Critical limit for phosphorus in a Red Latosol of the Brazilian Cerrado[1]

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ABSTRACT - Successive applications of phosphate fertilisers can exceed the phosphorus (P) requirements of plants and soil, resulting in surface accumulation and loss to aquatic systems, and therefore require monitoring. The aim of this study was to determine a mathematical model for the critical limit for phosphorus in the environment (CLPE) in a Red Latosol of the Cerrado, and to monitor the amount of P in the soil of 46 rural properties in the micro watershed of the Ribeirão Abóbora River. Sand was added to a dystroferric Red Latosol to simulate soils with textures of 10%, 20%, 30%, 40% and 50% clay, in order to quantify the maximum P adsorption capacity (MPAC) and determine the CLPE. Soil from 46 rural properties that make up the micro watershed of the Ribeirão Abóbora River was collected, and the P and clay content was determined. The MPAC decreased proportionally to the reduction in clay content. The estimating equation to determine the CLPE for the Red Latosol of the Cerrado is CLPE = 16.64 + 0.432* (% clay), which can be simplified to CLPE = 20 + 0.5* (% clay). The P content on four of the properties in the micro watershed of the Ribeirão Abóbora River is greater than the CLPE. It is therefore recommended that the use of phosphate sources be suspended until appropriate P levels return, and that close attention be paid to these properties so that P values again reach acceptable levels, and no P is transferred to the water sources.

Key words: Phosphate fertilisation. Environmental control. Soil management.

INTRODUCTION

The Brazilian Cerrado is widely used for planting soya beans, maize and sorghum, among other crops. In general, the soils in this region are naturally acidic and not very fertile (Novais; Smyth, 1999), and for agricultural use it is necessary to use high doses of fertiliser, particularly phosphates (Gotz *et al*., 2023). Successive applications of phosphate fertiliser, especially when the phosphorus (P) exceeds the requirements of the plants and soil, can result in P accumulating in the surface layers of the soil, and increase the risk of losses to aquatic systems, an environment where excess P can cause eutrophication, affecting the entire river basin (Geletu, 2023; Liang *et al*., 2022; Wang *et al*., 2022).

Currently, the southwestern region of Goiás is one of the most important in Brazil for poultry and pig production (Tavares *et al*., 2019), increasing the generation of P-rich waste that needs to be properly managed to prevent water pollution. Due to the high cost of phosphate fertilisation, and nutrient sources for fertiliser production being limited (Anlauf, 2023), some farm owners end up using the waste as fertiliser (Niu *et al*., 2023; Tavares *et al*., 2019), albeit without properly monitoring the levels of P in the soil.

The Ribeirão Abóbora River is the main water supply of the population of Rio Verde in Goiás. The waters of the Ribeirão are also used in the production process of an agro-industrial complex, for supplying rural properties for domestic use, and for watering animals. The only authorised uses are the abstractions made by SANEAGO for public supply and agro-industry. It is therefore important to establish limits for P applications in cultivated soils in the central-west of Brazil, especially in annual crops, to ensure good plant nutrition and minimise the impact on the environment.

To this end, the critical limit for phosphorus in the environment (CLPE) was established based on the clay content of the soil, and represents approximately 80% of the soil P retention capacity using the Mehlich-1 method of P extraction (Gatiboni *et al*., 2015). Mehlich-1 is sensitive to the buffering effect of the soil, and extracts less P in more-clayey soils. On the assumption that each CLPE differentiated by Mehlich-1 is sensitive to the clay content of the soil, then, due to this clay content, there is not just one, but several CLPE, and it becomes necessary to determine the CLPE for each region and type of soil.

The Maximum P Adsorption Capacity (CMAP) shows what can happen to the P added to the soil in terms of both use and subsequent use, comprising the so-called 'residual effect'. The MPAC was used to determine the CLPE. Since the CLPE is dependent on the clay content and on the adsorption properties of the soil, and is directly related to the degree of soil P saturation, it is a measure of soil P availability and the absorption capacity of the soil complex (Bai *et al*., 2013; Mcdowell *et al*., 2001).

The aim was therefore to determine a mathematical model, the CLPE, for a Red Latosol in the Cerrado, and monitor the P content in soils of 46 rural properties in the micro watershed of the Ribeirão Abóbora River.

MATERIAL AND METHODS

Preparing and characterising the soils – determining the isotherms

The study was conducted from March 2019 to June 2020, at the University of Rio Verde (UniRV), in Rio Verde, Goiás, Brazil. As all of the stages were carried out in a controlled environment, a completely randomised design was adopted for each stage. A Red Latosol (of unique mineral composition) with a high clay content, typical of the region, and obtained from an agricultural property in the municipality of Rio Verde, Goiás, at a depth of 0 to 20 cm, was used to produce soils of different textures.

