



# Combining lime and organic amendments based on titratable alkalinity for efficient amelioration of acidic soils

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**Abstract.** Ameliorating soil acidity using a combination of lime and organic amendments (OAs) can be an alternative to lime alone, but determining the appropriate OA rates can be difficult. We developed a new method for calculating the combined application rate of lime and OAs (wheat straw, faba bean straw, blended poultry litter, biochar, and compost) that is based on the titratable alkalinity of OAs and the equilibrium lime buffer capacity ( $LBC_{eq}$ ) of acidic soils. The effect of calculated soil amendment rates on soil pH was validated at soil water contents of 60 %, 100 %, and 150 % of field capacity (FC). The soil used to develop and validate the method was a sandy loam with a soil pH in deionised water ( $pH_W$ ) of 4.84 and a soil pH in 0.01 M  $CaCl_2$  solution ( $pH_{Ca}$ ) of 4.21. The  $LBC_{eq}$  of the soil was  $1657 \text{ mg } CaCO_3 \text{ kg}^{-1} \text{ pH}^{-1}$  (where “ $CaCO_3 \text{ kg}^{-1} \text{ pH}^{-1}$ ” denotes the amount of lime required to raise the pH of 1 kg of soil by one unit). The titratable alkalinity of the OAs ranged from  $11.7 \text{ cmol H}_{eq}^+ \text{ kg}^{-1}$  for wheat straw to  $357 \text{ cmol H}_{eq}^+ \text{ kg}^{-1}$  for compost. At 60 % FC, faba bean and wheat straw amendment increased the soil  $pH_W$  to 6.48 and 6.42, respectively, but lower pH values were reached in soil amended with less biodegradable or resistant OAs (ROAs) (i.e. blended poultry litter, biochar, and compost). At 150 % FC, the two straws increased the soil  $pH_W$  to only 5.93 and 5.75, respectively, possibly due to slower decomposition under submerged conditions, resulting in limited alkalinity production, whereas amendment with ROAs produced  $pH_W$  values close to 6.5. With an increasing lime-equivalent value (LEV) of the OA, from  $5.8 \text{ g } CaCO_3 \text{ kg}^{-1}$  (wheat straw) to  $179 \text{ g } CaCO_3 \text{ kg}^{-1}$  (compost), the lime requirement to reach  $pH_W$  6.5 in lime–OA combinations decreased from 2.72 to  $0.09 \text{ g } CaCO_3 \text{ kg}^{-1}$ . The developed method was shown to be effective in determining the appropriate rates of OAs (with or without additional lime) for the management of acidic sandy loam soils in this study and highlights the importance of the soil water content with respect to its acid-neutralising effect.

## 1 Introduction

Acidic soils with a soil pH in deionised water ( $pH_W$ )  $\leq 5.5$  are considered harmful to sensitive plants. Soil acidification can be caused by various biogeochemical processes such as oxidation and acid-dissolution reactions, root exudates, solubilisation and hydrolysis of  $Al^{3+}$  which releases  $H^+$ , and leaching of cations ( $K^+$ ,  $Ca^{2+}$ ,  $Na^+$ ,  $Mg^{2+}$ ) (Brady and Weil, 2016; Goulding, 2016; Marschner and Noble, 2000;

Mosley et al., 2014). Soil acidification in agricultural systems can also be attributed to high inputs of urea and ammonium ( $NH_4^+$ )-containing fertilisers, as protons ( $H^+$ ) are generated during the nitrification of  $NH_4^+$  to nitrate ( $NO_3^-$ ) and by  $NH_4^+$  uptake by plants as well as by removal of alkalinity via the removal of plant material at harvest (Bolan et al., 2003; Hume et al., 2022; Iticha and Takele, 2019; Kunhikrishnan et al., 2016). Even a small decrease in soil pH can have a large impact on the nutrient availability, nitrogen fixation by soil

microbes, and sustainability of crop production (Kopittke et al., 2019).

