

The P saturation index (saturation-P) was calculated from the contents of P, Fe, and Al obtained by the extraction of Mehlich-3 (P<sub>M3</sub>, Fe<sub>M3</sub>, Al<sub>M3</sub>) (Mehlich, 1984) according to Eq. 7.

$$\text{Saturation-P (\%)} = \frac{P_{M3}}{0.5 \times (\text{Fe}_{M3} + \text{Al}_{M3})} \times 100 \quad (7)$$

The legacy-P index (Eq. 8) used in the present study was considered how many times the critical level for plants are accumulated in the soil in forms that are potentially available for plants, as suggested by (Gatiboni and Condron, 2021). This index differs from the legacy P (P stocks in kg ha<sup>-1</sup>), which is defined as P from past applications, which requires knowledge of P at the starting point, before the period of monitoring or study. The regional critical level is 8 mg dm<sup>-3</sup> of P extracted with Mehlich-1 solution (Fontoura et al., 2015). This value was then converted to the equivalent of P extracted with Mehlich-3 according to IPNI (2015), i.e., 23 mg dm<sup>-3</sup>. P contents extracted with Mehlich-3 were converted from unit of mass to unit of volume using the average density of the soil in the experimental area (1.00 ± 0.06 kg dm<sup>-3</sup>) (Silva, 2013).

$$\text{Legacy P index} = \frac{(\text{Soluble P} \times 1.0) + (\text{Available P} \times 1.0) + (\text{NaOH Pi} \times 0.5) + (\text{NaOH Po} \times 0.5) + (\text{HCl P} \times 0.5) + (\text{Occluded P} \times 0.1)}{\text{Mehlich 3 critical level of P}} \quad (8)$$

To calculate the legacy P index in this study, we considered the lability of Hedley's P fractions based on the findings from Gatiboni et al. (2007). Studying an Oxisol from southern Brazil similar to our experimental site, they measured the depletion of P forms after up to 15 successive crops and found the fractions of soluble P and P extracted by Mehlich-3 as the most prone to plant uptake, and therefore, it was considered to be 100% available. The fractions of organic and inorganic P extracted by NaOH 0.5 mol L<sup>-1</sup> and the inorganic P extracted by HCl 1.0 mol L<sup>-1</sup> were considered moderately labile P, with 50% of potential availability. Finally, the occluded P was considered to be non-labile P, with only 10% of potential availability.

## 2.6. Phosphorus desorption

P desorption was evaluated by using successive extractions with Mehlich-1 extractor (0.05 mol L<sup>-1</sup> HCl + 0.0125 mol L<sup>-1</sup> H<sub>2</sub>SO<sub>4</sub>). This procedure consisted of conditioning 1.0 g of air-dried soil into Falcon tubes, in which 10 mL of Mehlich-1 extractant solution was added. The samples were shaken for five minutes on a horizontal shaker and then centrifuged for five minutes at 2500 rpm. Then, the P content in the supernatant was measured using Murphy and Riley (1962) method. The Mehlich-1 extraction was repeated successively until the extracted P content became constant and close to zero, which in this study occurred in the seventeenth extraction. The first extraction was considered as the readily available P, and the sum of the 17 extractions was considered the potentially available P.

## 2.7. Statistical analysis

For the analysis of variance (ANOVA) of yield and the indexes of

phosphorus efficiency use, the following model was used:  $Y_{ijk} = \mu + B_i + T_j + \text{error a (i, j)}$ ; where  $\mu$  = overall experimental average;  $B$  = blocks ( $i = 1, 2, 3$ );  $T$  = treatment ( $j = 1, 2, 3, 4, 5$ ); and  $\text{error}$  = experimental error. For the ANOVA of the soil data, the following model was used:  $Y_{ijk} = \mu + B_i + T_j + \text{error a (i, j)} + D_k + \text{error b (i, k)} + TD_{jk} + \text{error c (i, j, k)}$ ; where  $\mu$  = overall experimental average;  $B$  = blocks ( $i = 1, 2, 3$ );  $T$  = treatment ( $j = 1, 2, 3, 4, 5$ );  $D$  = depth ( $k = 1, 2, 3$ ) and  $\text{error}$  = experimental error. When treatment effects were significant at  $p < 0.05$  probability of error by the F test, the differences between means of treatments were compared by Scott-Knot ( $p < 0.05$ ).

## 3. Results

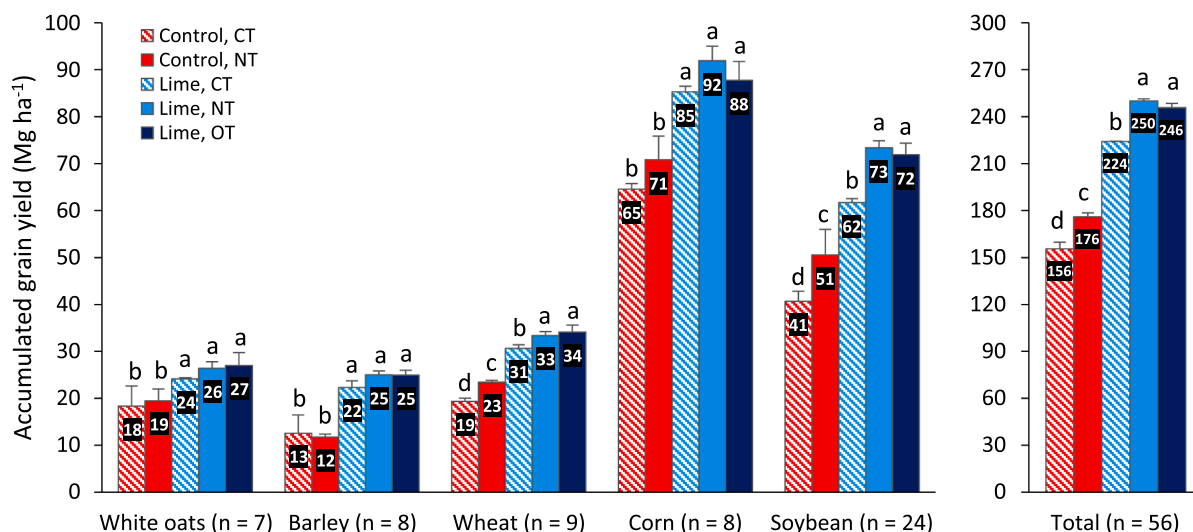
### 3.1. Long-term effect of lime management and tillage systems on grain yield and P efficiency use indexes

The grain yield and the P use efficiency indexes were evaluated (partial factor productivity – PFP, and partial phosphorus balance – PPB) considering all crop seasons from 1987 to 2019 were more affected by lime application than by soil tillage (Figs. 2 and 3). For white oat, corn, and barley, there was no effect of tillage systems on grain yield, PFP, and PPB. However, the lime application (lime-CT, lime-NT) increased about 31, 34 and 95% the grain yield of corn, white oats, and barley, respectively, when compared to the same tillage system management with no liming (control-CT, control-NT) (Fig. 4). These results are mainly due to the great difference in soil pH in water and Al saturation in the 0–20 cm