The soil was dried in a forced ventilation oven at 60 °C and sieved through a 2-mm mesh. The initial analysis showed that the soil contained 52.2% clay, 15.30% silt, 32.50% sand and 38.10 g dm^{-3} organic matter (EMBRAPA, 1997). The iron content was 7.90 mg dm^{-3}, the cation exchange capacity of the soil at pH 7.0 was 13.70 cmol$_c$ dm^{-3}, the pH (CaCl$_2$) was 5.10, and the base saturation was 59.30% (EMBRAPA, 1997).

Soils with a different clay content were prepared by mixing the soil with sand (Oliveira *et al*., 2010) in the following proportions: C1 - 100% sand: 0% soil; C2 - 75% sand: 25% soil; C3 - 50% sand: 50% soil; C4 - 25% sand: 75% soil; C5 - 0% sand: 100% soil. Before mixing, the sand was washed in running water, stirred for 30 minutes, and then left for one hour in contact with hydrochloric acid (HCl 0.1 mol L^{-1}) in a 1:1 ratio. The sand was then washed with running water, dried in a forced air oven at 60 °C and sieved through a 1-mm mesh.

After mixing with the sand, the soils were placed in recyclable plastic bags and incubated for 30 days to induce minimal recombination of the soil particles and stabilise the physical and chemical properties of the soil-sand mixture. The moisture content at field capacity of the samples was estimated following saturation and 24 hours drainage (Casaroli; Van Lier, 2008).

The soils were incubated for 30 days, during which time the samples were homogenised weekly. The soils were then incubated for a further 30 days with a limestone filler (PRNT = 98.1%) to correct the pH to 6.0. The amount of limestone was calculated using the SMP

index, as recommended by the Soil Chemistry and Fertility Commission of Rio Grande do Sul and Santa Catarina (Sociedade Brasileira de Ciência do Solo, 2004). Sixty days after incubation, the soil was dried in a forced air oven at 60°C, sieved through a 2-mm mesh, and analysed in the multi-user laboratory at UniRV. At the end of the incubation process, the soils presented the following clay content: C1 = 12.90%; C2 = 25.20%; C3 = 34.20%; C4= 44.35% and C5= 52.20% (Table 1), with textures that ranged from sandy to clayey.

Determining the maximum P adsorption capacity (MPAC) of the soils

For this step, the method of Vilar *et al.* (2010) was used, in which 2.5 g of each soil were placed in a Falcon tube, with four replications, to which was added a 0.01 mol L^{-1} KCl solution at pH 6 containing 0.5, 10, 20, 60, 80 and 120 mg L^{-1} P in the form of KH_2PO_4. The samples were stirred for 16 hours at 30 revolutions per minute (rpm) in a Wagner stirrer. The supernatant was then separated from the soil by centrifuging at 5,000 rpm for 14 minutes.

The P concentration of the supernatant (solution in equilibrium) was determined using the colorimetric method (EMBRAPA, 1997). The amount of adsorbed P was determined as the difference between the initial concentration and the concentration of the solution. The data from incubating the soils with different rates of P were used to verify the dependence of P-water on P-Mehlich-1 and the clay content, carrying out multiple regression analysis (p < 0.05) using the SigmaPlot software and Excel spreadsheets. Adsorption isotherms were constructed, plotting the amount of adsorbed P on the ordinate and the concentration of the solution in equilibrium on the abscissa. The hyperbolic form of the Langmuir equation is given by the following expression:

$$x/m = (abC)/(1+aC) \tag{1}$$

where, x/m: number of ions adsorbed on the solid phase, b: maximum capacity of the solid phase to adsorb ions, and C: ion concentration on the solid phase in the solution in equilibrium.

To estimate the a and b constants, the Langmuir hyperbolic equation was transformed into its linear form:

$$C/(x/m) = 1/(ab) + (1/b)C \tag{2}$$

MPAC (b) was determined as the inverse value of the slope of the line, the adsorption energy constant (a) from the relationship between the slope of the line and its intersection with the ordinate axis, and the maximum P capacity factor (PCF max.) as the product of the maximum P adsorption capacity and the adsorption energy (Bahia Filho *et al.*, 1983; Novais; Smyth, 1999).