Lime and, more recently, organic amendments (OAs) can be used to improve the productivity of acidic soils (Garbowski et al., 2023). Agricultural lime ( $\text{CaCO}_3$ ) produces alkalinity ( $\text{OH}^-$  and  $\text{CO}_3^{2-}/\text{HCO}_3^-$  ions) and neutralises soil acidity by reacting with  $\text{Al}^{3+}$  and  $\text{H}^+$  ions to form  $\text{Al}(\text{OH})_3$  precipitates and  $\text{H}_2\text{O}$ . OAs, on the other hand, can provide alkalinity via their surface organic functional groups (e.g. carboxylic,  $-\text{COO}^-$ ), the presence of solid and dissolved carbonates, and the release of organic and inorganic anions, which can neutralise or remove toxic ions such as  $\text{Al}^{3+}$ ,  $\text{Fe}^{3+}$ ,  $\text{Mn}^{2+}$ , and  $\text{H}^+$  from the soil by reacting, complexing, and binding (Brown et al., 2008; Larney and Angers, 2012; McCauley et al., 2009). Alkalinity production by OAs is related to the excess cation content and dissolution or decomposition rate of the amendments, which are in turn influenced by factors such as the chemical composition of the amendments and soil water content (Anderson et al., 2020; Cai et al., 2020; Védère et al., 2022). As a result, it is important to investigate the effect of the soil water content on the timing of OA application.

Determination of the optimum rates of lime and OAs is necessary for the efficient management of acidic soils. Lime requirement can be determined using various methods including titration, incubation, field experiments, standard buffer solutions, and predictive equations developed from measured soil properties (Aitken and Moody, 1994; Nelson and Su, 2010). Titration is considered the most effective approach for lime recommendations (Wang et al., 2015). It involves the addition of bases such as  $\text{Ca}(\text{OH})_2$  to acidic soils in a given soil-to-liquid ratio (e.g. 1 : 5 soil to deionised water or 0.01 M  $\text{CaCl}_2$  solution) followed by measurement of changes in soil pH and pH buffering capacity (pHBC) with time to predict the lime requirement of the soils (Barouchas et al., 2013; Liu et al., 2004; Thompson et al., 2010). Titration results are often validated by experiments to determine the equilibrium lime buffer capacity ( $\text{LBC}_{\text{eq}}$ ), i.e. a measure of the amount of soil acidity that must be neutralised by the addition of lime to raise the soil pH by one unit in order to calculate the lime requirements of acidic soils (Kissel et al., 2012).

The amounts of OAs required to neutralise soil acidity and achieve a desired pH can be calculated using the alkalinity of the amendments. Alkalinity is determined by titrating acidified OAs with a base to neutrality, using either ashed amendments, i.e. potential or “ash” alkalinity (Slattery et al., 1991), or air-dried amendments, i.e. titratable or available alkalinity (Feizi et al., 2017; Fidel et al., 2017; Singh et al., 2017). The ash alkalinity method overestimates the alkalinity or liming potential of OAs due to the loss of anions such as sulfur and chlorine during the ashing process (Noble et al., 1996), leading to the underestimation of the amounts of amendments required to neutralise soil acidity. The short-term acid-neutralising effect of OAs is better determined by

the available alkalinity, because potential alkalinity becomes only gradually available over a long period of time (Sakala et al., 2004).

Previous studies have shown that a combination of lime and OAs can generate more alkalinity than lime or OA alone (Butterly et al., 2021; Lauricella et al., 2021; Li et al., 2022). However, little effort has been made to develop the methods to calculate the application rates of lime combined with OAs needed to neutralise soil acidity and achieve the desired pH for plant growth. Previous research aimed at developing appropriate lime and OA combinations has been conducted in field trials, which involve mixing different rates of lime and OAs into acidic soils and determining the response of acidity and crop yield (Celestina et al., 2018; Khoi et al., 2010; Li et al., 2019). This approach is time-consuming and costly, and it is difficult to predict the soil acidity that can be neutralised. In addition, little information exists about the impact of an alkalinity-based mixture of OAs on soil pH. A laboratory method for determining lime and OA combinations based on titratable alkalinity could shorten the time required and improve the cost-effectiveness of soil acidity amelioration.

