

Division - Soil Use and Management | Commission - Lime and Fertilizer

Evaluation of traditional methods for estimating lime requirement in Brazilian soils

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ABSTRACT: The optimal soil pH for most annual crops in Brazil varies between 5.7 and 6.0. Numerous methods have been developed for estimating lime requirement (LR), but they vary widely in their predictions and fail to raise pH to desired values for optimum crop production in the highly weathered soils of Brazil. The objectives of this study were to (i) compare seven traditional methods for estimating LR in Brazilian soils; (ii) assess the effects of LR predicted by these methods on soil-acidity related properties, and (iii) determine if these methods are predicting LR to attain target pH values of 5.8 and 6.0, which are within the pH range recommended to optimize crop yields. The traditional LR methods evaluated in this study are based on the following criteria: exchangeable acidity (EA), base saturation (BSAT), exchangeable acidity along with Ca^{2+} and Mg^{2+} as proposed by the 4th (MG4A) and 5th (MG5A) Approximations to the Minas Gerais State, SMP soil-buffer pH (SMP), potential acidity (PA), and soil pH along with organic matter (pHOM). These methods were compared with the standard incubation method using correlation-regression analysis and, alternatively, the identity test designed for assessing equivalence between methods. Representative agricultural soils ($n = 22$) were incubated for 60 days with incremental amounts of lime determined by the tested methods. On average, LR predictions differed among methods, and increased in the following order: $\text{EA} < \text{BSAT} \approx \text{MG5A} \leq \text{MG4A} \approx \text{SMP} \leq \text{PA} < \text{pHOM}$. Suitable changes in soil pH, exchangeable acidity, potential acidity, base saturation, and Ca^{2+} and Mg^{2+} were achieved upon application of LR estimated by all methods except the EA and pHMO, which resulted in undesirable soil acidity characteristics. All methods evaluated in this study were unable to predict LR for attaining target pH values of 5.8 and 6.0 as revealed by the identity test, even though they were moderate to strongly correlated with the standard incubation method as indicated by the correlation-regression analysis. Further research should focus on the development of reliable methods for predicting LR to attain desired pH values and consequently maximize crop production on Brazilian soils.

Keywords: lime requirement predictions, soil acidity, comparison between methods, correlation, identity.

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INTRODUCTION

Soil acidity is a major factor that limits crop growth and yield in many regions of the world (Fageria and Nascente, 2014). In the most productive agricultural land in Brazil, known as tropical savanna (Cerrado), the majority of the soils are categorized as acidic, with low natural fertility, high exchangeable acidity saturation, and high P fixation capacity (Lopes and Guilherme, 2016). Liming is the most common practice used for the amelioration of acidic soils thereby providing suitable conditions for crop growth. Adding lime to acid agricultural soils has the overall goal of increasing pH up to values (i.e., pH 5.5-6.0) that maximize nutrient availability, eliminate toxicity due to high levels of Al^{3+} and Mn^{2+} , and decrease P immobilization thus enhancing crop production (Kunhikrishnan et al., 2016; Kalkhoran et al., 2019). Suitable prediction of LR is therefore needed to obtain desired soil pH values that allow an optimum crop production.

Several methods to estimate lime requirement (LR) of Brazilian acid soils are available. These methods have been developed since the 1970s, when soil analysis was implemented as a diagnostic tool to assess soil fertility and determine lime and fertilizer recommendations in Brazil (Lopes and Guilherme, 2016). Traditional methods largely used in Brazil to estimate LR are based on the increase of soil base saturation (BSAT method) (Joris et al., 2016; Moreira et al., 2017; Nowaki et al., 2017), neutralization of exchangeable acidity (M^{x+}) and increase of Ca^{2+} and Mg^{2+} (MG5A method) (Silva et al., 2009; Guarçoni and Sobreira, 2017), and increase of the soil pH to reference values based on the pH change of a soil:SMP buffer suspension (SMP method) (Alves et al., 2019; Nunes et al., 2019). Other methods used to a lesser extent for predicting LR are based on exchangeable acidity, potential acidity, and target pH along with organic matter (Borges Júnior et al., 1998; Almeida et al., 1999; Campanharo et al., 2007; Caballero et al., 2019).

Despite the variety of methods available for predicting LR, uncertainties about their efficiency have been constantly reported on literature. For instance, many researchers have shown that the desired soil base saturation was not achieved when LR was predicted by the BSAT method, particularly at the highest lime rates and for soils with high buffering capacity, observing the need of a much higher LR for achieving the corresponded increase in soil pH (Alleoni et al., 2005; Soratto and Crusciol, 2008; Predebon et al., 2018). The method aiming to neutralize M^{x+} and increase Ca^{2+} and Mg^{2+} have resulted in excessive LR to medium texture soils (clay <30 %) containing low cation exchange capacity at pH 7.0 ($T < 4 \text{ cmol}_c \text{ dm}^{-3}$) and high base saturation ($V > 61 \%$) (Sousa et al., 1989). The use of both criteria was also shown to underestimate the LR of soils with $T > 12 \text{ cmol}_c \text{ dm}^{-3}$ and $V < 34 \%$, implying in the partial or total use of the formula according to the soil properties (Sousa et al., 1989).

It is well established that soil-lime incubation with $CaCO_3$ is the most reliable method to estimate the LR needed to raise soil pH to desirable values, being used as a standard to evaluate other methods through linear correlation analysis. However, evaluating whether a certain method is efficiently predicting LR based solely on its linear correlation to a standard method is inappropriate, since the Pearson's correlation coefficient (r) simply reveals a linear association rather than a reliable agreement between two methods (van Stralen et al., 2008). As evidenced in previous studies, several analytical methods have been found to be efficient for estimating LR when they are highly correlated with standardized methodologies (Quaggio et al., 1985; Ernani and Almeida, 1986; Borges Júnior et al., 1998; Almeida et al., 1999; Demattê et al., 2019), even though they can under or overestimate the LR. This is because high r values along with intercepts and slopes quite different from 0 and 1, respectively, may be obtained in linear relationships between methods, indicating differences in their LR predictions even when they are highly correlated.

To assess the equivalence (i.e., agreement) between two different methods, an alternative statistical procedure known as an identity test has been proposed (Leite and Oliveira,

2002). In this approach, two methods are statistically equivalent when: i) intercepts and slopes of the regression line are not different from 0 and 1, respectively; ii) differences between LR predictions are casual; and iii) linear correlation coefficients (r) are higher than $(1 - |\bar{e}|)$. Although the identity test has potential to elucidate whether two measurement methods give similar results, studies using this approach for comparisons between LR methods are still scarce in the literature.

We hypothesize that traditional methods for predicting LR (i) vary widely in their predictions, and (ii) fail to raise the pH to desired values for optimum crop production in the highly weathered soils of Brazil. Furthermore, we hypothesize that (iii) the identity test is preferable for the comparison of LR methods. As such, the objectives of this study were to (i) compare seven traditional methods for estimating LR in Brazilian soils; (ii) assess the effects of LR predicted by these methods on soil-acidity related properties; and (iii) determine if these methods are predicting LR to attain target pH values of 5.8 and 6.0, which are within the pH range recommended to optimize crop yields.

MATERIALS AND METHODS

Soil sampling and characterization

Soil samples from 22 representative sites across the Minas Gerais State were collected in the 0.00-0.20 m layer for a lime incubation study. All soils were below pH 5.5 and obtained from native areas under forest and tropical savanna (Cerrado) that had never been limed (Figure 1).

These soils were selected to be representative of the agricultural Brazilian soils with a wide range of chemical and physical properties. Soils were classified up to the 4th category level (sub-group) according to the Brazilian System of Soil Classification (Santos et al., 2013) and corresponding Soil Taxonomy (Soil Survey Staff, 2014) (Table 1).