Determining the critical limit for phosphorus in the environment (CLPE)

For this step, the method proposed by Gatiboni *et al.* (2014) was adopted. After determining the MPAC for each soil, the information was used to incubate the soils with a dose of P proportional to the MPAC. For each soil, a dose of P close to the MPAC was selected, and the soil separately incubated with 0%, 12.5%, 25%, 50%, 75% and 100% of this dose, with four replications. The soil with added P was incubated for 20 days, during which the samples were kept at 80% field capacity.

Table 1 - Physical and chemical attributes of the prepared soils following incubation for 30 days with water and a further 30 days with limestone

Attribute	Unit	Soil:sand ratio (%)				
		100:0	75:25	50:50	25:75	0:100
pH (CaCl$_2$)	-	7.05	7.05	6.85	6.65	6.45
Ca	cmol$_c$ dm^{-3}	1.15	2.7	4	5.2	5.85
Mg	cmol$_c$ dm^{-3}	0.49	1.21	1.64	1.86	2.00
Al	cmol$_c$ dm^{-3}	0	0	0	0	0
H + Al	cmol$_c$ dm^{-3}	0.55	0.6	0.7	0.8	0.8
K	mg dm^{-3}	15	48	87	118	145
P Mehlich-1	mg dm^{-3}	4.45	11.85	13.15	11.15	10.75
Clay	%	12.9	25.2	34.2	44.35	52.2
Silt	%	7.1	9.8	13.3	15.65	15.3
Sand	%	80	65	52.5	40	32.5
CEC	cmol$_c$ dm^{-3}	2.23	4.64	6.56	8.16	9.02
Base saturation	%	75.25	87.05	89.35	90.2	91.15

After incubation, the levels of water-soluble P (P-water) and plant-available P (P-Mehlich-1) were determined. To determine the P-water, 1.0 cm^3 of soil and 10 mL of distilled water were used. The samples were stirred for one hour in a Wagner stirrer at 30 rpm. The samples were then centrifuged at 5,000 rpm (approximately 2,000 G) for 10 minutes, and the P in the solution was determined (Murphy; Riley, 1962). The available P by Mehlich-1 was found using 1.0 cm^3 of soil and 10 mL of extraction solution. After stirring the soil with the extraction solution for five minutes, the samples were centrifuged at 5,000 rpm (approx. 2,000 G) for five minutes to determine the P in the solution (Murphy; Riley, 1962).

The CLPE was obtained using soils incubated with doses of P, adjusting the functions relating the available P content by Mehlich-1 (P-Mel) and the content of P in water (P-water), using a two-segment linear regression. The SigmaPlot software and Excel spreadsheets were used to determine the levels of P-Mehlich-1 at which the change point occurs and there is a dramatic increase in P released into the water. The adjusted mathematical functions are two-segment linear equations, where the intercept point between the two segments is the 'P-limit' of the soil, i.e. the P-Mehlich-1 level above which there is a high risk of P transferring from the soil to the water. The P-limit was set at 80%.

Effectiveness of the CLPE in soil samples from the micro watershed of the Ribeirão Abóbora River

The 100 rural properties that make up the watershed of the Ribeirão Abóbora River were identified in 2019 with the assistance of the Municipal Department of the Environment of Rio Verde, Goiás, and the Association of Water Producers of the Municipality of Rio Verde. The P and clay content was determined in 46 properties at a depth of 0-20 cm using a Dutch auger, as per the instructions in the Methods Manual of Chemical Analysis from Embrapa (Teixeira *et al.*, 2017). The soil samples were dried in a forced air oven at 60 °C

and sieved through a 2-mm mesh. The 46 rural properties were standardised based on their cereal production, under a soybean + maize/sorghum/millet cropping system. Properties growing only pasture crops were not considered.

In the 46 sampled soils, P levels were determined using the Mehlich-1 method and clay levels using the densimetric method (Silva *et al.*, 1999), in the multiuser laboratory at UniRV. The P levels were then classified based on the clay content of each property (Sousa; Lobato, 2004) and the soils classified as below or above the limit for P, and plotted on a graph relating P-Mehlich-1 to P-water.