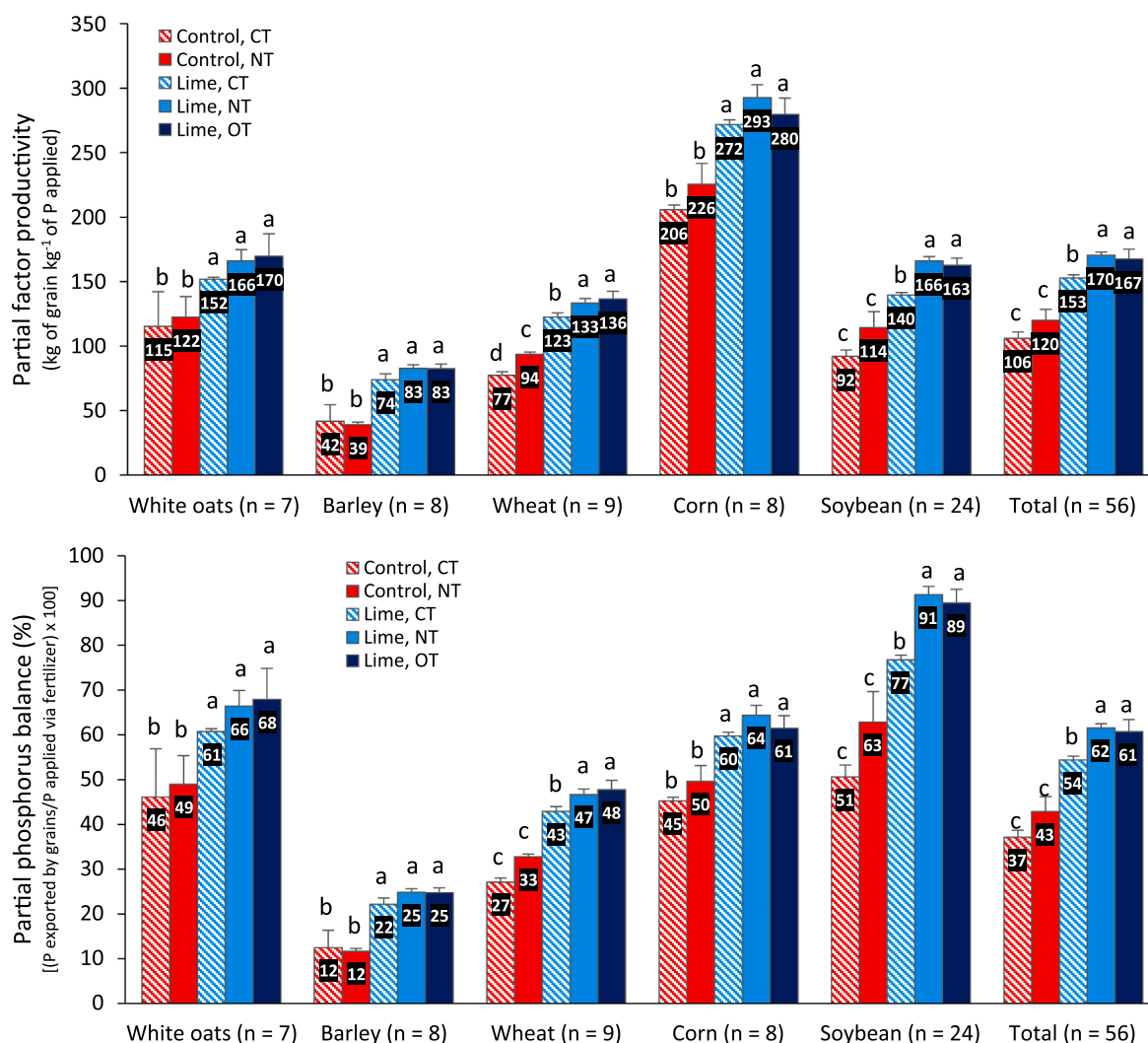
soil layer (Fig. 5). The treatments with lime application (lime-CT, lime-OT, lime-NT) presented on average a soil pH of  $5.4 \pm 0.2$ , almost one unit higher than the control treatments ( $4.5 \pm 0.2$  - control-CT, control-NT) (Fig. 5a). The Al saturation was on average  $61 \pm 5\%$  in the control treatments and only  $4 \pm 5\%$  in the limed treatments (Fig. 5b).

Lime also increased the grain yield of wheat, soybean, and the cumulative grain yield of all crops (Fig. 2). The yield of these crops was also affected by soil tillage management and decreased in the following order: lime-NT = lime-OT > lime-CT > control-NT > control CT (Fig. 2). The soybean, wheat, and the cumulative grain yield of all crops increased about 22, 15, and 12%, respectively, when using NT compared to the CT (Fig. 4). By contrast, the yield increase due to liming (lime-NT, lime-CT) was much higher and about 48, 53, and 43% for soybean, wheat, and the sum of all crops, respectively, as compared to the no limed treatments (control-NT, control-CT) (Fig. 4). The same trend of grain yield was observed for PFP in wheat (Fig. 3a). The PFP and PPB of soybean, the sum of all crops yield, and the PPB of wheat followed almost the same trend but with no difference between the no lime treatments (lime-NT = lime-OT > lime-CT > control-NT = control CT) (Fig. 3).

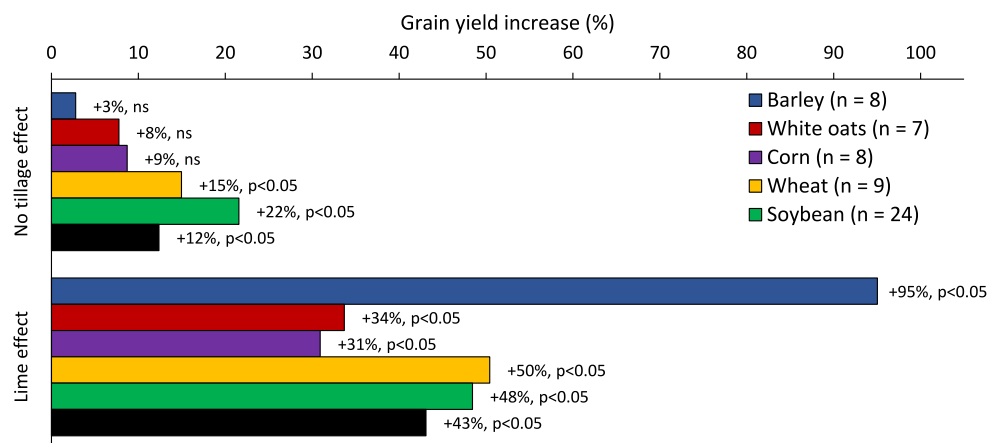
Grain yield per unit of applied P (PFP) varied greatly among crops and also between treatments with and without liming (Fig. 3a). The minimum and maximum values of PFP for each crop, in ascending order, were: 39–83 kg kg<sup>-1</sup> for barley, 77–136 kg kg<sup>-1</sup> for wheat, 92–166 kg kg<sup>-1</sup> for soybean, 115–170 kg kg<sup>-1</sup> for white oats, and 206–293 kg kg<sup>-1</sup> for corn (Fig. 3a). The same variation was observed for P recovery via grains (PPB), but the increasing order of crops was different from that observed for PFP: 12–25% for barley, 27–48% for wheat, 45–64% for corn, 46–68% for white oats, and 51–91% for soybean (Fig. 3b).