Soil samples were air-dried, ground, and passed through a 2-mm sieve for analyses of particle size and chemical properties. Soil texture was analyzed by the pipette method using NaOH 0.1 mol L⁻¹ as dispersing agent and the silt + clay determination as an additional step (Ruiz, 2005). Methods described by Defelipo and Ribeiro (1997) were applied for the soil chemical characterization, which comprised: pH(H₂O) (1:2.5 v/v);

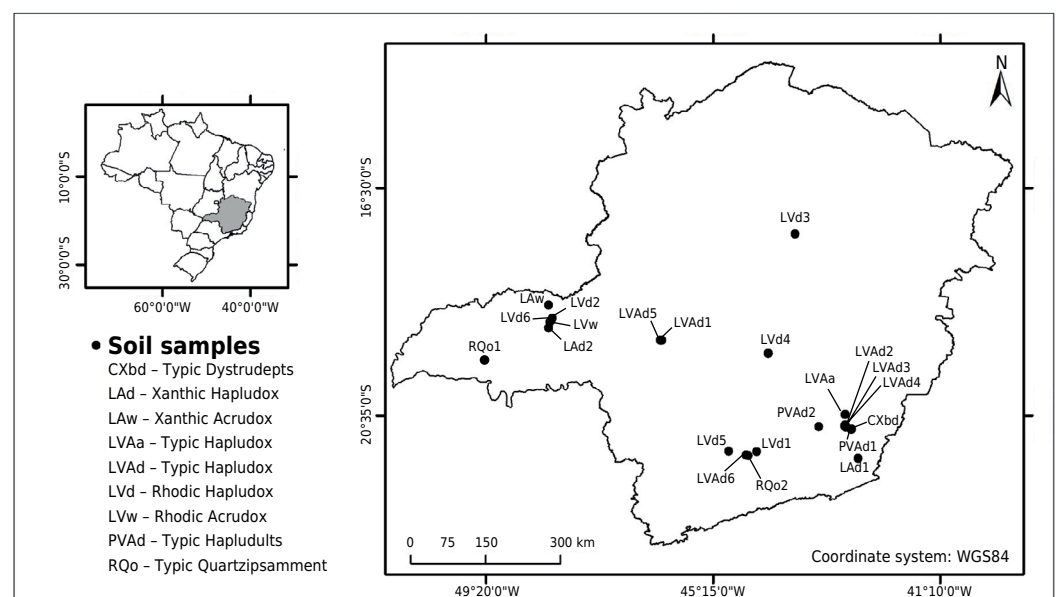


Figure 1. Soil sampling sites across the Minas Gerais State, Brazil.

Table 1. Classification of the soils used in the lime incubation study

Order	Symbol	SiBCS ⁽¹⁾	Soil Taxonomy ⁽²⁾	n ⁽³⁾
Inceptisol	CXbd	<i>Cambissolo Háplico Tb distrófico típico</i>	Typic Dystrustepts	1
Oxisol	LAd	<i>Latossolo Amarelo distrófico típico</i>	Xanthic Haplustox	2
Oxisol	LAW	<i>Latossolo Amarelo Ácrico típico</i>	Xanthic Acrustox	1
Oxisol	LVAa	<i>Latossolo Vermelho-Amarelo alumínico</i>	Typic Haplustox	1
Oxisol	LVAd	<i>Latossolo Vermelho-Amarelo distrófico típico</i>	Typic Haplustox	6
Oxisol	LVd	<i>Latossolo Vermelho distrófico típico</i>	Rhodic Haplustox	6
Oxisol	LVw	<i>Latossolo Vermelho ácido</i>	Rhodic Acrustox	1
Ultisol	PVAd	<i>Argissolo Vermelho-Amarelo distrófico típico</i>	Typic Haplustults	2
Entisol	RQo	<i>Neossolo Quartzarênico órtico</i>	Typic Quartzipsamment	2

⁽¹⁾ Brazilian Soil Classification System (Santos et al., 2013); ⁽²⁾ Soil Taxonomy classes (Soil Survey Staff, 2014); ⁽³⁾ n: number of samples.

exchangeable cations of basic reaction (Ca^{2+} , Mg^{2+} , K^+ , and Na^+) and exchangeable acidity (M^{x+} : exchangeable cations of acid reaction, i.e., Al^{3+} , H^+ , Fe^{2+} , Mn^{2+} ...) extracted with KCl 1 mol L^{-1} ; and potential acidity (HAI: exchangeable and non-exchangeable forms of H and Al, i.e., H^+ , Al^{3+} , covalently bonded H, hydroxy-Al polymers, hydroxi-Al compounds, and organo-Al complexes) extracted with $\text{Ca}(\text{CH}_3\text{CO}_2)_2$ 0.5 mol L^{-1} solution buffered at pH 7.0 (soil:extractant ratio 1:10). The sum of bases ($\text{SB} = \text{Ca}^{2+} + \text{Mg}^{2+} + \text{K}^+ + \text{Na}^+$), cation exchange capacity at pH 7.0 ($\text{T} = \text{SB} + \text{HAI}$); effective cation exchange capacity at the original soil pH ($\text{t} = \text{SB} + \text{M}^{x+}$), base saturation [$\text{V} = (\text{SB}/\text{T}) \times 100$], and exchangeable acidity saturation [$\text{m} = (\text{Mx}^+/\text{t}) \times 100$] were then estimated.

The remaining phosphorus concentration (cP-rem) was determined in solution after stirring 60 mg L^{-1} of P in CaCl_2 10 mmol L^{-1} for one hour in a soil:solution ratio of 1:10 (Alvarez V et al., 2000). The organic matter level was estimated from the total C content (TOC) of organic compounds. The procedure consisted of determining the C content by oxidation with potassium dichromate ($\text{K}_2\text{Cr}_2\text{O}_7$) using the modified Walkley-Black procedure (Nelson and Sommers, 1996). Soil buffer pH (SMP) was determined in a 10:25:5 (w/v/v) soil (air-dried fine earth): CaCl_2 10 mmol L^{-1} :buffer solution ratio as proposed by van Raij et al. (1979).

After soil chemical analyses, the following properties were used for estimating the LR by the methods evaluated in this study: $\text{pH}(\text{H}_2\text{O})$, M^{x+} , V, T, Ca^{2+} and Mg^{2+} , HAI, and OM.

Lime requirement methods

Seven LR methods were selected from literature and evaluated in this study (Table 2). These methods were selected according to their reliability and traditional use in different regions of Brazil for estimating LR. Selected LR methods comprised the exchangeable acidity (EA) method (Cate and Nelson, 1965), the base saturation (BSAT) method (van Raij et al., 1996), the 4th (MG4A) (Lopes and Guimarães, 1989) and 5th (MG5A) (Alvarez V and Ribeiro, 1999) Approximations to the Minas Gerais State, the Shoemaker-McLean-Pratt (SMP) buffer (Shoemaker et al., 1961), the potential acidity (PA) method (Teixeira et al., 2014), and the soil pH-organic matter (pHOM) method (Defelipo et al., 1972). In particular, the SMP was slightly altered from the one first proposed by Shoemaker et al. (1961) in which the ratio soil: CaCl_2 10 mmol L^{-1} :buffer solution of 10:25:5 (w/v/v) as proposed by van Raij et al. (1979) was used.

The predictions of LR by the MG4A and MG5A methods were determined based on the nutritional requirements of corn (*Zea mays* L.): desired optimum base saturation ($\text{V}_2 = 50 \%$), Ca^{2+} and Mg^{2+} crop requirements ($\text{X} = 2 \text{ cmol}_c \text{ dm}^{-3}$), and maximum exchangeable acidity saturation tolerated by the crop ($\text{m}_t = 15 \%$) (Alvarez V and Ribeiro, 1999). The corn crop

Table 2. Description of traditional methods to determine the lime requirement (LR) used in the lime incubation study

Method	LR equations ⁽¹⁾	Reference
EA ⁽²⁾	$LR = 1.5 M^{x+}$	Cate and Nelson (1965)
BSAT ⁽³⁾	$LR = (V_2 - V_1) T/100$	van Raij et al. (1996)
MG4A ⁽⁴⁾	$LR = Y M^{x+} + [X - (Ca^{2+} + Mg^{2+})]$	Lopes and Guimarães (1989)
MG5A ⁽⁵⁾	$LR = Y [M^{x+} - (m_t t/100)] + [X - (Ca^{2+} + Mg^{2+})]$	Alvarez V and Ribeiro (1999)
SMP ⁽⁶⁾	-	van Raij et al. (1979)
PA ⁽⁷⁾	$LR = -0.086 + 0.7557 HAI$	Teixeira et al. (2014)
pHOM ⁽⁸⁾	$LR = 0.16 (6 - pH) OM$	Defelipo et al. (1972)