RESULTS AND DISCUSSION
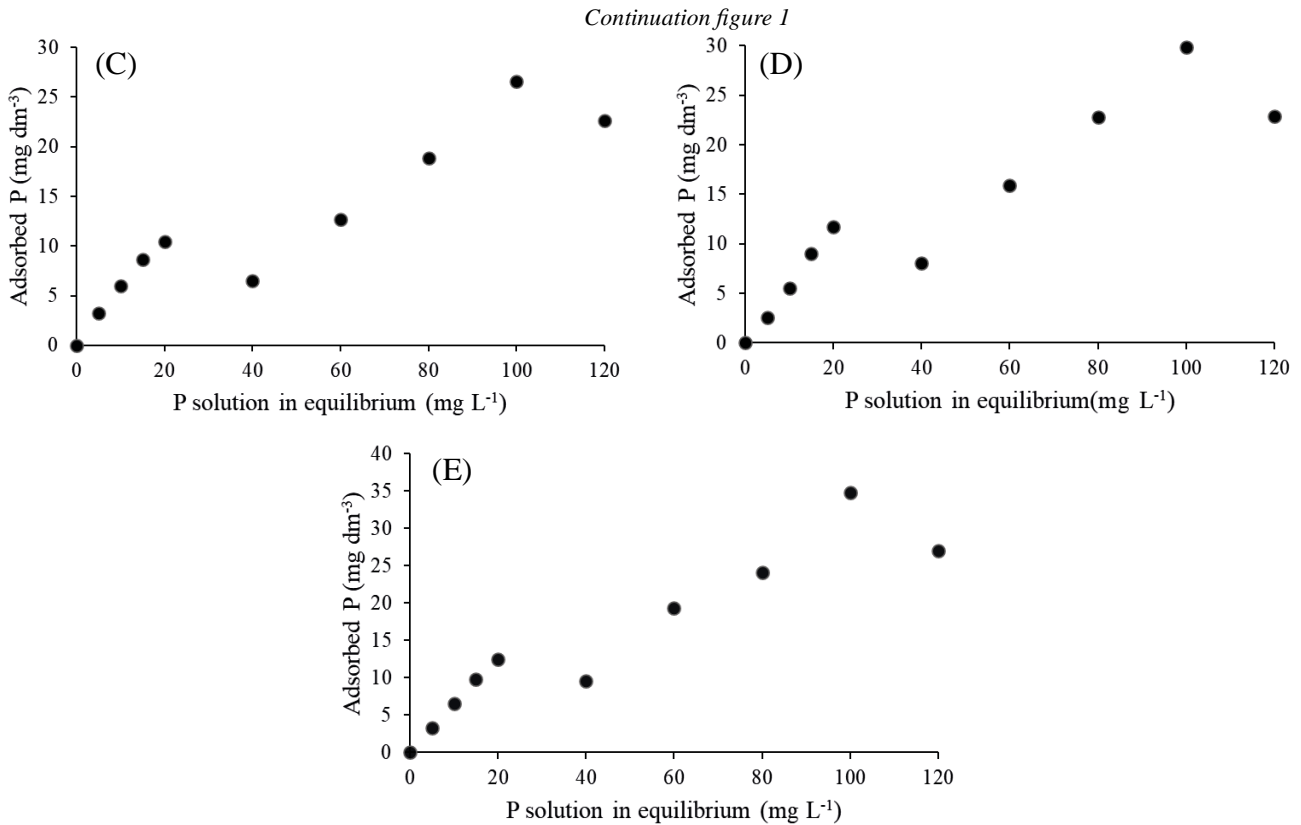
Determining the isotherms

The shape of the P adsorption isotherms is close to an S-curve (Figure 1). These curves characterise the low affinity of the absorbate (ion in solution) for the absorbent (solid phase of the soil) at low concentrations due to the interference of other substances. It was therefore necessary to divide the P concentration into two orders, 0-20 and 40-120 mg L^{-1}. For soil, the relationship between the number of ions chemically adsorbed on the solid phase and their concentration in the soil solution can generally be described by four types of isotherm: type L, type S (found in the present study), type C and type H (Meurer; Rheinheimer; Bissani, 2017).

Maximum P adsorption capacity (MPAC)

Based on the P adsorption curves, the MPAC of the soils were 6 mg dm^{-3} for soil with 10% clay, 53 mg dm^{-3} for soil with 20% clay, 82 mg dm^{-3} for soil with 30% clay, 87 mg dm^{-3} for soil with 40% clay, and 137 mg dm^{-3} for soil with 50% clay (Figure 2A). For soils that received P doses between 0 and 20 mg dm^{-3}, the values did not adjust to the model and are therefore not shown.