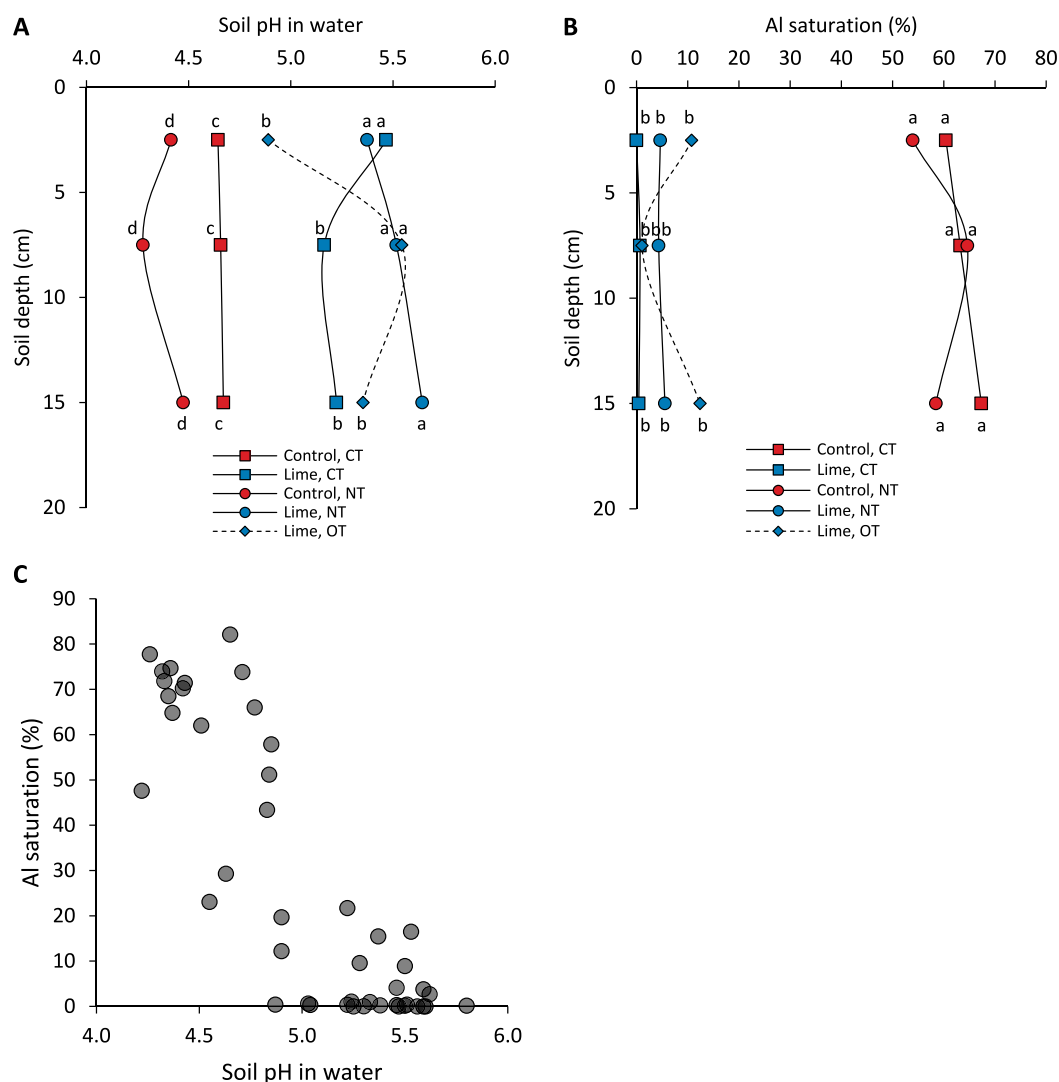
**Fig. 2.** Accumulated grain yield of the crops grown in different soil tillage managements (NT, no-tillage; CT, conventional tillage; OT, occasional tillage) combined or not with liming in a Brazilian subtropical Oxisol.



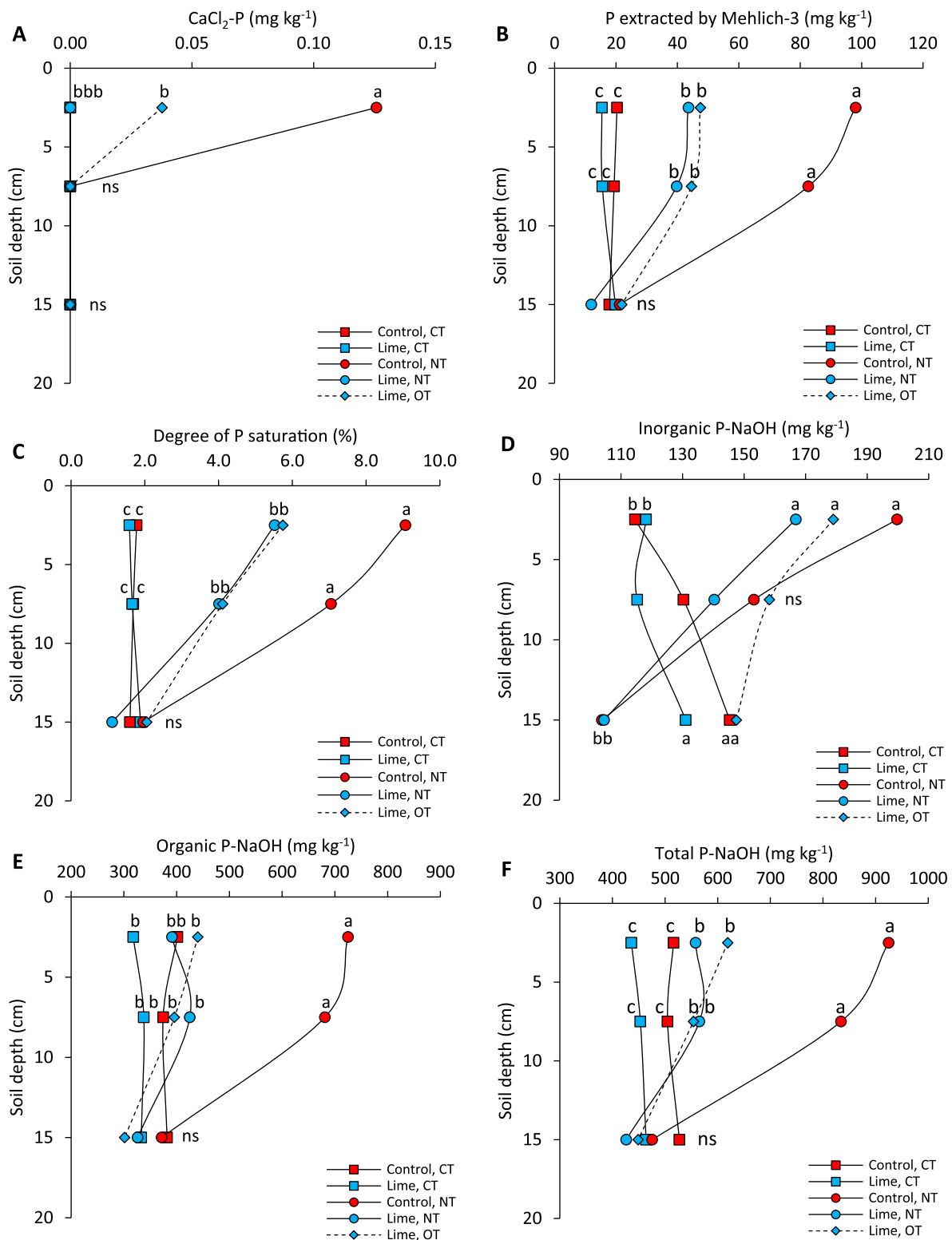
**Fig. 3.** Partial factor productivity (kg of grain produced per kg of P applied via fertilizer) (A) and partial phosphorus balance [(P exported by grains and P applied ratio) × 100] (B) of the crops grown in different soil tillage managements (NT, no-tillage; CT, conventional tillage; OT, occasional tillage) combined or not with liming in a Brazilian subtropical Oxisol.