⁽¹⁾ LR expressed as $t\ ha^{-1}$ of pure $CaCO_3$ or limestone with total relative neutralizing power 100 %. ⁽²⁾ EA: exchangeable acidity; M^{x+} : soil exchangeable acidity ($cmol_c\ dm^{-3}$). ⁽³⁾ BSAT: base saturation; V_1 : existing soil base saturation (%); V_2 : desired soil base saturation (%); T: cation exchange capacity at pH 7.0. ⁽⁴⁾ MG4A and ⁽⁵⁾ MG5A: 4th and 5th Approximation to the Minas Gerais State, respectively; Y: variable as a function of the soil pH buffering capacity; X: variable as a function of the Ca^{2+} and Mg^{2+} requirement of the crop; m_t : maximum M^{x+} saturation tolerated by the crop (%); t: effective cation exchange capacity at the soil's original pH ($cmol_c\ dm^{-3}$). ⁽⁶⁾ SMP: Shoemaker-McLean-Pratt buffer. ⁽⁷⁾ PA: potential acidity; HAI: soil potential acidity ($cmol_c\ dm^{-3}$). ⁽⁸⁾ pHOM: soil pH-organic matter; OM: soil organic matter ($g\ kg^{-1}$).

requirements were used because this species was grown after the 60 days incubation to verify the effect of LR predictions on the yield responses in subsequent research.

Lime incubation study

The lime incubation study was conducted under greenhouse conditions for a period of 60 days. The treatments derived from a factorial combination ($22 \times (1 + 7 + 2)$) of 22 soils and 10 LR rates, which comprised one control treatment (without lime), seven rates estimated by different traditional LR methods, and two additional rates chosen to have well-spaced rates along the response curve. Treatments were laid out in a randomized complete block design, with four replicates.

Air-dried soil samples ($0.5\ dm^3$) sieved to a size fraction smaller than 2 mm were placed into plastic bags and mixed with the LR rates. The liming material consisted of a mixture of reagent-grade $CaCO_3$ (100 % $CaCO_3$ equivalent) and dolomitic limestone (34 % CaO and 13 % MgO , 92 % of total relative neutralizing power) to have a 4:1 molar ratio of Ca:Mg. Treated soil samples were moistened to 80 % of the field capacity with distilled water, as previously estimated by the moisture equivalent method (Ruiz et al., 2003). During the 60-days incubation period at room temperature, the soil moisture was kept near 80 % of the field capacity by adding distilled water at regular intervals, and the soils were thoroughly mixed. Daily, the plastic bags were opened to allow the release of evolved CO_2 .

Soil pH at a 1:2.5 soil:water ratio was measured in five different treatments, including the control (0 lime) at 15, 30, and 45 days after beginning the incubation period to ensure the equilibrium pH was reached. At the end of the incubation period, when the pH of all soils have reached a relatively steady state, soil samples of all treatments were air-dried, ground to pass a 2-mm sieve, and reanalyzed for soil $pH(H_2O)$, M^{x+} , HAI, Ca^{2+} and Mg^{2+} levels using the procedures mentioned above.

Lime requirement from incubation and associated soil properties

The incubation with lime was used as a standard method to evaluate whether the selected traditional methods were suitably predicting LR for the soils used in this study. As such, soil $pH(H_2O)$ values (\hat{y}) measured at the end of the incubation period were plotted as a function of the ten lime rates (x , $t\ ha^{-1}$) to determine soil acidity neutralization curves using linear and curvilinear regression analysis. The equivalent amounts of lime needed to raise the soil $pH(H_2O)$ to 5.8 ($LR_{5.8}$) and 6.0 ($LR_{6.0}$) were then estimated from the soil

acidity neutralization curves for all 22 soils used in the incubation study. These pH values were selected based on the optimal range of pH (5.7 to 6.0) reported in the literature for most crops in Brazil (Sousa et al., 2007). The levels of M^{x+} , HAI, base saturation and Ca^{2+} and Mg^{2+} associated with $LR_{5.8}$ and $LR_{6.0}$ were then estimated.

Statistical analysis

The means of LR predicted from the seven traditional methods along with soil properties associated with $LR_{5.8}$ and $LR_{6.0}$ were compared using the Tukey test, at 5 % significance level ($p \leq 0.05$). Linear regression analyses were performed to establish the relationship between incubation LR and LR predicted by traditional methods, in which intercepts (β_0), slopes (β_1), and Pearson's correlation coefficients (r) were assessed. Predictions of LR were compared to the equality line ($x = y$) where the values would be exactly equal. The efficiency of the various methods at predicting LR relative to the standard incubation method (relative efficiency) was estimated. This was done by estimating LRs from each traditional method that was associated with incubation LRs ($LR_{5.8}$ and $LR_{6.0}$) using the linear regression models. Hence, the lower (or higher) the LR predicted from each recommendation method relative to the standard incubation method, the lower (or higher) its relative efficiency, which indicates how much the LR was under or overestimated by each method.

Further, the identity test (Leite and Oliveira, 2002) was employed to determine whether the LR predicted by the seven traditional methods (Y_j) and the LR predicted by the standard incubation method (Y_1) were identical (i.e., equivalent). In this test, the soil-lime incubation was considered as the reference method, while the seven traditional methods were evaluated as alternative methods. A combination of the statistic F [$F(H_0)$] as modified from Graybill (1976), the mean error test ($t\bar{e}$), and analysis of the Pearson's linear correlation coefficient ($r_{Y_1Y_j}$) was used. Thus, after fitting a linear regression equation ($Y_j = \beta_0 + \beta_1 Y_1 + e_i$), the identity between methods ($Y_j = Y_1$) was verified when: (i) $F(H_0)$ is not significant: $F(H_0) < F_\alpha(2, n-2 \text{ d.f.})$; (ii) the mean error is statistically equal to zero: $\bar{e} = 0$ (non-significant); and (iii) the Pearson's linear correlation coefficient is significant and greater than $(1 - |\bar{e}|)$: $r_{Y_1Y_j} \geq (1 - |\bar{e}|)$.

RESULTS

Initial soil properties

The soil samples used in the lime incubation study encompassed four major orders, according to the USDA Soil Taxonomy (Table 1). Oxisols ($n = 17$) were the most common, followed by Entisol ($n = 2$), Ultisol ($n = 2$), and Inceptisol ($n = 1$). Descriptive analyses of the measured soil properties are given in table 3. Large variability of textures was observed between soils, ranging from sandy to loamy and clayey classes with 73 % of the soils classified into the clay-size fraction.

Soils were also heterogenous for the chemical properties, as such the active acidity ranged from very strongly acid to strongly acid [$pH(H_2O)$: 4.12 - 5.26]. The exchangeable acidity (M^{x+} : 0.21 - 1.98 $cmol_c \text{ dm}^{-3}$) and potential acidity (HAI: 1.68 - 13.06 $cmol_c \text{ dm}^{-3}$) ranged from very low to very high levels. All soils had low base saturation (V up to 35 %) and about half of the soils (54 %) had high exchangeable acidity saturation (m up to 96 %). The remaining P concentrations (5.44 - 60 $mg \text{ L}^{-1}$) and OM levels (3.78 - 79.35 $g \text{ kg}^{-1}$) varied greatly, revealing the wide range of buffering capacities of the soils, with highly buffered soils predominating. Hence, a wide range in LR predictions is expected to occur, implying that the soils used were representative for the research.