Therefore, the first aim of this study was to determine the lime requirement of acidic soil using titration and equilibration methods as well as to estimate the application rates of OAs and OA mixes based on their titratable alkalinity. The second aim was to develop a laboratory method to calculate the rates of lime and OA in combinations required to achieve a desired soil pH, based on the titratable alkalinity of the amendments and  $\text{LBC}_{\text{eq}}$  of the acidic soil. Finally, the third aim was to assess the effect of the soil water content on amelioration by incubating acidic soil with the amendments at different soil water contents.

## 2 Materials and methods

### 2.1 Collection of soil and amendments

The soil used in this study was collected from the 0–10 cm layer of the non-limed treatment from the acid soil management trial site at Sandilands on the Yorke Peninsula of South Australia. The site is located north-west of Adelaide ( $34^\circ 33' 14'' \text{ S}$ ,  $137^\circ 42' 14'' \text{ E}$ ). The long-term mean annual rainfall and temperature at the site were 409 mm and  $21.9^\circ \text{C}$ , respectively. Penlime Plus™ (Angaston lime, Penrice Quarry & Mineral, South Australia) with a neutralising value of 98 % was used as the lime source. The OAs used in this study were wheat straw, faba bean straw, blended poultry litter, biochar, and compost, which differ with respect to their decomposability. The two straws are more decomposable than the other OAs because they have not undergone decomposition. Due to its lower C/N ratio, faba bean straw is likely to be more readily decomposable than mature wheat straw. The blended poultry litter comprised equal proportions of poultry manure and sawdust/wood shavings. The biochar was made from

the pyrolysis (400 °C) of *Eucalyptus* species (sourced from Green Man Char Pty Ltd, Victoria, Australia), and the compost was prepared from a mixture of various organic wastes (sourced from Bunnings Pty Ltd, Australia). Blended poultry litter, biochar, and compost are partially decomposed, leaving few readily available compounds for decomposition after addition to soil and are, therefore, referred to as poorly decomposable or resistant amendments.

The soil and OAs were dried at 30 °C in a fan-forced oven. Prior to their use in the experiment, the oven-dried soil was crushed and sieved to pass through a 2 mm sieve size, and the dried OAs were ground and sieved to 0.25–2 mm.

## 2.2 Analysis of soil and amendments

Soil particle size distribution was determined using the hydrometer method (Bouyoucos, 1962). To measure gravimetric water content at field capacity, air-dried soil was weighed into a small container with a 10 cm diameter and 10 cm height, watered to saturation, covered at the top with plastic film to prevent evaporation, drained to a constant mass for 2 to 3 d, and reweighed. Soil pH was measured potentiometrically using a calibrated glass electrode in a soil / water (1 : 5) suspension (pH<sub>W</sub>) and a soil / 0.01 M CaCl<sub>2</sub> (1 : 5) suspension (pH<sub>Ca</sub>) (Rayment and Higginson, 1992). The pH and electrical conductivity (EC) of OAs were measured in a 1 : 10 amendment-to-water ratio (Singh et al., 2017). Exchangeable acidity was determined by extracting soil with 1 M KCl solution (Pansu and Gautheyrou, 2006). The cation exchange capacity (CEC) of the soil was determined using a colorimetric method after displacing cations with 1 M ammonium acetate (pH 7) and then extracting the ammonium ions with 1 M KCl (Carter and Gregorich, 2007). Total organic carbon (TOC) in the soil and OAs was determined using the Walkley and Black wet digestion method (Walkley and Black, 1934), whereas total N was determined using the Kjeldahl method (Bremner and Mulvaney, 1982). After digestion with concentrated nitric acid and hydrogen peroxide, the total concentrations of Fe and Al in soil as well as Ca, K, Mg, Na, P, and S in OAs were determined using inductively coupled plasma optical emission spectroscopy (ICP-OES). The excess cation content in the OAs was then calculated by subtracting anions (SO<sub>4</sub><sup>2-</sup> and H<sub>2</sub>PO<sub>4</sub><sup>-</sup>) from cations (Ca<sup>2+</sup>, Mg<sup>2+</sup>, K<sup>+</sup>, and Na<sup>+</sup>) (Tang and Yu, 1999).