**Fig. 4.** Average increase in the accumulated grain yield of white oats, barley, wheat, corn, soybean, and all crops combined (total) due to the use of soil conservation tillage and lime.



**Fig. 5.** Soil pH in water (A), Al saturation (B), and the relationship between soil pH in water and Al saturation (C) in the soil to a depth of 20 cm after 32 years of different soil tillage managements (NT, no-tillage; CT, conventional tillage; OT, occasional tillage) combined or not with liming in a Brazilian subtropical Oxisol. Means followed by the same letter are not statistically different by the Scott-Knot test at  $p < 0.05$ . ns, not significant.



**Fig. 6.** Soil P content extracted by  $\text{CaCl}_2$  0.01  $\text{mol L}^{-1}$  (A), P extracted by Mehlich-3 (B), degree of P saturation (C), inorganic P extracted by NaOH 0.5  $\text{mol L}^{-1}$  (D), organic P extracted by NaOH 0.5  $\text{mol L}^{-1}$  (E), total P extracted by NaOH 0.5  $\text{mol L}^{-1}$  (F), ratio of inorganic P-NaOH/Total-P (G), and ratio of organic P-NaOH/Total-P (H) up to 20 cm depth after 32 years of different soil tillage managements (NT, no-tillage; CT, conventional tillage; OT, occasional tillage) combined or not with liming in a Brazilian subtropical Oxisol. Means followed by the same letter are not statistically different by the Scott-Knot test at  $p < 0.05$ . ns, not significant.



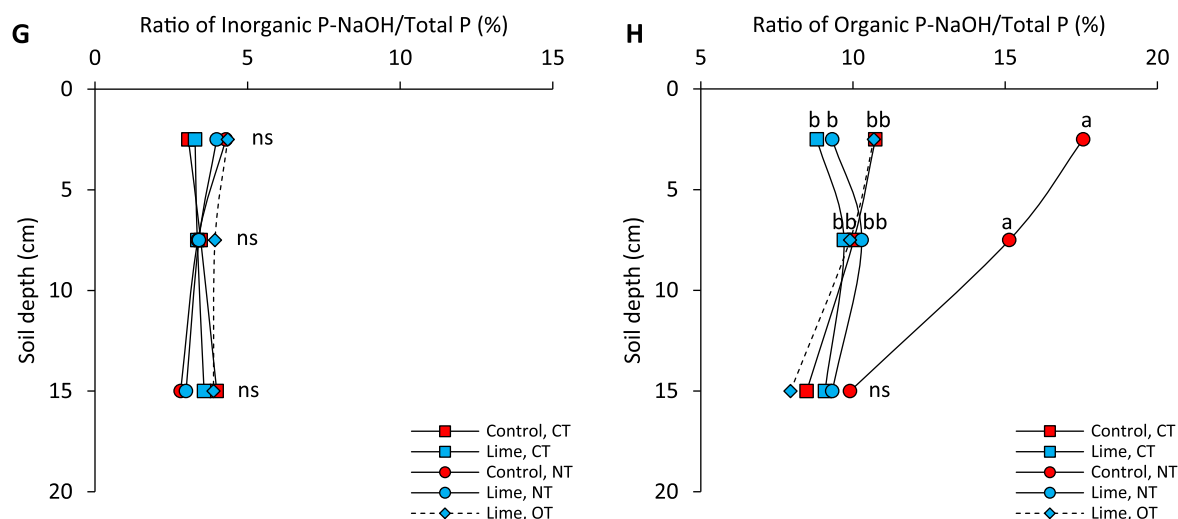


Fig. 6. (continued).

### 3.2. Effect of soil tillage on soil P forms

Unlike the crop's response, soil tillage management had a greater influence on P forms in the soil than liming (Figs. 6–8). In addition, the effect of treatments was more pronounced in the more superficial soil layers (0–5 and 5–10 cm). Below 10 cm depth, the P forms were very similar, except for the inorganic P content extracted with NaOH (Fig. 6d).

After 32 years of no-tillage system (NT), there was a higher content of P extracted with Mehlich-3 (Fig. 6b), P extracted with Mehlich-1 (Fig. 8a), total P extracted with 0.5 mol L<sup>-1</sup> NaOH (Fig. 6f), P extracted with HCl (Fig. 7a), occluded P (Fig. 7b) and total P (Fig. 7c) in the 0–10 cm soil layer as compared to CT. The degree of P saturation showed a similar trend to the P extracted with Mehlich-3 and was on average  $1.7 \pm 0.1\%$  and  $4.9 \pm 1.0\%$  in the 0–10 cm layer in the soil under CT and NT, respectively (Fig. 6c). The content of inorganic P extracted with HCl was about three times higher in NT compared to CT in the 0–10 cm layer (Fig. 7a). The inorganic P content extracted with 0.5 mol L<sup>-1</sup> NaOH was higher in NT only in the 0–5 cm layer compared to CT, and higher in CT in the 10–20 cm layer compared to NT (Fig. 6d).

Occasional tillage (OT) following lime application resulted in levels of P forms similar to NT with surface liming. The exceptions were for inorganic P extracted with NaOH, where OT had a higher content in the 10–20 cm layer compared to NT, being similar to that found in CT (Fig. 6d), and P extracted with HCl, where the P content in the 5–10 cm layer was lower than that observed in NT with liming (Fig. 7a).

In treatments under CT, there is little or no variation in P forms up to 20 cm deep (Figs. 6–8). By contrast, in the NT system, the P content extracted by Mehlich-3 (Fig. 6b), the inorganic P extracted with NaOH (Fig. 6d), P extracted with HCl (Fig. 7a), occluded P (Fig. 7b) and total P (Fig. 6c) decreased with increasing depth, indicating a concentration gradient from the soil surface. The occluded P content was the largest fraction of P in all treatments (on average  $85 \pm 3\%$ , Fig. 7b).