Predictions of lime requirement

The average values of LR predicted from incubation for the 22 soils to achieve pH 5.8 and 6.0 were 2.97 and 4.07 $t \text{ ha}^{-1}$, respectively, as indicated by the dashed and solid lines

Table 3. Descriptive statistics for soil texture and chemical properties of the soils used in the lime incubation study

Soil properties ⁽¹⁾	Mean	Lowest	Highest	CV ⁽²⁾
Soil texture (%)				
Sand	33.14	4.90	92.60	76
Silt	7.42	0.60	23.00	73
Clay	59.44	5.40	88.40	40
Chemical				
pH(H ₂ O)	4.63	4.12	5.26	8
pH(SMP)	5.72	4.82	6.78	7
Exchangeable acidity (cmol _c dm ⁻³)	0.87	0.21	1.98	58
Potential acidity (cmol _c dm ⁻³)	6.63	1.68	13.06	42
Ca ²⁺ (cmol _c dm ⁻³)	0.45	0.02	1.66	94
Mg ²⁺ (cmol _c dm ⁻³)	0.20	0.00	0.87	118
Sum of bases (cmol _c dm ⁻³)	0.77	0.04	2.04	83
T (cmol _c dm ⁻³)	7.40	1.72	14.06	39
t (cmol _c dm ⁻³)	1.64	0.50	3.02	43
Base saturation (%)	10.64	0.77	34.53	84
Exchangeable acidity saturation (%)	57.41	9.60	95.77	46
Remaining P (mg L ⁻¹)	18.51	5.44	60.00	70
Organic matter (g kg ⁻¹)	41.87	3.78	79.35	50

⁽¹⁾ T: cation exchange capacity at pH 7.0; t: effective cation exchange capacity; ⁽²⁾ CV: coefficient of variation in %.

in figure 2. The ranges of LR predictions from incubation, however, were substantially large, varying from 0.57 to 6.62 t ha⁻¹ to achieve pH 5.8, and from 0.77 to 8.72 t ha⁻¹ to achieve pH 6.0.

Wide ranges of LR, on average, were also found in predictions by the traditional methods, which varied from 1.31 to 9.54 t ha⁻¹ across soils (Figure 2). Irrespective of soils, the pHOM method predicted the highest LR (mean: 9.54 t ha⁻¹), which was 7.3-fold larger than the lowest LR predicted by the EA method (mean: 1.31 t ha⁻¹). Predictions of LR by the other traditional methods were intermediate, which increased in the following order: BSAT (mean: 2.93 t ha⁻¹) ≈ MG5A (mean: 2.78 t ha⁻¹) ≤ MG4A (mean: 3.33 t ha⁻¹) ≈ SMP (4.21 t ha⁻¹) ≤ PA (4.93 t ha⁻¹).

In comparison to the incubation LR, most traditional methods predicted average LR that closely approximated the incubation LR to achieve either pH 5.8 or 6.0. The exceptions were the EA method that predicted LR 44 and 32 % lower and the pHOM method that predicted LR 321 and 234 % higher than those determined from incubation to attain pH 5.8 and 6.0, respectively. In turn, average LR predictions from the BSAT, MG5A, and MG4A methods corresponded to 98, 93, and 112 % of that required from incubation to achieve pH 5.8, respectively. The SMP and PA methods predicted 103 and 121 % of the incubation LR to achieve pH 6.0, respectively.

Effects of lime requirement on soil properties

Soil acidity-related characteristics analyzed after the 60 days incubation period for the 22 soils were also in a diverse range of values as per criteria proposed by Alvarez V et al. (1999) (Figure 3). When LR_s, as predicted by the seven traditional methods, were applied, soil properties ranged on average from medium to weak active acidity [pH(H₂O): 5.45 to 6.55], very low exchangeable acidity (M^{x+}: 0.01 to 0.19 cmol_c dm⁻³), low to high potential acidity (HA: 1.68 to 5.45 cmol_c dm⁻³) and low

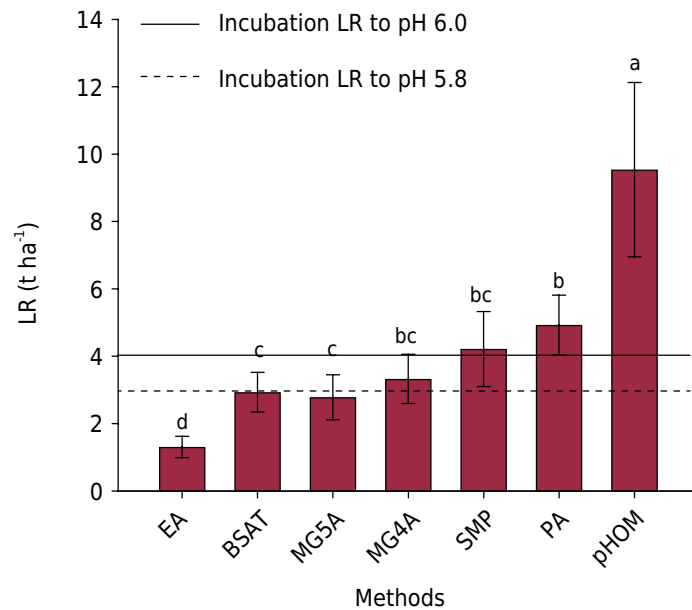


Figure 2. Lime requirement (LR) determined from the exchangeable acidity (EA), base saturation (BSAT), 5th Approximation to Minas Gerais State (MG) (MG5A), 4th Approximation to MG (MG4A), Shoemaker-McLean-Pratt buffer (SMP), potential acidity (PA), and soil pH-organic matter (pHOM) methods. Data plotted as means of the 22 soils used in the incubation study. Dashed and solid lines indicate the mean LR determined from incubation to raise soil pH to 5.8 and 6.0, respectively. Means followed by the same letter are not significantly different ($p > 0.05$, Tukey test). Error bars represent 95 % confidence interval.

to good base saturation (V: 22 to 71 %), and Ca^{2+} (1.0 to 3.72 $\text{cmol}_c \text{dm}^{-3}$) and Mg^{2+} (0.43 to 0.95 $\text{cmol}_c \text{dm}^{-3}$) levels. In turn, the application of LR predicted from incubation to achieve either pH 5.8 or 6.0 would result in the following soil properties on average, as indicated by the dashed and solid lines in figure 3: very low $\text{M}^{\text{x}+}$ (0.07 to 0.03 $\text{cmol}_c \text{dm}^{-3}$), medium HAI (4.33 to 3.68 $\text{cmol}_c \text{dm}^{-3}$), low to medium V (36 to 45 %), and medium Ca^{2+} (1.84 to 2.23 $\text{cmol}_c \text{dm}^{-3}$) and Mg^{2+} (0.68 to 0.80 $\text{cmol}_c \text{dm}^{-3}$) levels.

On average across soils, the lowest LR predicted by the EA method (mean: 1.31 t ha^{-1}) was insufficient to raise the soil pH above 5.45 (Figure 3a), which was lower than the desired target values of 5.8 and 6.0 considered in this study. As a result, the highest values of $\text{M}^{\text{x}+}$ (mean: 0.19 $\text{cmol}_c \text{dm}^{-3}$) and HAI (mean: 5.45 $\text{cmol}_c \text{dm}^{-3}$) as well as the lowest V (mean: 22 %) and Ca^{2+} (mean: 1.0 $\text{cmol}_c \text{dm}^{-3}$) and Mg^{2+} (mean: 0.43 $\text{cmol}_c \text{dm}^{-3}$) levels were achieved when LR was predicted by the EA method (Figures 3b and 3f). In contrast, the highest LR predicted by the pHOM method (mean: 9.54 t ha^{-1}) raised the soil pH to the highest values (mean: 6.55), resulting in the lowest $\text{M}^{\text{x}+}$ (mean: 0.01 $\text{cmol}_c \text{dm}^{-3}$) and HAI (mean: 1.68 $\text{cmol}_c \text{dm}^{-3}$) as well as the highest V (mean: 71 %) and exchangeable Ca^{2+} (mean: 3.72 $\text{cmol}_c \text{dm}^{-3}$) (Figures 3a and 3f).

Not surprisingly, the close predictions of LR by the BSAT (mean: 2.93 t ha^{-1}) and MG5A (mean: 2.78 t ha^{-1}) methods also resulted in quite close values of soil pH (means: 5.73 and 5.82), $\text{M}^{\text{x}+}$ (mean: 0.06 $\text{cmol}_c \text{dm}^{-3}$), HAI (means: 4.42 and 4.47 $\text{cmol}_c \text{dm}^{-3}$), V (means: 37 and 39 %), and exchangeable Ca^{2+} (means: 1.88 and 1.78 $\text{cmol}_c \text{dm}^{-3}$) and Mg^{2+} (means: 0.66 and 0.65 $\text{cmol}_c \text{dm}^{-3}$) (Figures 3a and 3f). The MG4A (mean: 3.33 t ha^{-1}) and SMP (mean: 4.21 t ha^{-1}) methods resulted in similar average values of soil pH (means: 5.95 and 5.96) and $\text{M}^{\text{x}+}$ (mean: 0.03 $\text{cmol}_c \text{dm}^{-3}$) (Figures 3a and 3b) as well as Ca^{2+} (means: 2.01 and 2.37 $\text{cmol}_c \text{dm}^{-3}$) and Mg^{2+} (means: 0.71 and 0.82 $\text{cmol}_c \text{dm}^{-3}$) (Figures 3e and 3f), though their LR predictions were slightly different. The values of HAI (means: 4.10 and 3.64 $\text{cmol}_c \text{dm}^{-3}$) and V (means: 43 and 45 %) achieved when LRs were predicted by these methods were also similar, with little but significant differences (Figures 3c and 3d).