Figure 1 - Relationship between the P concentration of the solution in equilibrium and the amount of adsorbed P, in soils with 10% (A), 20% (B), 30% (C), 40% (D) and 50% (E) clay

Continuation figure 1
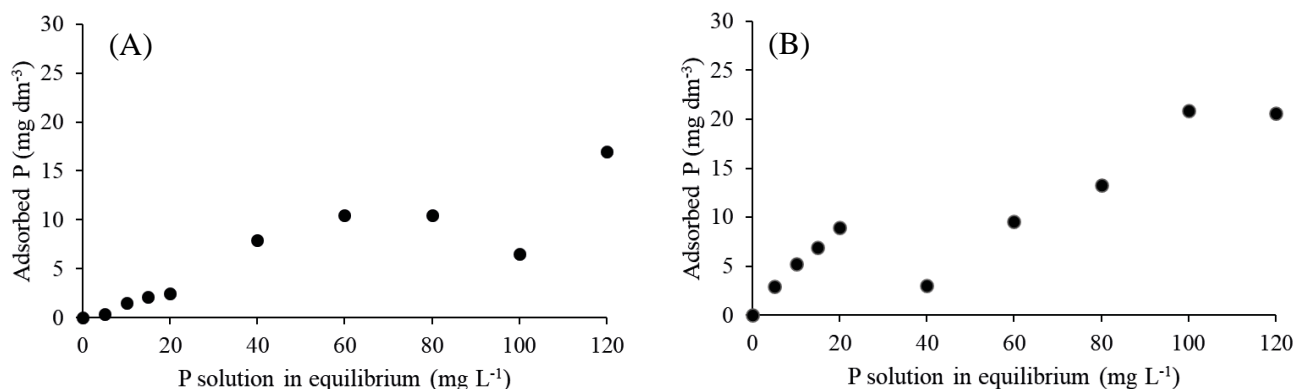
Figure 2 - P adsorption isotherms using the Langmuir model, in soils with 10%, 20%, 30%, 40% and 50% clay (A), and the increase in the maximum P adsorption capacity (MPAC) as a function of the soil clay content (B)
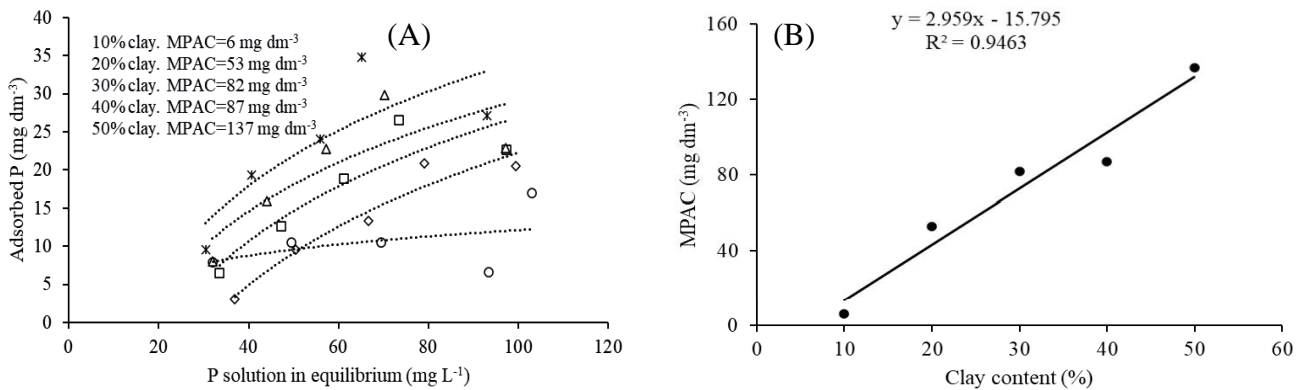
From the results of the isotherms, an increase can be seen in the MPAC of 2.959 mg dm^{-3} (Fig. 2B) for each percentage unit of clay in the soil. The P adsorption process described by the Langmuir isotherm considers at least two distinct stages of adsorption. During the initial stage with the addition of small doses of P, due to the high fixation capacity of the element in the soil, almost all the added P is absorbed by the soil, leaving little P in the solution. During the second adsorption phase, the amount of added

P was higher and the binding sites in the soil were at least partially saturated, so the amount of P adsorbed onto the soil particles was smaller, leaving more P in the solution (Muljadi; Posner; Quirk, 1966).

When the soil reaches the MPAC, the P content of the solution in equilibrium is very high; if fertiliser at this strength is added to the soil, P will be transferred to the water. It is therefore not possible to use the MPAC to estimate the limit dose of P that can be applied to the soil without causing a

significant impact on the environment. In addition, generating adsorption isotherms is laborious and time-consuming, making it impossible to determine the MPAC in conventional soil-testing laboratories (Allen; Mallarino, 2006).

The values of P in water and available P determined by Mehlich-1 after incubating the soil sample for 20 days with six doses of P showed that the results for P-water were very close to zero when low doses of P were used (Figure 3). This result shows that in soils that are not saturated with P, particularly clayey soils, most of the P is adsorbed in the soil, with no increase in P-water. This result is in line with that of Gatiboni *et al.* (2014). However, when higher P doses were added, the levels of P-water increased, first in sandy soils and then in the more-clayey soils.