## 2.3 Properties of soil and amendments

In the Australian Soil Classification (Isbell and NCST, 2021), the soil was categorised as a Chromosol, although it is classified as Lixisol in the World Reference Base for Soil Resources (WRB; FAO, 2015). The upper A horizon, which has a sandy loam texture, was used in this experiment (Table 1). The soil pH<sub>W</sub> and pH<sub>Ca</sub> measured in 1 : 5 soil-to-water or soil-to-0.01 M CaCl<sub>2</sub> solution ratios are 4.84 and 4.21, respectively. The exchangeable acidity of the soil was

**Table 1.** Basic properties of the soil used in this study (mean ± SD).

Parameters	Mean
Clay (%)	14.41±1.16
Silt (%)	10.67±2.31
Sand (%)	74.92±2
pH <sub>W</sub>	4.84±0.02
pH <sub>Ca</sub>	4.21±0.01
EA (cmol kg <sup>-1</sup> )	2.95±0.14
CEC (cmol kg <sup>-1</sup> )	21.21±1.62
TOC (%)	1.43±0.04
Total Al <sup>3+</sup> (g kg <sup>-1</sup> )	3.42±0.44
Total Fe <sup>2+</sup> (g kg <sup>-1</sup> )	6.78±0.25

SD: standard deviation; pH<sub>W</sub>: soil pH in deionised water; pH<sub>Ca</sub>: soil pH in 0.01 M CaCl<sub>2</sub> solution; EA: exchangeable acidity; CEC: cation exchange capacity; TOC: total organic carbon.

2.95 cmol kg<sup>-1</sup>. Based on the ratings of Hazelton and Murphy (2016), the soil has a moderate CEC (21.38 cmol kg<sup>-1</sup>), likely due to its TOC content (1.43 %) (Table 1).

The mean pH<sub>W</sub> of the OAs varied between 5.50 (wheat straw) and 9.75 (biochar) (Table 2). The EC of the amendments ranged from 0.55 dS m<sup>-1</sup> for faba bean straw to 3.22 dS m<sup>-1</sup> for compost. Total concentrations of cations were highest in compost (383 cmol kg<sup>-1</sup>) and lowest in wheat straw (53 cmol kg<sup>-1</sup>) (Table 2).

## 2.4 Determination of the lime requirement of acidic soil

### 2.4.1 Titration with calcium hydroxide

Titrations and pH measurements in this study were carried out using 1 : 5 soil-to-deionised water or soil-to-0.01 M CaCl<sub>2</sub> solution ratios. First, 5 g of dry soil was weighed into 50 mL polyethylene tubes in triplicate; 25 mL of deionised water or 25 mL of 0.01 M CaCl<sub>2</sub> solution was then added into the tubes. The initial soil pH was measured after 30 min of stirring and calibration of the pH meter with standard pH 7.0 and 4.0 buffers. Next, 0.5 mL aliquots of 0.022 M Ca(OH)<sub>2</sub> were added to the suspensions, continuously stirred, and pH measurements were taken at the end of each time interval. Based on Liu et al. (2004), the reaction time allowed between consecutive titrations to obtain constant pH measurements was 30 min. The titrations with incremental additions of 0.022 M Ca(OH)<sub>2</sub> were carried out while recording the cumulative volume of the 0.022 M Ca(OH)<sub>2</sub> added versus the corresponding soil pH. A digital titrator (Burette Digital Titrette<sup>®</sup> bottle-top burette, 50 mL capacity, Class A, Australian Scientific, Australia) was used for the titrations.

These data were used to plot a regression curve between incremental rates of equivalent CaCO<sub>3</sub> (Mg ha<sup>-1</sup>) consumed and the corresponding soil pH. The regression curve was used to derive the slope, which was then used to calculate

**Table 2.** Chemical properties of organic amendments used in this study ( $n = 3$ ). Different letters indicate significant differences ( $p \leq 0.05$ ).