The PA method, which predicted the second highest LR for all but 5 soil samples (mean: 4.93 t ha⁻¹), resulted in the second-highest average values of soil pH (mean: 6.17), V (mean: 53 %), and Ca²⁺ (mean: 2.66 cmol_c dm⁻³) (Figures 3a, 3d, and 3e) as well as the second-lowest HAI (mean: 3.20 cmol_c dm⁻³) (Figure 3c). Levels of M^{x+} (mean: 0.01 cmol_c dm⁻³) and Mg²⁺ (mean: 0.88 cmol_c dm⁻³) achieved by the PA method were not significantly different from those achieved when LR was predicted by the pHOM method (mean M^{x+}: 0.01 cmol_c dm⁻³ and mean Mg²⁺: 0.95 cmol_c dm⁻³) (Figures 3b and 3f).

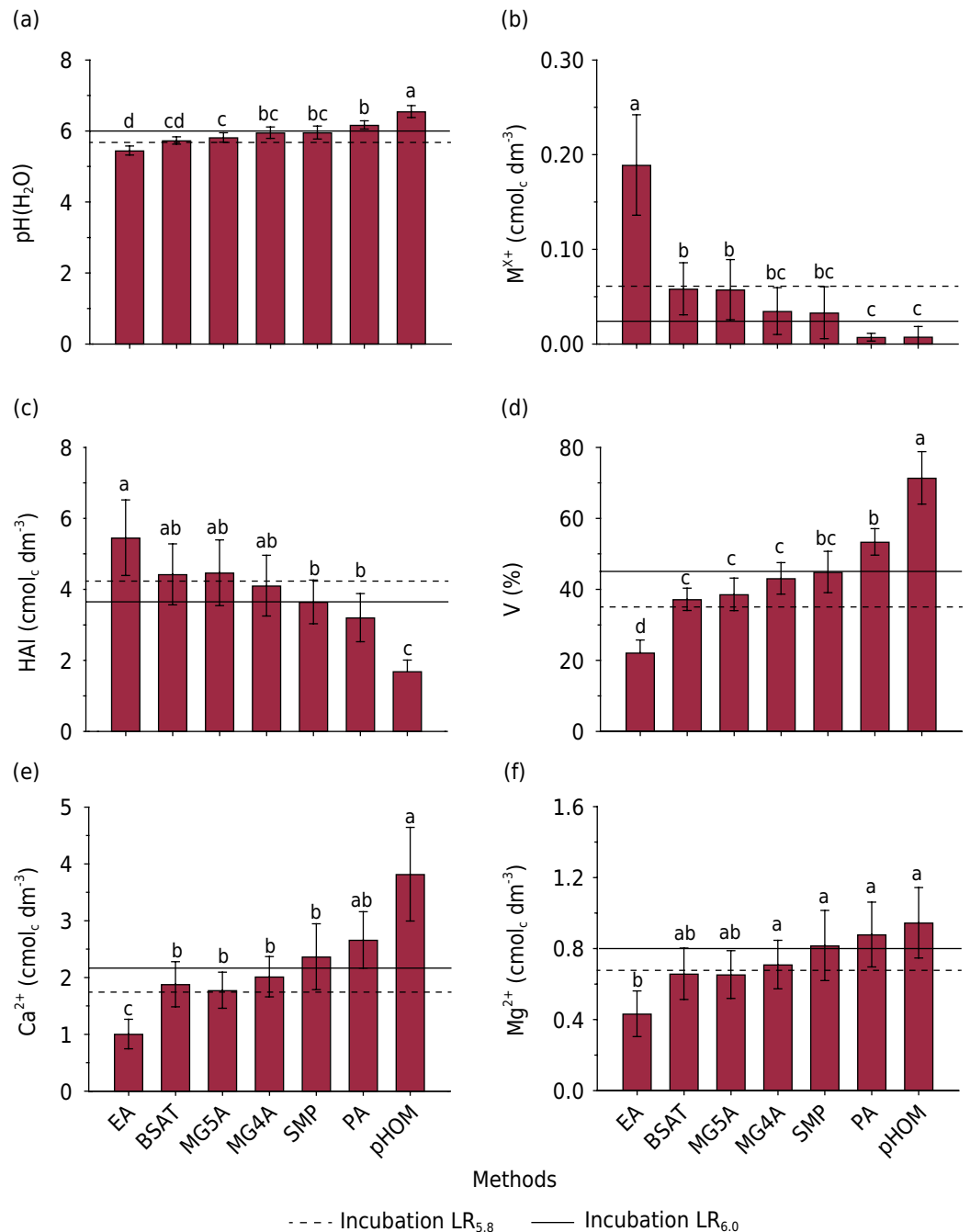


Figure 3. Soil pH (a); exchangeable acidity (b); potential acidity (c); base saturation percentage (d), exchangeable calcium (e), and exchangeable magnesium (f) determined from the exchangeable acidity (EA), base saturation (BSAT), 5th (MG5A) and 4th (MG4A) Approximation to the Minas Gerais State, Shoemaker-McLean-Pratt buffer (SMP), potential acidity (PA), and soil pH-organic matter (pHOM) methods. Data plotted as means of the 22 soils used in the incubation study. Dashed and solid lines indicate the respective property associated with LR determined from incubation to raise soil pH to 5.8 and 6.0, respectively. Means followed by the same letter are not significantly different (p < 0.05, Tukey test). Error bars represent 95 % confidence interval.

Relationships between lime requirement prediction methods

The relationships between LR predicted from incubation and those predicted by the seven traditional methods are shown in figure 4, and the linear regression coefficients (intercept, slope, and correlation coefficient) derived from these relationships along with the relative efficiency of the various methods are given in table 4. Lime requirements predicted by the traditional methods and LRs predicted from incubation were significantly and positively correlated ($p < 0.05$).

The highest correlation coefficients (r) were found between incubation LR to attain either pH 5.8 or 6.0 and LR predicted by the BSAT (0.73** and 0.79**), SMP (0.67** and 0.74**), PA (0.76** and 0.83**), and pHOM (0.71* and 0.76**) methods. Conversely, the lowest r values were obtained between incubation LR to achieve either pH 5.8 or 6.0 and LRs predicted by the EA (0.50* and 0.58*), MG5A (0.50* and 0.54*), and MG4A (0.59* and 0.64*) methods. Despite the significant correlations, all methods tended to either under or overestimate LR when compared with the actual LR predicted from incubation, as indicated by most of the data being lower or higher than the 1:1 line (intercept and slope different from zero and one, respectively) in figure 4.

The EA method underestimated the LR to attain both pH 5.8 and 6.0, predicting about 53 and 37 % of the amount of lime predicted from incubation, respectively, as indicated by its relative efficiency (Table 4). The same behavior was observed for the BSAT method, which underestimated by 85.47 % ($\beta_1 = 1.17$ **) the LR to attain pH of 6.0, compared to the incubation method. The opposite was found for the MG5A, MG4A, SMP, PA, and pHOM methods, which overestimated the LR to achieve both pH values, predicting about 47, 45, 58, 41, and 81 % more LR to attain pH 5.8, and 29, 24, 42, 22, and 75 % more LR to achieve pH 6.0, respectively, when compared to those predicted from incubation (Table 4). The BSAT method also overestimated LR and predicted 16 % ($\beta_1 = 0.84$ **) more lime to a target pH of 5.8.