In Brazil, the acceptable P limit in water varies from 0.02 to 0.15 mg L^{-1}, depending on the intended use of the water and the type of aquatic environment (National Environmental Council, 2005). These limit values are valid for waters from aquatic systems (rivers, lakes, etc). According to Gatiboni *et al.* (2015), using the above equations it is difficult to assess the change point at

which P-water begins to increase rapidly in response to the addition of phosphate, making it necessary to use fractional equations, which are more accurate and more widely used in similar studies that estimate change points.

In the soil with 10% clay, P begins to be transferred from the soil to the water at a P content of 18 mg dm^{-3} (Fig 4A). The change points for the soils with 20% (Fig. 4B), 30% (Fig. 4C), 40% (Fig. 4D) and 50% (Fig. 4E) clay were 40, 41, 42 and 43 mg dm^{-3} P, respectively. The change points may vary according to the characteristics of the soil, including the soil P sorption capacity and pH of the water (Bai *et al.*, 2013).
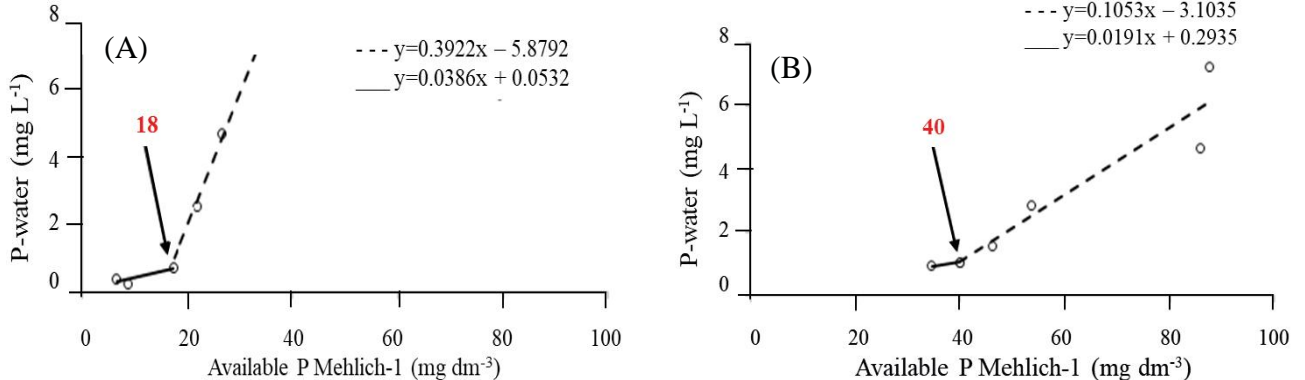
Determining the CLPE equation

There was a linear increase in the value of these parameters with the increase in the clay content. As mentioned above, the CLPE was established at 80% of the P-limit; the equation that represents the CLPE is therefore: CLPE (mg dm^{-3}) = 16.64 + 0.432* (% clay), where % clay is the soil clay content as a percentage (Figure 5). It is suggested the equation be simplified to CLPE (mg dm^{-3}) = 20 + 0.5* (% clay). The use of the simplified equation has the advantage of being easy to memorise and easy to use (Gatiboni *et al.*, 2015). Another advantage of using the simplified equation is that the appropriate CLPE can be calculated for each type of soil, since if the idea of establishing ranges of clay content used by CQFS-RS/SC (Brazilian Society of Soil Science, 2004) were adopted, a soil with 21% clay and another with 39%, for example, would have the same CLPE, as they fall within the same range of clay content.