Organic amendment	pH <sub>w</sub> (1 : 10)	EC (dS m <sup>-1</sup> ) (1 : 10)	cmol kg <sup>-1</sup>					TOC (g kg <sup>-1</sup> )	TN (g kg <sup>-1</sup> )	C/N	
			Na	K	Ca	Mg	P				S
Wheat straw	5.50 <sup>a</sup>	1.87 <sup>a</sup>	15.62 <sup>ad</sup>	28.89 <sup>a</sup>	11.28 <sup>a</sup>	6.68 <sup>a</sup>	2.65 <sup>a</sup>	6.37 <sup>a</sup>	453 <sup>a</sup>	7.30 <sup>a</sup>	63.15 <sup>a</sup>
Faba bean straw	6.96 <sup>b</sup>	0.55 <sup>b</sup>	3.12 <sup>b</sup>	7.17 <sup>b</sup>	69.46 <sup>b</sup>	13.52 <sup>ac</sup>	17.27 <sup>b</sup>	10.49 <sup>b</sup>	429 <sup>b</sup>	12.88 <sup>b</sup>	33.40 <sup>b</sup>
Blended poultry litter	7.88 <sup>c</sup>	1.39 <sup>c</sup>	12.20 <sup>a</sup>	15.49 <sup>c</sup>	168.95 <sup>c</sup>	41.33 <sup>b</sup>	24 <sup>c</sup>	27 <sup>c</sup>	256 <sup>c</sup>	17.09 <sup>c</sup>	15.09 <sup>cd</sup>
Biochar	9.75 <sup>d</sup>	0.91 <sup>d</sup>	25.03 <sup>c</sup>	15.74 <sup>c</sup>	108.82 <sup>d</sup>	22.16 <sup>c</sup>	10.82 <sup>d</sup>	6.98 <sup>a</sup>	180 <sup>d</sup>	9.63 <sup>a</sup>	18.96 <sup>c</sup>
Compost	8.12 <sup>e</sup>	3.22 <sup>e</sup>	17.24 <sup>d</sup>	29.94 <sup>a</sup>	87.67 <sup>e</sup>	327.15 <sup>d</sup>	41.49 <sup>e</sup>	37.12 <sup>d</sup>	160 <sup>d</sup>	20.18 <sup>c</sup>	7.95 <sup>d</sup>

EC: electrical conductivity; TOC: total organic carbon; TN: total nitrogen; C/N: carbon-to-nitrogen ratio.

the 30 min pHBC (pHBC<sub>30</sub>) of the acidic soil. The amount of base needed to neutralise H<sup>+</sup> (independent variable) was plotted on the  $x$  axis and the change in pH (dependent variable) was plotted on the  $y$  axis to generate the slope of the titration curve as follows:

$$\text{Slope} = \frac{\Delta Y}{\Delta X} = \frac{\Delta \text{pH}}{V_{\text{Ca(OH)}_2}} = \frac{\Delta \text{pH}}{\Delta \text{H}^+}, \quad (1)$$

where  $\Delta Y$  is the change in pH,  $\Delta X$  is the amount of base consumed or protons removed to neutralise acids,  $V_{\text{Ca(OH)}_2}$  is the volume of base consumed during titration (mL), and  $\Delta \text{H}^+$  is the amount (mmol kg<sup>-1</sup>) of H<sup>+</sup> removed during titration. The amount of protons ( $\Delta \text{H}^+$ ) neutralised during the titration is equivalent to the amount of Ca(OH)<sub>2</sub> consumed. From this model, the lime requirement (LR) of the acidic soil can be calculated as follows:

$$\text{LR} = \frac{\Delta \text{pH}}{\text{Slope}} = \Delta \text{pH} \times \text{pHBC}. \quad (2)$$

Given that the molecular weight of CaCO<sub>3</sub> is 100 g mol<sup>-1</sup>, the amount of Ca(OH)<sub>2</sub> consumed by the acidic soil, expressed as CaCO<sub>3</sub> equivalent (Mg ha<sup>-1</sup>), was calculated as follows:

$$\text{CaCO}_3 \text{ (Mg ha}^{-1}\text{)} = \frac{V \times M \times 100 \text{ g CaCO}_3 \text{ mol}^{-1} \times 10^{-9} \times \text{Wt (kg ha}^{-1}\text{)}}{S \text{ (kg)}}, \quad (3)$$