The efficiency of traditional methods at predicting LR relative to the standard incubation method ranged from 37.71 to 352.82 % and ranked in the following order: pHOM (265.43-352.82 %) > PA (120.58-165.28 %) > SMP (114.73-156.58 %) > MG4A (89.50-123.28 %) > MG5A (81.98-109.70 %) > BSAT (73.44-101.52 %) > EA (37.71-53.17 %) (Table 4). Higher efficiencies were shown for LR predictions to attain pH 5.8.

Identity between lime requirement methods

From the results of the F -test ($H_0: \beta' = [0 \ 1]$), the intercept (β_0) and slope (β_1) were not significantly ($p < 0.05$) different from 0 and 1, respectively, only between LR_{5.8} and MG4A and LR_{6.0} and SMP (Table 5). Therefore, the MG4A and SMP methods predicted LR very close to those estimated from incubation to attain pH 5.8 and 6.0, respectively. As regards the t -test ($H_0: \bar{e} = 0$), the errors in LR predictions were randomly distributed ($p < 0.05$) between LR_{5.8} and BSAT, LR_{5.8}, and MG5A as well as between LR_{6.0} and MG5A and LR_{6.0} and MG4A. However, most of the traditional methods estimated LR differing systematically from those estimated by the incubation method. In the coefficient correlation analysis, correlations between LR predictions by traditional methods and the standard incubation method were sufficiently high ($r_{Y_1Y_j} \geq (1 - |\bar{e}|)$) in half of the relationships, whereas the other half exhibited high dispersion in LR predictions.

As none of the relationships between incubation LR (Y_1) and LR predicted by the alternative methods (Y_j) had the three assumptions [$\beta_0 = 0$ and $\beta_1 = 1$, $\bar{e} = 0$, $r_{Y_1Y_j} \geq (1 - |\bar{e}|)$] simultaneously satisfied, the results of the identity test (Leite and Oliveira, 2002) lead to the conclusion that all traditional methods estimated LR significantly different from the standard incubation method. Hence, no traditional LR method was identical to the standard incubation method.

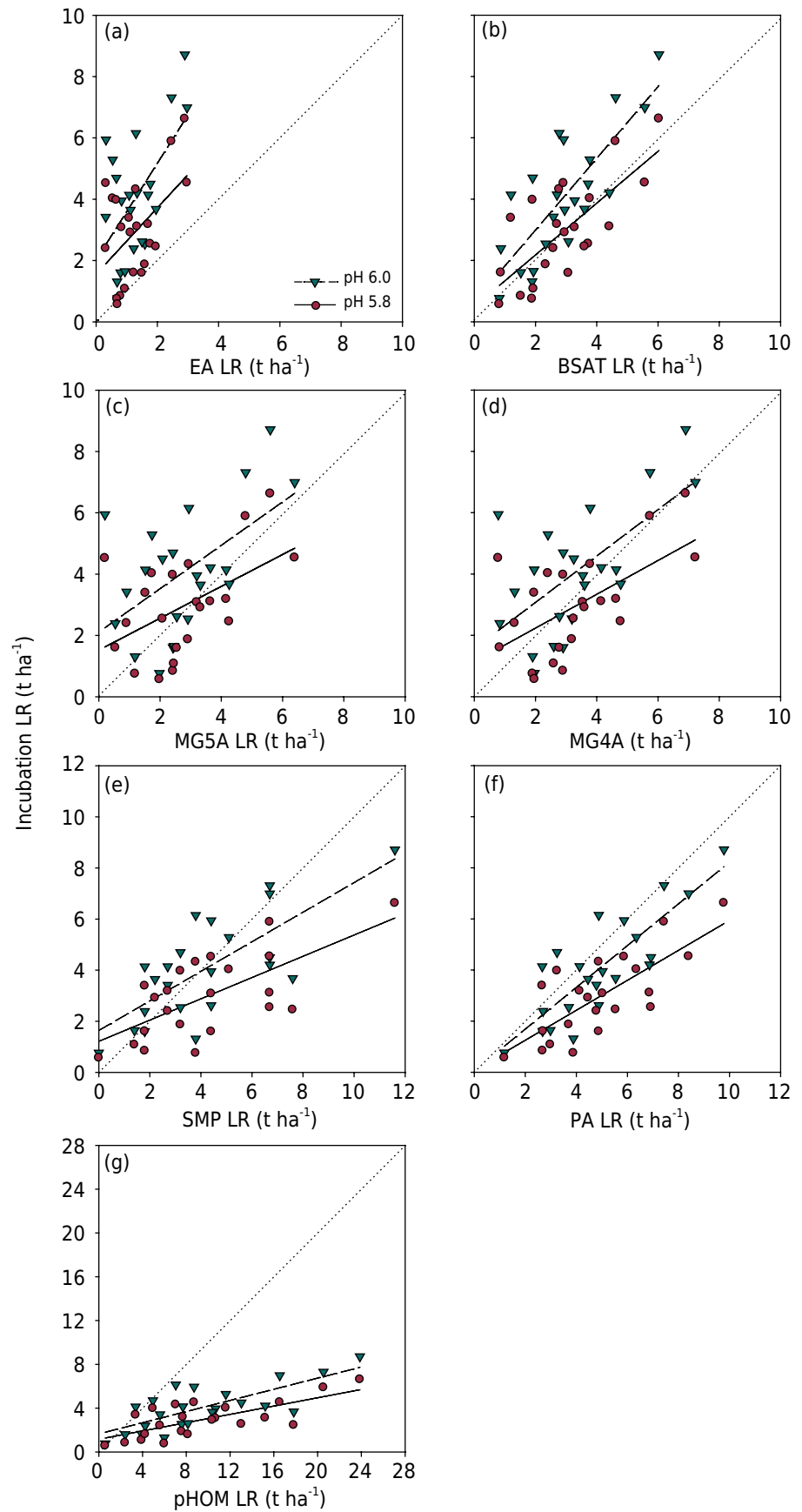


Figure 4. Relationships between lime requirement (LR) determined from the standard incubation method to raise soil pH to 5.8 and 6.0 and LR determined from the (a) exchangeable acidity (EA), (b) base saturation (BSAT), (c) 5th Approximation to the Minas Gerais State (MG) (MG5A), (d) 4th Approximation to MG (MG4A), (e) Shoemaker-McLean-Pratt buffer (SMP), (f) potential acidity (PA), and (g) soil pH-organic matter (pHOM) methods. The dotted line indicates the 1:1 ratio between methods. Regression parameters are shown in table 4.

Table 4. Regression parameters of the relationships between lime requirement (LR) determined from the standard incubation method to raise soil pH to target values of 5.8 and 6.0 (\hat{y} , t ha⁻¹) and LR determined by traditional methods (x , t ha⁻¹), as well as the corresponded relative efficiency (RE) of each prediction method

Traditional method ⁽¹⁾	Target pH	t ha ⁻¹		r	RE ⁽²⁾ %
		Intercept	Slope		
EA	5.8	1.55	1.08*	0.50*	53.17
	6.0	2.03	1.58*	0.58*	37.71
BSAT	5.8	0.49	0.84**	0.73**	101.52
	6.0	0.65	1.17**	0.79**	73.44
MG5A	5.8	1.52	0.53*	0.50*	109.70
	6.0	2.11	0.71*	0.54*	81.98
MG4A	5.8	1.13	0.55*	0.59*	123.28
	6.0	1.56	0.76*	0.64*	89.50
SMP	5.8	1.21	0.42**	0.67**	156.58
	6.0	1.64	0.58**	0.74**	114.73
PA	5.8	0.08	0.59**	0.76**	165.28
	6.0	0.05	0.82**	0.83**	120.58
pHOM	5.8	1.16	0.19*	0.71*	352.82
	6.0	1.65	0.25**	0.76**	265.43

⁽¹⁾ EA: exchangeable acidity; BSAT: base saturation; MG5A and MG4A: 5th and 4th Approximation to the Minas Gerais State, respectively; SMP: Shoemaker-McLean-Pratt buffer; PA: potential acidity; pHOM: soil pH-organic matter. *: 0.05 > p ≥ 0.01. **: p < 0.01. ⁽²⁾ RE: relative efficiency, which means the efficiency of traditional methods at predicting LR relative to the standard incubation method.