### 3.3. Effect of lime on soil P forms

In the CT system, the use of liming did not affect P forms (Figs. 6–8). By contrast, in the NT it is possible to verify some effects of liming on the content of P extracted with CaCl<sub>2</sub>, Mehlich-1, Mehlich-3, organic P extracted with NaOH, P extracted with HCl, and total P. In the 0–5 cm layer, the control-NT treatment showed the highest content of soluble P extracted with CaCl<sub>2</sub> ( $0.13 \text{ mg kg}^{-1}$  – Fig. 6a). In the other treatments and soil layers, the CaCl<sub>2</sub>-P content was null or very close to zero. In the 0–10 cm layer in control-NT, the content of P Mehlich-1 (Fig. 8a) and

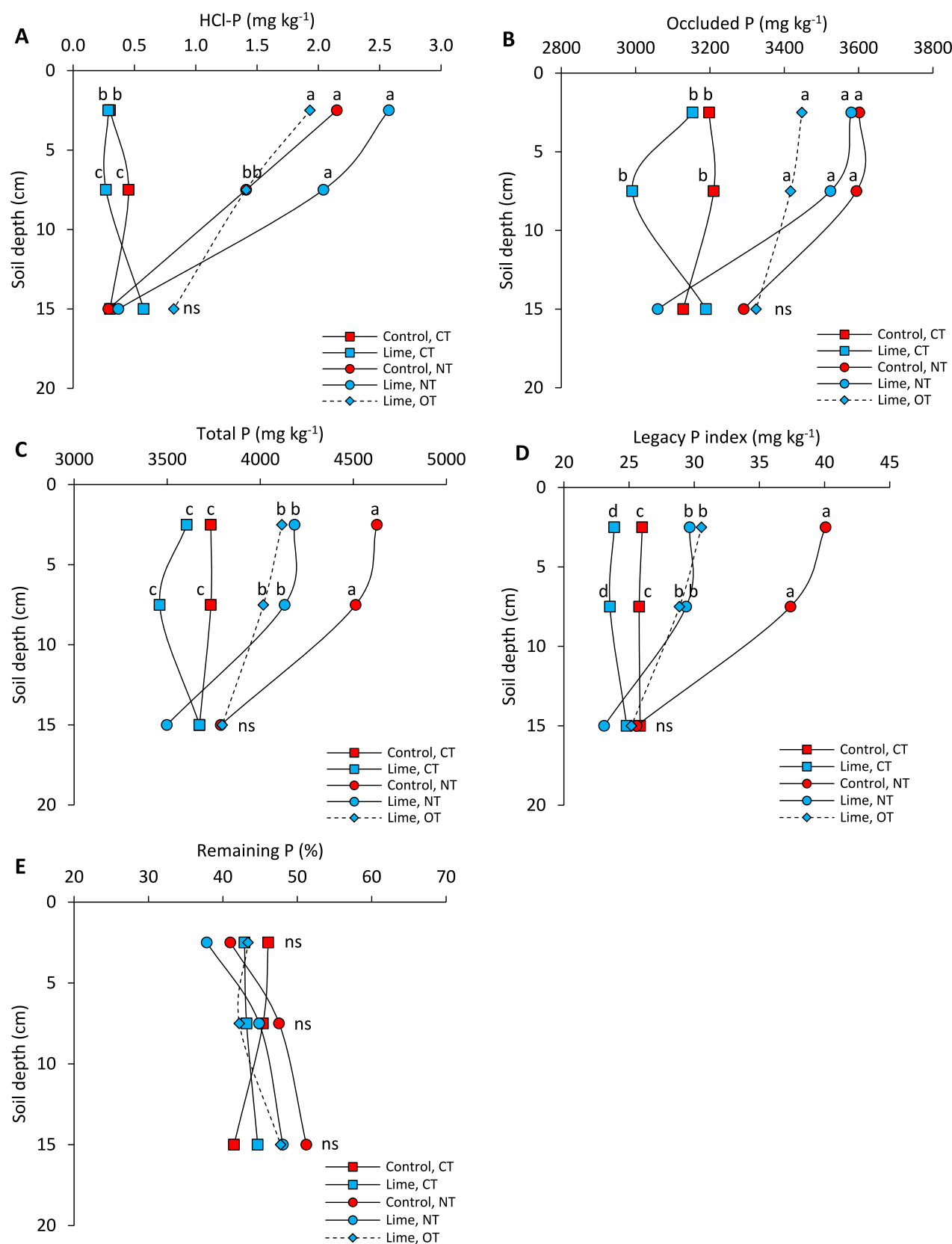
Mehlich-3 (Fig. 6b) was at least double that of the lime-NT treatment. In the 5–10 cm layer, an inverse behavior was observed for P extracted with HCl, with a higher content in lime-NT compared to control-NT (Fig. 7a). The organic P content extracted with NaOH was very similar in all treatments and soil layers evaluated ( $379 \pm 45 \text{ mg kg}^{-1}$ ), except in the control-NT treatment, which presented the highest organic P content in the 0–10 cm ( $703 \pm 31 \text{ mg kg}^{-1}$ ) (Fig. 6e). In the 0–10 cm layer, the control-NT, and lime-CT treatments showed the lowest total P content ( $3634 \text{ mg kg}^{-1}$ ), while NT-control showed the highest content ( $4570 \text{ mg kg}^{-1}$ ), and lime-NT showed intermediate levels ( $4158 \text{ mg kg}^{-1}$ ) (Fig. 7c).

Only the legacy P index was sensitive in differentiating the effect of liming on CT (Fig. 7d). The legacy P index in the 0–10 cm soil layer increased in the following order: lime-CT < control-CT < lime-NT = lime-OT < control-NT (Fig. 7d). The sum of P extracted by the 17 successive extractions using Mehlich-1 (Fig. 8b) followed the same trend of the first extraction (Fig. 8a). The control-NT treatment not only had the highest P content extracted with one (readily available P) and 17 Mehlich-1 (potentially available P) extractions but was also the treatment where the proportion between the readily available P and the potentially available P was higher (Fig. 8c). The same occurred for the proportion between potentially available P and total P, indicating that in this treatment P accumulation is occurring in more available forms compared to the others. The P desorption curves with Mehlich-1 demonstrate that there is an important desorption rate throughout the extractions, indicating that this is a high-buffered soil with a great capacity to supply P to plants, especially in the more superficial soil layers and in treatments without liming (Fig. 9).