where  $V$  is the volume (mL) of 0.022 M Ca(OH)<sub>2</sub> consumed to raise the initial pH of the acidic soil to the target pH,  $M = 0.022$  is the molarity of Ca(OH)<sub>2</sub> (in mmol mL<sup>-1</sup>),  $10^{-9}$  is the conversion factor from CaCO<sub>3</sub> (mg mmol<sup>-1</sup>) to CaCO<sub>3</sub> (Mg mmol<sup>-1</sup>),  $S$  is the mass of soil used for the titration (0.005 kg), and  $\text{Wt}$  is the weight of soil per hectare which (in this case) was calculated as  $1.5 \times 10^6$  kg (assuming a liming depth of 0.1 m and soil bulk density of 1500 kg m<sup>-3</sup>). By using these known variables in Eq. (3), the CaCO<sub>3</sub> (Mg ha<sup>-1</sup>) equivalent can be simply calculated as  $V \times 0.66$ .

The pH buffer capacity (pHBC<sub>30</sub>), expressed in mmol H<sup>+</sup> (kg soil)<sup>-1</sup> pH<sup>-1</sup>, was calculated from the titration curve as the inverse of the slope of the linear regression between pH and the added base (Mg CaCO<sub>3</sub> ha<sup>-1</sup>)

(Shi et al., 2019; Thompson et al., 2010). The unit of pHBC derived from the slope was Mg CaCO<sub>3</sub> ha<sup>-1</sup> pH<sup>-1</sup> (where “CaCO<sub>3</sub> kg<sup>-1</sup> pH<sup>-1</sup>” denotes the amount of lime required to raise the pH of 1 kg of soil by one unit). This unit was converted to mg CaCO<sub>3</sub> kg<sup>-1</sup> pH<sup>-1</sup> and then to mmol H<sup>+</sup> kg<sup>-1</sup> pH<sup>-1</sup>.

#### 2.4.2 Equilibration experiment

An additional experiment was conducted to assess whether the 30 min titration time above was sufficient to complete the equilibrium exchange reaction between H<sup>+</sup> and 0.022 M Ca(OH)<sub>2</sub>. As there was no significant difference between the slopes of regression lines fitted for titrations in water and 0.01 M CaCl<sub>2</sub> ( $p$  value of 0.231 at  $\alpha = 0.05$ ), the equilibration experiment was only carried out using a 1 : 5 soil-to-deionised water ratio. The experiment consisted of unamended soil (control) and soils amended with the equivalent titration point (ETP) multiplied by 0.5, 0.75, 1, 1.25, and 1.5, respectively. The ETP is the final volume of 0.022 M Ca(OH)<sub>2</sub> solution consumed over a 30 min complete titration to achieve the target pH<sub>w</sub> of 6.5. These treatments were added in 50 mL polypropylene tubes with replicates of 5 g dried soil and 25 mL deionised water suspension. After stirring the mixture for 30 min, the initial 30 min soil pH was measured, which was used to calculate the lime buffer capacity (LBC<sub>30</sub>) for each rate of 0.022 M Ca(OH)<sub>2</sub>. Then, three drops of chloroform were added to minimise microbial activity. The tubes were covered with PARAFILM with only a small opening left for air exchange to reduce evaporation, stirred regularly, and incubated at room temperature. The pH was measured every 24 h for 5 d while stirring the suspensions. The lime buffer capacity (mg CaCO<sub>3</sub> kg<sup>-1</sup> pH<sup>-1</sup>) was calculated for each incubation period as follows (Kissel et al., 2007):

$$\text{LBC} = \frac{V \times M \times M_W}{S \times (\text{pH}_e - \text{pH}_o)}, \quad (4)$$

where  $V$  is the volume of Ca(OH)<sub>2</sub> added (mL),  $M$  is the molarity of Ca(OH)<sub>2</sub> used for the titration (= 0.022 M),  $M_W$  is the molecular weight of CaCO<sub>3</sub> (100 mg mmol<sup>-1</sup>),  $S$  is

the weight of soil titrated (kg),  $\text{pH}_e$  is the pH of the suspension taken after addition of certain volume of  $\text{Ca}(\text{OH})_2$  and equilibration for specific incubation time, and  $\text{pH}_0$  is the pH of a suspension without  $\text{Ca}(\text{OH})_2$  taken at similar incubation time.