Table 5. Summary of the statistical procedure to test the identity between lime requirement (LR) determined from the standard incubation method to raise soil pH to target values of 5.8 and 6.0, and LR determined from the alternative traditional methods

Standard (Y ₁)	Method		target pH	F(H ₀) ⁽²⁾	t \bar{e} ⁽³⁾	r _{Y1Yj} ≥ (1 - \bar{e})	Conclusion ⁽⁴⁾
	Alternative (Y _j) ⁽¹⁾						
Incubation	EA	5.8	104.37*	6.34*	No	Y _j ≠ Y ₁	
		6.0	281.30*	14.02*	Yes	Y _j ≠ Y ₁	
Incubation	BSAT	5.8	4.09*	1.45 ^{ns}	No	Y _j ≠ Y ₁	
		6.0	31.46*	3.31*	No	Y _j ≠ Y ₁	
Incubation	MG5A	5.8	3.89*	1.16 ^{ns}	No	Y _j ≠ Y ₁	
		6.0	18.17*	1.44 ^{ns}	No	Y _j ≠ Y ₁	
Incubation	MG4A	5.8	2.56 ^{ns}	2.23*	Yes	Y _j ≠ Y ₁	
		6.0	8.41*	0.24 ^{ns}	No	Y _j ≠ Y ₁	
Incubation	SMP	5.8	4.37*	2.55*	Yes	Y _j ≠ Y ₁	
		6.0	0.11 ^{ns}	0.52 ^{ns}	No	Y _j ≠ Y ₁	
Incubation	PA	5.8	21.38*	4.76*	Yes	Y _j ≠ Y ₁	
		6.0	6.33*	3.30*	Yes	Y _j ≠ Y ₁	
Incubation	pHOM	5.8	28.64*	6.32*	Yes	Y _j ≠ Y ₁	
		6.0	23.68*	5.84*	Yes	Y _j ≠ Y ₁	

⁽¹⁾ EA: exchangeable acidity; BSAT: base saturation; MG5A and MG4A: 5th and 4th Approximation to the Minas Gerais State, respectively; SMP: Shoemaker-McLean-Pratt buffer; PA: potential acidity; pHOM: soil pH-organic matter. ⁽²⁾ ns and *: the hypothesis H₀: β' = [0 1] (β₀ = 0 and β₁ = 1, simultaneously) is accepted and rejected, respectively, at 0.05 probability level by the F-test modified from Graybill (1976). ⁽³⁾ ns and *: the hypothesis H₀: \bar{e} = 0 (differences between the standard method and the alternative method are casual) is accepted and rejected, respectively, at 0.05 probability level by the t-test for the medium error. ⁽⁴⁾ According to the identity test (Leite and Oliveira, 2002).

DISCUSSION

The variation in LR obtained by either incubation or traditional methods (Figure 2) has resulted from the large variability in the chemical and physical properties of the soils

used in this study (Table 3). This is indicative that the various soil types occurring in Minas Gerais were properly selected for this research, in addition, to highlight the need of methods for suitably predicting LR for a diverse range of soils. Regardless of the method used for predicting LR, liming changed the various components of soil acidity (pH, M^{x+} , and HAI) and increased the levels of basic cations (Ca^{2+} and Mg^{2+}) (Figure 3). However, lime-induced changes depended on the amount of lime applied, indicating that methods differed in their LR predictions. In fact, when comparing the predictions of LR, large discrepancies were found among methods, which is due to their different principles for correcting soil acidity (Figure 2).

Of the seven methods evaluated, both EA and pHOM predicted LR that most differed from the reference LR values to attain pH 5.8 and 6.0 (Figure 2). These methods use exchangeable acidity (M^{x+}) and organic matter (OM) as the basis for predicting LR to attain soil pH values of 5.5 and 6.0, respectively, where the levels of M^{x+} are nil, enabling maximum crop yields if plant nutrients are in adequate supply (Cunha et al., 2018; Rabel et al., 2018).

Exchangeable Al is the most toxic Al species to plants and the major component of soil exchangeable acidity (M^{x+} , extracted by the KCl method), which is accounted by the EA method. Nevertheless, the use of M^{x+} alone as a liming criterion does not provide an adequate prediction of LR since M^{x+} is not the only fraction contributing to the soil exchangeable acidity. As evidenced in previous studies, non-exchangeable forms of Al can be transformed into exchangeable and labile Al forms upon changes in soil pH (with fertilizers or liming input) and hence contribute to the KCl-exchangeable acidity, particularly in soils with high content of OM (Wen et al., 2014; Wang et al., 2015). Non-exchangeable Al forms are extracted with selective extraction methods (such as ammonium oxalate, and $CuCl_2$), and include amorphous Al, weak and strongly organically bound Al, and Al sorbed onto mineral surfaces (Heckman et al., 2013; Li and Johnson, 2016). An underestimation of exchangeable fractions of Al extracted by the standard KCl procedure was found by Yvanes-Giuliani et al. (2014) in soils containing significant levels of OM. These authors suggested that $CuCl_2$ is more suitable than KCl to estimate the fraction of exchangeable Al associated with OM. Since the EA method underestimates LR for not considering the non-exchangeable fractions of soil acidity, it undesirable to predict LR of moderately to strongly buffered soils, with significant amounts of OM, such as those used in this study.

Conversely, the pHOM method estimated LR that increased soil pH up to 6.55 on average across soils, which is excessively high for most crops. The pHOM method has overestimated LR because it was originally designed from soils containing medium to high levels of OM and hence inappropriate for poorly to moderately buffered soils ($OM < 40 \text{ g kg}^{-1}$), which comprised about half (54 %) of the soils used in this study. Recently, Caballero et al. (2019) found that the pHOM method overestimated LR in a range of Brazilian soils. Excessive LR prediction is an undesirable feature of any LR method, causing micronutrient deficiencies (Silva et al., 2015) and degradation in soil physical properties (Nunes et al., 2017), in addition to leading to profit losses. Since OM is a good predictor of the soil pH buffering capacity (Wang et al., 2015), its use as a liming criterion is expected to be a suitable approach. However, soils having OM varying from low to high levels are needed for developing an OM-based LR method, thus avoiding over predictions of LR.

The BSAT method predicted LR that increased V up to 37 % on average across soils, which was much lower than that desirable for the optimum yield of corn crop ($V_2 = 50 \%$). Base saturation lower than 50 % indicates the dominance of M^{x+} in the soil cation exchange capacity (T), which can result in toxicity to plant roots. This underestimation of LR (Figure 4b) may be attributed to two possible reasons. Firstly, because of limitations of using calcium acetate buffered at pH 7.0 to extract all HAI that is summed to the soil

basic cations to obtain the soil T, which is in turn used by the BSAT method to estimate LR. Since this buffered solution is poorly buffered in the pH range of 6.5-7.0, the levels of HAl and, consequently, the soil T will be underestimated, resulting in LR predictions lower than those actually required to raise V to the desired target value.

Further, the BSAT method underestimated LR likely because it is based on a linear relationship of V against soil pH. However, such a linear relationship is known to vary widely with soils, being valid only for soils containing similar base exchange minerals and OM levels (Nicolodi et al., 2008; Silva et al., 2008). In this study, V was not linearly related to soil pH (data not shown), since soil samples showed a mixed composition of permanent- and variable-charge clay minerals (data not shown) and contained a wide range of OM levels (3.78-79.35 g kg⁻¹) (Table 3). This is in line with other findings in literature where relationships between soil pH and V were non-linear, being described by either quadratic (Wang et al., 2019) or sigmoidal models (Kabala and Labaz, 2018; Wu and Liu, 2019). Our results also revealed that the BSAT method overestimated LR to attain pH 5.8 (Figure 4b; Table 4), mostly on soil samples containing T less than 7 cmol_c dm⁻³, which suggests a contradictory behavior of the method.

The MG5A method predicted average LR similar to that predicted by the BSAT method (Figure 2) and thus resulted essentially in the same changes in soil properties (Figure 3). In contrast to the BSAT, the MG5A method overestimated LR to attain both pH values as predicted from incubation, as well as the MG4A method (Figures 4c and 4d; Table 4). Both MG5A and MG4A methods were designed for predicting LR to neutralize the M^{x+} and meet the crop requirements of Ca²⁺ and Mg²⁺, differing from each other in the tolerance by crops to the maximum M^{x+} saturation taken into account by the former. In fact, MG5A was developed due to the concern that MG4A recommended too much lime, which could decrease micronutrient availability at high soil pH values. Since crops have different tolerances to M^{x+}, LR can be estimated to attain a target M^{x+} saturation rather than neutralize all the M^{x+} levels, which would result in higher rates of lime (Kamprath and Smyth, 2005). This explains why LR predictions by the MG5A were lower than those predicted by the MG4A in this study (Figure 2).