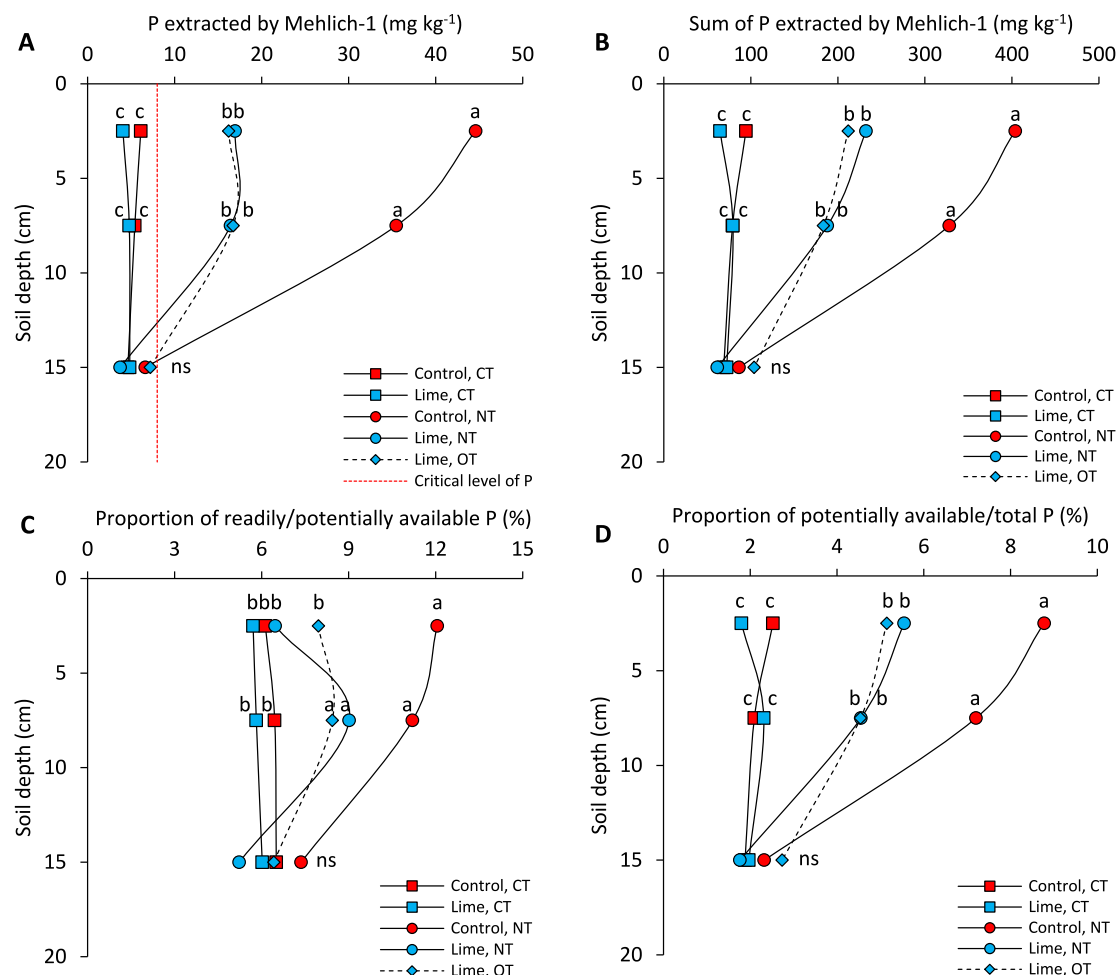
## 4. Discussion

The long-term results show a marked decrease in the P use efficiency for soybean, corn, and winter cereals when soil acidity is not corrected. Barley was the most positively impacted crop by liming, despite having the lowest P use efficiency (Fig. 3) and almost doubled the yield and P use efficiency in liming treatments compared to the control, while other crops (soybean, corn, wheat, and white oat) increased from 1.3 to 1.5 times (Figs. 2–4). These results agree with a global meta-analysis by Yu et al. (2021), who found that barley showed lower P use efficiency than corn, wheat, and rice in of cereal crops.

Our results demonstrate that grain yield (Fig. 3a) and recovery of applied P (Fig. 3b) can increase significantly when lime is used to maintain soil acidity at tolerable levels (soil pH =  $5.4 \pm 0.2$  and Al saturation =  $4 \pm 5\%$  in the 0–20 cm layer, Fig. 5). These results contrast



**Fig. 7.** Soil P content extracted by HCl 1.0 mol L<sup>-1</sup> (A), occluded P (B), total P (C), legacy P index (D), and remaining P (E) up to 20 cm depth after 32 years of different soil tillage managements (NT, no-tillage; CT, conventional tillage; OT, occasional tillage) combined or not with liming in a Brazilian subtropical Oxisol. Means followed by the same letter are not statistically different by the Scott-Knot test at  $p < 0.05$ . ns, not significant.



**Fig. 8.** Soil P content in a single extraction of Mehlich-1 (readily available P) (A), the sum of P extracted in 17 successive Mehlich-1 extractions (potentially available P) (B), the proportion of readily and potentially available P (C), the proportion of potentially available and total P (D) up to 20 cm depth after 32 years of different soil tillage managements (NT, no-tillage; CT, conventional tillage; OT, occasional tillage) combined or not with liming in a Brazilian subtropical Oxisol. Means followed by the same letter are not statistically different by the Scott-Knot test at  $p < 0.05$ . ns, not significant.

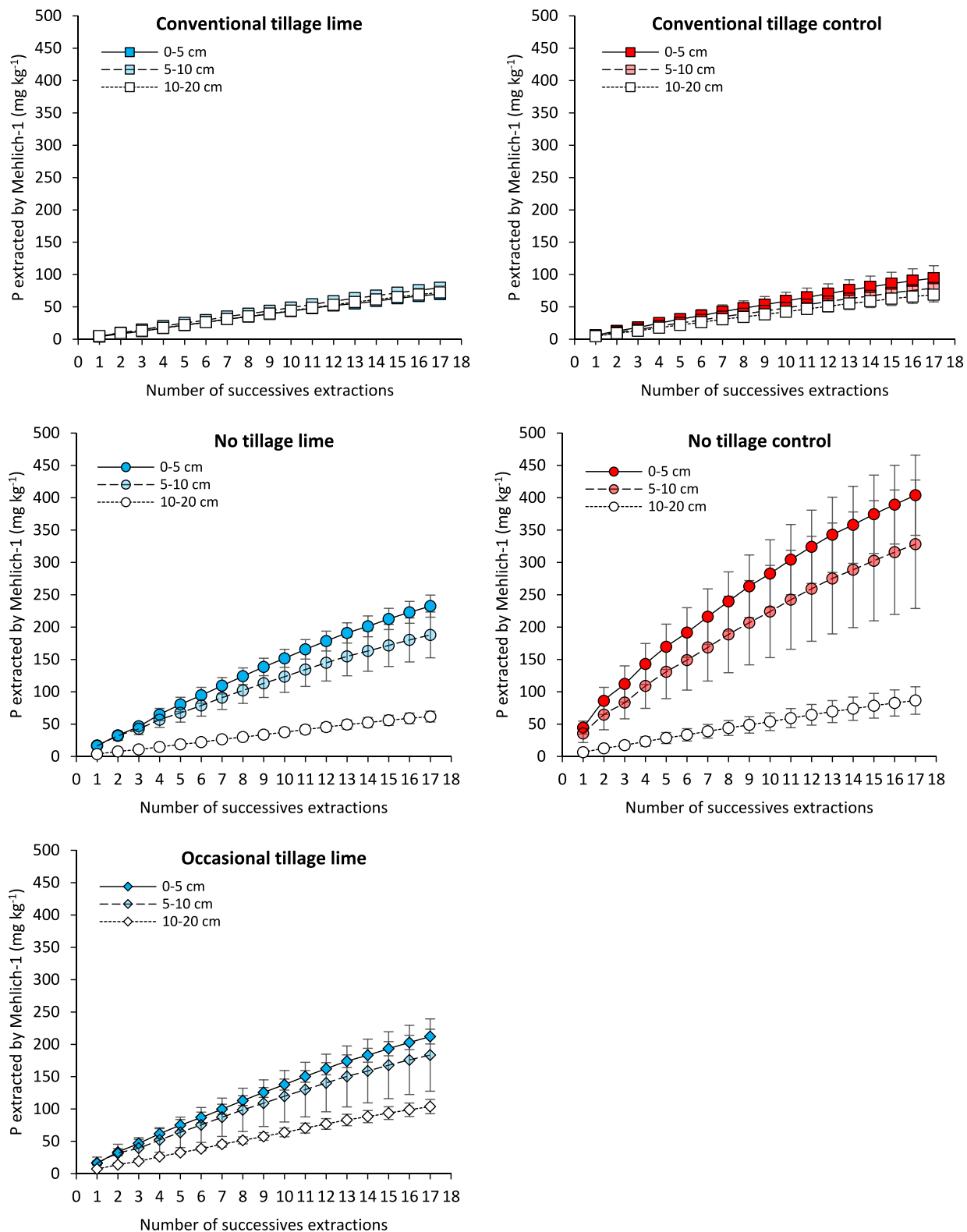
with those obtained by Yu et al. (2021). They found that the lowest P use efficiency was observed in the soil pH range close to neutrality (6.0–7.0) and that the efficiency increased as the pH decreased up to 4.5, because plants can lower pH of the rhizosphere by excreting protons or organic acids, thus increasing P uptake (Chen & Liao, 2016). In our study, however, the acidity condition in treatments without lime ( $\text{pH } 4.5 \pm 0.2$ , Fig. 5a) was associated with a high Al saturation ( $61 \pm 5\%$ , Fig. 5b). This may have hindered root development and, consequently, decreased P uptake by diffusion (Morel et al., 2021). In addition, the acidic pH conditions favor the adsorption of P by Fe oxides, decreasing its availability to plants.