The data confirm that clay soils support higher P doses than do sandy soils, without causing problems to the environment, and also confirm the hypothesis that the CLPE determined by Mehlich-1 is dependent on the clay content, and should be taken into account when developing a model. This finding is similar to that supported by Mcdowell *et al.* (2001) and Bai *et al.* (2013), that the CLPE should take into account the P retention capacity of the soil.
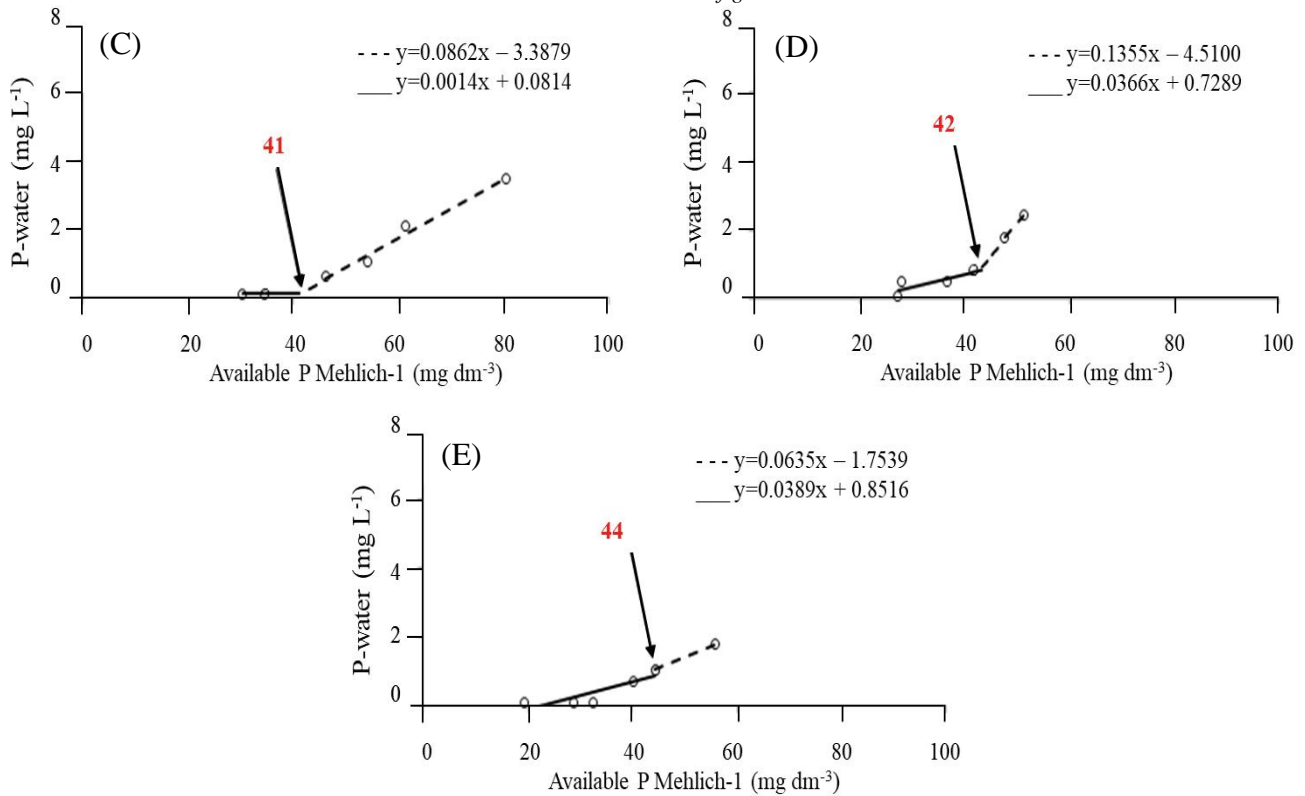
Figure 3 - P adsorption isotherms using the Langmuir model, in soils with 10%, 20%, 30%, 40% and 50% clay
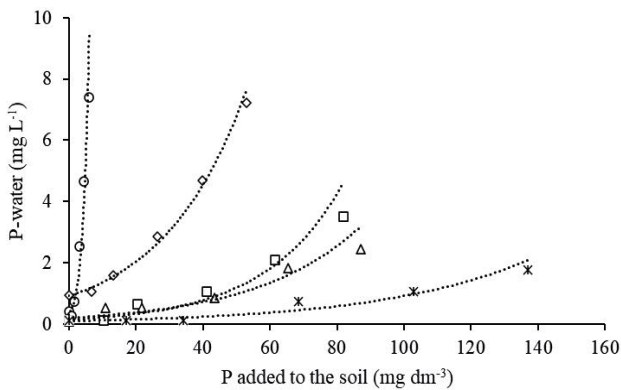
Figure 4 - Relationship between the P-water content as a function of the P-Mehlich-1 content in soils with 10% (A), 20% (B), 30% (C), 40% (D) and 50% (E) clay. The point at which the equations intersect is the value at which the P moves from the soil to the water

Continuation figure 4

(C) --- y=0.0862x − 3.3879
___ y=0.0014x + 0.0814

(D) --- y=0.1355x − 4.5100
___ y=0.0366x + 0.7289

(E) --- y=0.0635x − 1.7539
___ y=0.0389x + 0.8516

Figure 5 - Critical limit for P in the environment (CLPE) based on the soil clay content. PLim: P content limit for an increase in the P released into the water; and CLPE (simplified): simplified function for calculating the CLPE