Noteworthy, the MG5A and MG4A methods behaved very similar at overestimating LR to attain pH 5.8 (109.70 and 123.28 %) and 6.0 (81.98 and 89.50 %) as indicated by their prediction efficiencies relative to the standard incubation method (Table 4), suggesting that both measured comparable forms of soil acidity or comparable pH buffering capacities. This is due to the similar principles on which they are based. Both MG5A and MG4A methods predicted LR as high as 250 % of the LR predicted from incubation for soils containing medium to very high levels of exchangeable acidity saturation (46 ≤ m ≤ 92 %) even at low levels of T (1.70 ≤ T ≤ 4.30 cmol_c dm⁻³). On the other hand, for soils containing low to very high levels of exchangeable acidity saturation (24 ≤ m ≤ 96 %), and T levels varying from medium to high (5.74 ≤ T ≤ 14.06 cmol_c dm⁻³), overestimations of LR by these methods were less pronounced, being up 95 % of the LR predicted from incubation. Hence, our results showed that MG5A and MG4A will likely overestimate LR to soils containing either low or high levels of T irrespective of the levels of M^{x+} saturation. Such methods were previously assessed by Guarçoni and Sobreira (2017) in a study using 600 soil samples from different sites of Minas Gerais State and provided over predictions of LR which agreed with our results.

The SMP and PA methods predicted similar average amounts of LR (Figure 2). They hence caused similar changes in soil acidity-related properties (Figure 3), especially because the principles behind both methods are quite similar. Such principles consist of reacting a buffered salt solution (i.e., SMP buffer for the SMP method and calcium acetate for the PA method) with soil to directly measure the proportion of soil acidity that must be neutralized by CaCO₃ to achieve a target pH (van Lierop, 1990). Such a soil acidity is regarded as the potential acidity, also known as residual (non-exchangeable)

acidity, which represents the buffering capacity of a soil. Nevertheless, both methods overestimated LR to attain either pH 5.8 or 6.0, particularly for soils containing medium to low M^{x+} levels ($<1.0 \text{ cmol}_c \text{ dm}^{-3}$) and high levels of HAI ($>5 \text{ cmol}_c \text{ dm}^{-3}$), which may be attributed to the fact that they were not calibrated on the soils used in this study.

In our lime-incubation study, we used LR predicted by the modified SMP buffer developed by van Raij et al. (1979) to reach a target pH of 6.0 on soils from São Paulo State. According to these authors, the modification of the SMP buffer provided lower LR as soil-buffer pH values decrease, allowing higher sensitivity for predicting LR of soils with low LR, as those from São Paulo. For determining LR based on the levels of HAI, we used the empirical equation proposed by Teixeira et al. (2014), which was calibrated for soils used for coffee production in the Minas Gerais State. In the original study (Teixeira et al., 2014), the authors highlighted the ability of the PA method to ensure an adequate supply of Ca^{2+} and Mg^{2+} to coffee plants ($\sum \text{Ca}^{2+} \text{ and } \text{Mg}^{2+} = 3.5 \text{ cmol}_c \text{ dm}^{-3}$) without exceeding the levels of HAI and hence avoiding overestimation of LR. It is therefore quite evident that both SMP and PA methods have a great potential of providing suitable recommendations of liming if properly calibrated on soils showing the same properties as those to which they will predict LR.

In this study, we used both the correlation-regression analysis and the identity test to compare seven methods traditionally used to predict LR in Brazil with the standard incubation method. The results from the former analysis showed that the traditional LR methods were moderate to strongly correlated (r : 0.50* - 0.83**) with the standard incubation method, even though they under or overestimated the LR to attain target pH values, as indicated by intercepts and slopes differing from 0 and 1. Since the Pearson's correlation coefficient indicates the linear association rather than equivalence between two methods, it is prone to erroneous conclusions in method comparison studies. For this reason, the identity test which enables the random error (bias) as well as the degree of association between two methods to be quantified was used in this study for verifying the statistical equivalence between the standard incubation method and each of the traditional methods for predicting LR.

The identity test indicated that no set of correlations between the standard incubation method to attain pH 5.8 or 6.0 (Y_1) and each of the seven alternative methods (Y_j) evaluated in this study had linear regression parameters [$\beta_0 = 0$ and $\beta_1 = 1$, $\bar{e} = 0$, $r_{Y_j Y_1} \geq (1 - |\bar{e}|)$] meeting the condition for identity (Table 5). However, the H_0 null hypothesis for the F -test ($\beta' = [0 \ 1]$) was accepted for comparisons between LR as predicted by the MG4A and SMP methods and those determined from incubation to a target pH of 5.8 and 6.0, respectively. These results indicate that MG4A and SMP methods provided sufficient predictions of LR for attaining pH values of 5.8 and 6.0, respectively. In other words, their predictions are similar (but not identical) to the LRs determined by incubation.

However, accepting such hypothesis does not imply that LR predictions from MG4A and SMP were equivalent to incubation LR for attaining pH 5.8 and 6.0, respectively. This is because systematic differences were found between LR predictions from the MG4A as well as other traditional methods and incubation, as indicated by the significant values of mean error evaluated by the t -test ($H_0: \bar{e} = 0$). These findings elucidate that the magnitude of differences in LR predictions is critical for confirming equivalence between two methods, even when the regression line shows $\beta_0 = 0$ and $\beta_1 = 1$ simultaneously. When evaluating the degree of association between the seven traditional methods and the standard incubation method for predicting LR, the condition $r_{Y_j Y_1} \geq (1 - |\bar{e}|)$ was not satisfied in half of the comparisons, including the comparison between $\text{LR}_{6.0}$ and SMP. A plausible explanation is that the mean errors were considerably large for LR predictions by these traditional methods, resulting in the occurrence of negative $(1 - |\bar{e}|)$ values and thus yielding biased predictions.

As we mentioned above, studies reported in literature often compare methods for predicting LR by using correlation analysis and no comparisons between LR methods using the identity test were found beyond those reported in the original study for developing such procedure (Leite and Oliveira, 2002). However, previous studies have shown the feasibility of using the identity test to compare results obtained by several analytical methods. For example, Milagres et al. (2007) used the identity test to compare the inductively coupled plasma optical emission spectrometry (ICP OES) and atomic absorption spectrometry techniques for measuring soil-extracted micronutrients by different extractors. Soares et al. (2010) compared the ICP OES and titrimetry techniques for determining exchangeable cations extracted from soil samples. Cunha et al. (2014) compared three chemical dispersants (i.e., NaOH, $(\text{NaPO}_3)_n + \text{Na}_2\text{CO}_3$, and $(\text{NaPO}_3)_n + \text{NaOH}$) for particle size analysis. More recently, Ferreira et al. (2018) identified differences in the CO_2 efflux measured in alkaline solution compared with the infrared gas analyzer method from soils under caatinga and pasture vegetation in Brazil.

Although the equivalence between any of the seven traditional LR methods and the standard incubation method was not demonstrated by the identity test in this study, we showed that this statistical procedure is preferred over the correlation coefficient, as very rigorous requirements must be fulfilled before establishing equivalence between two methods.

CONCLUSIONS

Average predictions of LR differed greatly among methods, and increased in the following order: EA < BSAT \approx MG5A \leq MG4A \approx SMP \leq PA < pHOM.

Suitable changes in soil pH, exchangeable acidity, potential acidity, base saturation, and Ca^{2+} and Mg^{2+} were achieved upon application of LR estimated by all methods except the EA and pHMO, which resulted in undesirable soil acidity properties.




All methods evaluated in this study were unable to predict LR for attaining target pH values of 5.8 and 6.0 as revealed by the identity test, even though they were moderate to strongly correlated with the standard incubation method as indicated by the correlation-regression analysis.



Further research should focus on the development of reliable methods for predicting LR to attain desired pH values and consequently maximize crop production on Brazilian soils.




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