The control-NT treatment had a 30% lower accumulated grain yield (Fig. 2), but the Mehlich-1 P in the 0–20 cm layer was three times higher than the critical level and twice that found in lime-NT treatment (Fig. 8a). Other indicators of a richer P environment in control-NT included the highest content of P extracted with  $\text{CaCl}_2$  and Mehlich-3 P, the highest degree of P saturation, the higher organic P-NaOH, and the higher total P content in the soil (Figs. 6 and 7). This suggests that there is no problem of P deficiency in the control-NT, but that plants are unable to access the nutrient due to higher soil acidity and  $\text{Al}^{3+}$  toxicity as foreseen by Barrow (2017). Therefore, using lime to increase P use efficiency, a nutrient mostly imported for Brazilian agriculture (Wang et al., 2022), is a strategy with great benefits. However, although limestone is an abundant ore in Brazilian territory (Ruiz et al., 2020), recent soil surveys show that 1/3 of the soil analyses in southern Brazil

still need acidity correction, with pH values below 5.5 (Tiecher et al., 2016).

Over the years, a lot of research has been carried out to increase P use efficiency using different types of fertilizers and application methods (Oliveira et al., 2022), P fertilization timing (Alves et al., 2022), P-solubilizing microorganisms (Bargaz et al., 2021), crop rotations (Rodrigues et al., 2021), among others. However, in acidic soils with the presence of toxic  $\text{Al}^{3+}$ , all these strategies will be limited by low root development and lower P absorption capacity (Barrow, 2017; Bellinaso et al., 2021). Neutralizing acidity and eliminating  $\text{Al}^{3+}$  toxicity should be the first step towards increasing P use efficiency, as it also benefits soil microorganisms and mineralization of organic residues and soil organic matter. The lower microbial activity in the control-NT treatment is likely the reason for the higher content of organic P extracted with NaOH compared to other treatments with liming (Fig. 6e). This observation is confirmed when comparing the organic and inorganic P content extracted with NaOH with the total P content (Fig. 6g and 6h). Even with an increase in the total P content (Fig. 7c) in the treatment control-NT, the proportion of organic P was higher than that found in other treatments (Fig. 6h). However, the higher organic P due to lower microbial activity in acid conditions was not observed in the treatment control-CT due to the frequent disturbance of the soil which ends up accelerating the mineralization of organic P (Tiecher et al., 2018; Rheinheimer et al., 2019).

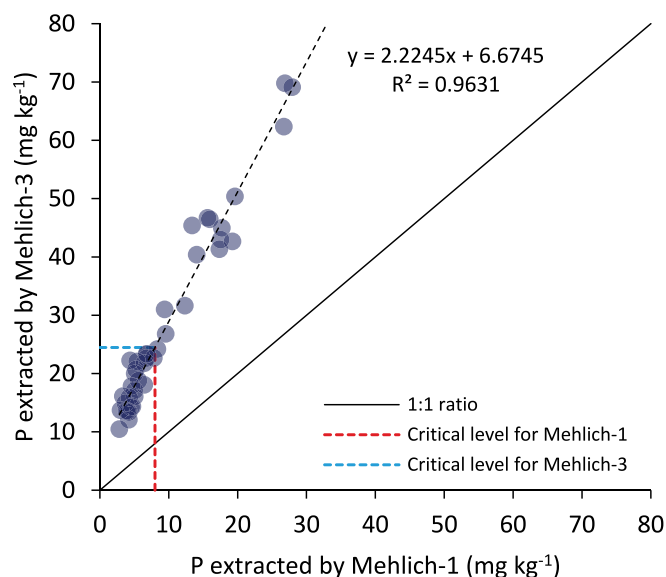
The results obtained in the present study indicate that the critical



**Fig. 9.** Accumulated P extracted by Mehlich-1 after 17 successive extractions up to 20 cm depth after 32 years of different soil tillage managements (NT, no-tillage; CT, conventional tillage; OT, occasional tillage) combined or not with liming in a Brazilian subtropical Oxisol.

level of P extracted with Mehlich-1 established for the south-central region of the state of Paraná is adequate (Fontoura et al., 2015). In treatments where acidity was not limiting (lime-CT, lime-OT, lime-NT) it is possible to verify that the maximum yield (Fig. 2) for all crops was obtained only in treatments with Mehlich-1 P greater than  $8.0 \text{ mg dm}^{-3}$

in the 0–20 cm layer, that is, in lime-OT and lime-NT (Fig. 8a). However, in the lime-CT treatment, the Mehlich-1 P was approximately half the critical level ( $4.6 \text{ mg dm}^{-3}$ ), resulting in grain yield 10% lower than with liming under NT or OT (Fig. 2). Although there is still no calibration for the Mehlich-3 in the studied region, the correlation between the



**Fig. 10.** Relationship between the content of P extracted by Mehlich-1 and the content of P extracted by Mehlich-3. The critical level for Mehlich-1 ( $8 \text{ mg dm}^{-3}$ ) was obtained by Fontoura et al. (2015), and the corresponding value of P critical level for Mehlich-3 ( $24 \text{ mg dm}^{-3}$ ) was estimated in the present study.

Mehlich-1 and Mehlich-3 indicates that the critical level for this Mehlich-3 is approximately  $24 \text{ mg dm}^{-3}$  (Fig. 10), very close to the equivalent value proposed by the IPNI (2015), i.e.,  $23 \text{ mg dm}^{-3}$ .