CLPE: CLPE=16.64+0.432*(%arg), R^2=0.6339
Simplified model: CLPE=20+0.5*(%clay)
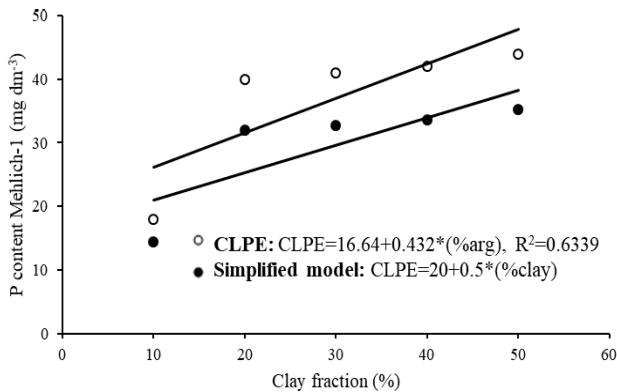
Assessing the CLPE on rural properties in the micro watershed of the Ribeirão Abóbora River

The P content on the 46 properties under analysis ranged from very low to very high, and was dependent on the clay content, with 51% of the soils classified as very poor, 18% as poor, 13% as moderate, 2% as adequate, 7% as high and 9% as very high. Two percent of the soils on the properties had a sandy texture, 72% had a medium texture, 2% a clayey texture, and 2% a very clayey texture (data not shown). The extraction efficiency of available P by the Mehlich-1 method is affected by the phosphate buffering capacity of the soil (Nunes *et al.*, 2021). Therefore, properties related to buffering capacity, such as the clay content or remaining P, are used to explain P availability.

The 'CLPE = 20 + 0.5* (% clay)' model can separate the samples into two levels, where the P content is above or below the CLPE (Figure 6). The soils of four of the rural properties under evaluation have a P content above the CLPE, and it is recommended that the application of phosphate sources be suspended in these locations to avoid contamination of the water sources.

The model was effective in identifying soil samples with the greatest potential for P transfer, i.e. those with a P content above the CLPE, suggesting they present a high risk of contamination to water sources through the transfer of P from the soil-water system by leaching and, particularly, by surface runoff, as these are a rich source of phosphorus. Runoff, which is rich in P, is one of the main factors promoting algal growth, leading to eutrophication (Geletu, 2023; Soro *et al.*, 2023).

Figure 6 - Mehlich-1 P and P-water content on 46 rural properties in the micro watershed of the Ribeirão Abóbora River
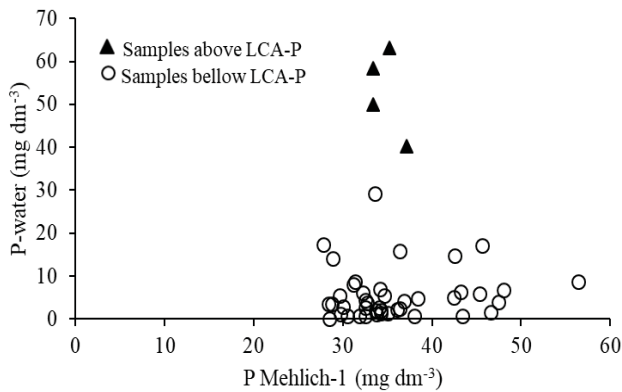
In addition to mineral fertilisation using phosphate fertilisers, a common practice, not only in the central-west of Brazil but in Brazil as a whole, is for farmers to use pig slurry as organic fertiliser, since they own pig farms and need to dispose of the waste, and since mineral fertiliser is expensive (Antoneli *et al*., 2019; Santos *et al*., 2022; Tavares *et al*., 2019). However, P in the soil must be properly managed, as despite the nutrient being fundamental to plant growth, too much is harmful to water resources.

Gatiboni *et al*. (2015), working with the CLPE of 82 samples collected in different areas of the state of Santa Catarina, showed that the calculated value will always be above the critical level, by almost three times in soils with 20% clay and eleven times in soils with 60% clay. As such, when using the CLPE model to limit fertilisation using animal waste, for example, there is no risk of restricting plant growth due to a P deficiency.

CONCLUSIONS

The estimating equation to determine the CLPE for the Red Latosol of the Cerrado is CLPE = 16.64 + 0.432* (% clay), which can be simplified to CLPE = 20 + 0.5* (% clay). The P content on four of the properties in the micro watershed of the Ribeirão Abóbora River is above the CLPE, and requires that the use of phosphate sources be suspended until appropriate P levels return, and close attention be paid to these properties so that P values again reach acceptable levels, and no P is transferred to the water sources.

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