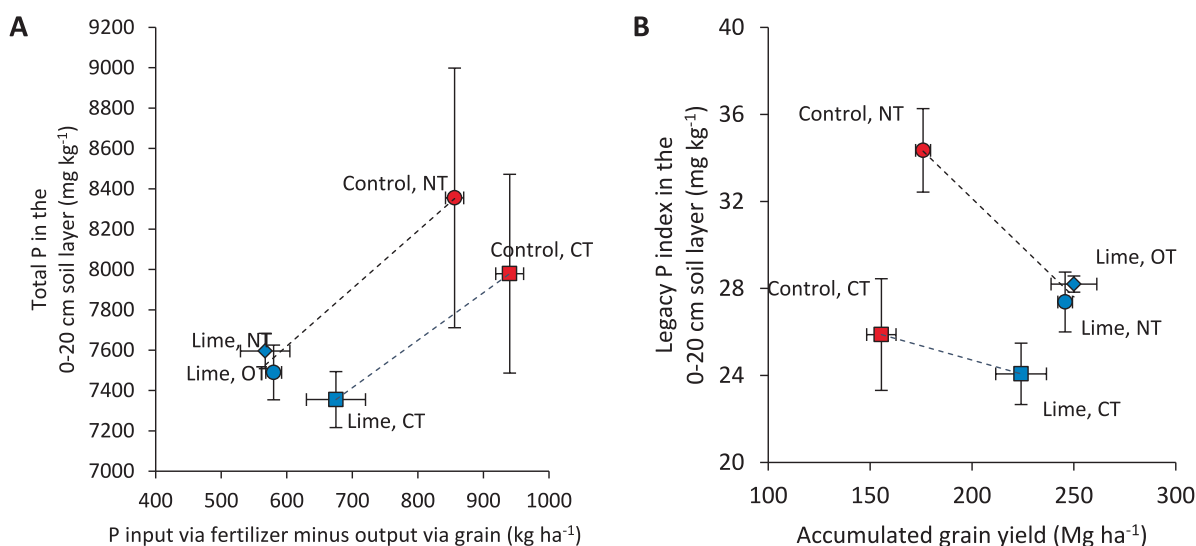
The lower content of Mehlich-1 P in the CT treatments occurs because the frequent tillage of the soil exposes P to new sorption sites, thus decreasing its availability (Tiecher et al., 2017). This is also evident from the higher content of moderately labile inorganic P extracted with NaOH in the 10–20 cm layer in the treatments with soil disturbance (Fig. 6d). In addition, two other factors may be contributing to the lower available P content in these treatments. Firstly, in NT and OT treatments, the lower soil disturbance favors the accumulation of P applied via fertilizer and the cycled by crops in the soil surface. Secondly, soil disturbance can dilute P in layers below 20 cm depth. The lower total P content in the CT is an indication that this is happening (Fig. 7c). Moreover, the difference in P input (fertilization) and output (grain exportation) is higher in treatments with no lime, resulting in higher

total P in the soil (Fig. 11a). However, this increase was higher in NT compared to CT, confirming that part of the P is probably being moved to soil depth below 20 cm due to soil plowing (Fig. 11a).

Soil tillage homogenizes topsoil and dilutes P in-depth, resulting in similar levels of all P fractions in the control-CT and lime-CT treatments (Figs. 6–8). By contrast, in conservation systems (NT and OT), P accumulates in the first 10 cm, forming a vertical gradient of readily available forms (Mehlich 1 and Mehlich 3), moderately available (NaOH and HCl), and occluded forms of P (Fig. 7b). According to Gatiboni et al. (2021b), the accumulation of P on the surface saturates the most avid sorption sites and accumulates the nutrient in forms of greater availability. This becomes evident when evaluating the degree of saturation of P (Fig. 6c). The results of P desorption with Mehlich-1 are also evidence of P accumulation in more available forms in soil under NT. This can be verified by the highest proportion of P readily/potentially available (Fig. 8c) and by the higher proportion of potentially available P/total P (Fig. 8d) in NT than CT treatments. Moreover the higher content of P and Ca on the soil surface also facilitates the formation of new Ca phosphates (Somavilla et al., 2021), which represents a small fraction (Fig. 7a) but that can act as an active buffer for available P in acidic soils (Tiecher et al., 2018).

The surplus of P applied not exported by grains is accumulating in the soil (Fig. 11a) in all systems (CT, NT, and OT), but only in conservation systems (NT and OT), this accumulation is occurring in potentially available forms. Interestingly, legacy P index was the only parameter used that was able to differentiate treatments with and without liming under CT (Fig. 7d). The difference in legacy P index can be explained by the removal of the nutrient by the crops, both in CT and in conservation systems (NT and OT). The higher the accumulated grain yield (Fig. 11a), the smaller the legacy P index, indicating that the applied P not exported by grains is accumulating in potentially available forms in the soil. However, the legacy P index is greater when using NT compared to CT, highlighting that conservationist soil systems are an important strategy to accumulate P in forms potentially available to plants.

The occasional tillage to incorporate lime did not improve crop yield or P accumulation in the soil compared to surface liming, as shown by the similar P use efficiency and legacy P index in lime-OT and lime-NT treatments. This indicate that it is not necessary to repeat the operation of lime incorporation even after more than four decades (42 years), as the last liming with incorporation in the lime-NT treatment in 1977 was sufficient to maintain an adequate root environment for the



**Fig. 11.** Relationship between the difference of P input minus P output and the total P in the 0–20 cm soul layer (A) and the relationship between accumulated grain yield from 1987 to 2019 and the legacy-P index in the 0–20 cm soil layer (B).



development of plants and ensure adequate P uptake. After the in-depth acidity correction, soil acidity can be managed by applying lime to the soil surface, as soil re-acidification starts in the topsoil, due to fertilizers with acidic reaction (Wang et al., 2019; van Oort et al., 2022). In addition, the root system of crops increases after a deep soil acidity correction, improving the aggregation and structure of the soil, forming bio pores, and accumulating organic matter. This allows the mobilization of broadcast limestone particles in-depth and greater downwards movement of limestone dissolution products. In no-till areas without a history of lime incorporation application, acidity correction is limited to the first 10 cm of the soil in the short-medium term (Rheinheimer et al., 2018; Bellinaso et al., 2021). By contrast, when the soil acidity has been deep corrected previously and the soil is properly managed with no-tillage and crop rotation, it is possible to detect an increase in soil pH up to 60 cm deep in the first year after surface lime application (Fontoura et al., 2019). These results are consistent with the third law of Soil Chemistry proposed by Barrow (1999), which states that the same soil cannot be limed twice because the soil is permanently altered after one single limestone application.

## 5. Conclusion

The results of this study indicate regardless of the soil management practices used, correcting soil acidity is a crucial step to ensure high crop yields and optimal use of phosphorus. However, this effect can be further enhanced by combining liming with conservation systems such as no-tillage. In other words, the combination of liming and NT can have a synergistic effect on crop yield and phosphorus use efficiency. Moreover, after an initial incorporation of lime, subsequent liming can be done on the soil surface without the need for additional lime incorporation. This approach can ensure high efficiency of phosphorus use for more than four decades.

## Declaration of Competing Interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: Tales Tiecher reports financial support was provided by National Council for Scientific and Technological Development. Cimelio Bayer reports financial support was provided by Yara Brasil SA.

## Data availability

Data will be made available on request.

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## Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.geoderma.2023.116495>.

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