Total acidity, expressed as the proton ( $\text{H}^+$ ) concentrations that were neutralised over the incubation periods to finally attain equilibrium pH, was calculated using Eq. (5), which was derived from  $\Delta\text{pH} \times \text{pHBC}$ , by substituting  $1/\text{slope}$  as  $\text{pHBC}$  in Eq. (1):

$$\text{H}^+ = (\text{LBC} \times \Delta\text{pH} \times 2)/100, \quad (5)$$

where  $\text{H}^+$  is the proton concentration ( $\text{mmol H}^+ \text{ kg}^{-1} \text{ soil}$ ) that was neutralised at a given incubation time ( $t_i$ ),  $\text{LBC}$  is the lime buffer capacity ( $\text{mg CaCO}_3 \text{ kg}^{-1} \text{ pH}^{-1}$ ) calculated for the incubation time ( $t_i$ ),  $\Delta\text{pH}$  is the difference between the initial soil pH taken after 30 min of shaking the soil with base and the final soil pH taken at the incubation time ( $t_i$ ), 100 is the conversion factor from  $\text{mg CaCO}_3 \text{ kg}^{-1} \text{ pH}^{-1}$  to  $\text{mmol CaCO}_3 \text{ kg}^{-1} \text{ pH}^{-1}$ , and 2 is the conversion factor from  $\text{mmol CaCO}_3$  to  $\text{mmol H}^+$ .

#### 2.4.3 Equilibrium buffer curves and lime rates

Nonlinear regression curves between soil pH and incubation time as well as between  $\text{LBC}$  and incubation time were plotted for all concentrations of  $0.022 \text{ M Ca}(\text{OH})_2$  added to evaluate the change in soil pH and  $\text{LBC}$  over time. The curves were used to determine the equilibrium pH and  $\text{LBC}$  at equilibrium ( $\text{LBC}_{\text{eq}}$ , i.e. the point where the pH did not change significantly with time). In addition, a linear regression was fitted between  $\text{LBC}_{30}$  and  $\text{LBC}_{\text{eq}}$ , with different rates of  $0.022 \text{ M Ca}(\text{OH})_2$  as the covariate. The regression equation between  $\text{LBC}_{30}$  and  $\text{LBC}_{\text{eq}}$  was used to calculate the equilibrium lime buffer capacity ( $\text{LBC}_{\text{eq}}$ ) for the acidic soil based on the  $\text{pHBC}$  determined during the 30 min titration ( $\text{LBC}_{30}$ ). After the determination of  $\text{LBC}_{\text{eq}}$ , the LR of the acidic soil was calculated as follows:

$$\text{LR} (\text{mg CaCO}_3 \text{ kg}^{-1} \text{ soil}) = (\text{LBC}_{\text{eq}} \times (\text{pH}_t - \text{pH}_i)) / \text{ENV}, \quad (6)$$

where  $\text{LBC}_{\text{eq}}$  is the lime buffer capacity at equilibrium soil pH ( $\text{mg CaCO}_3 \text{ kg}^{-1} \text{ pH}^{-1}$ ),  $\text{pH}_i$  is the initial  $\text{pH}_W$  before the addition of  $\text{Ca}(\text{OH})_2$ ,  $\text{pH}_t$  is the target  $\text{pH}_W$  (i.e. 6.5 used in this study), and  $\text{ENV}$  is the effective neutralising value of the lime used (in percent).

#### 2.5 Titratable alkalinity and the application rate of organic amendments

The titratable alkalinity was determined by extracting the dried OAs with acid and then back-titrating the suspension to pH 7 with base using the modified methods of Singh et

al. (2017) and Yuan and Xu (2011). There was no washing step in this method. The acid-neutralising value of all of the compounds released by acid pretreatment, including any labile compounds, was estimated by back-titrating the solution. Briefly, 0.5 g of dried and sieved (0.25–2 mm) OAs were weighed into 50 mL polyethylene tubes in replicates. Then, 10 mL of 1 M HCl was added, and the mixture was shaken on a reciprocal shaker for 2 h. The suspension was allowed to stand for 24 h and then titrated with 0.5 M NaOH using a digital titrator. The amount of acid neutralised by each amendment (expressed as  $\text{mmol H}^+$  per gram OA) was calculated as the difference between the volume of 0.5 M NaOH consumed by the blank and the sample. The lime-equivalent value (LEV), expressed as  $\text{CaCO}_3$  equivalents, of the OAs was then calculated as follows:

$$\text{LEV}_{\text{OA}} = \frac{(0.5) \cdot (b - s) \cdot M_W}{2 \cdot W \cdot 1000}, \quad (7)$$

where LEV is the lime-equivalent value ( $\text{g CaCO}_3 \text{ kg}^{-1} \text{ OA}$ ), 0.5 is the molarity of NaOH used for titration ( $\text{mol L}^{-1}$ ),  $b$  is the volume of NaOH consumed by the blank (mL),  $s$  is the volume of NaOH consumed by the sample (mL),  $M_W$  is the molecular weight of  $\text{CaCO}_3$  ( $\text{g mol}^{-1}$ ), 2 is the moles of  $\text{H}^+$  neutralised by 1 mol of  $\text{CaCO}_3$ ,  $W$  is the weight of sample (kg), and 1000 is the unit conversion factor. Then, the amounts of OAs required to raise soil pH to a desired level ( $\text{pH}_W$  6.5 in this study) were calculated using the following expression:

$$\text{RAR}_{\text{OA}} (\text{g OA kg}^{-1} \text{ soil}) = (\text{LBC}_{\text{eq}} \cdot (\text{pH}_t - \text{pH}_i) \cdot 1000) / \text{LEV}_{\text{OA}}, \quad (8)$$

where  $\text{RAR}_{\text{OA}}$  is the recommended application rate of OAs,  $\text{LBC}_{\text{eq}}$  is the lime buffer capacity at equilibrium pH ( $\text{mg CaCO}_3 \text{ kg}^{-1} \text{ soil pH}^{-1}$ ),  $\text{pH}_t$  is the target soil pH,  $\text{pH}_i$  is the initial soil pH, and 1000 is the unit conversion factor.

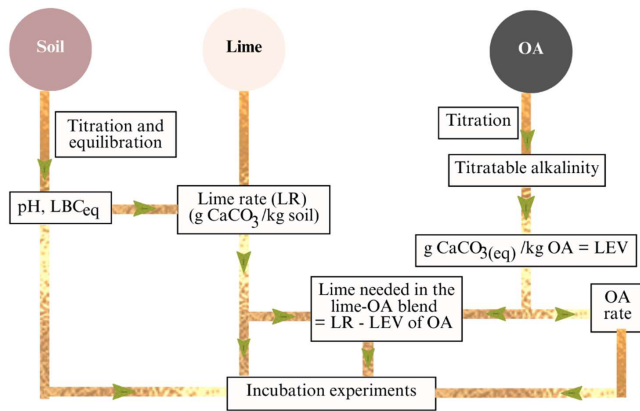
#### 2.6 Calculating rates of lime and organic amendments in their combinations

The application rates in the combinations of pure lime and OAs were calculated based on the titratable alkalinity of the OAs and the  $\text{CaCO}_3$  required to raise soil  $\text{pH}_W$  to 6.5 that depends on the  $\text{LBC}_{\text{eq}}$ . The method used to calculate lime–OA combinations is illustrated in Fig. 1.

The amount of pure lime that was combined with each OA to achieve a target  $\text{pH}_W$  of 6.5 was calculated by subtracting the LEV or  $\text{CaCO}_3$  equivalent of the OA (i.e. added at 1.5 %  $w/w$  in this study) from the lime rate using Eq. (9):

$$\text{LOA}_{\text{comb}} (\text{g CaCO}_3 \text{ kg}^{-1} \text{ soil}) = \text{LEV}_{\text{OA}} + \text{LR}_{\text{inorg}}, \quad (9)$$

where  $\text{LOA}_{\text{comb}}$  is the total  $\text{CaCO}_3$  required in the lime–OA combinations to raise the  $\text{pH}_W$  to 6.5,  $\text{LEV}_{\text{OA}}$  is the lime-equivalent value or  $\text{CaCO}_3$  content of each OA (i.e. added at



**Figure 1.** Conceptual method used to calculate the amount of lime, organic amendments (OAs), and lime–OA combinations.  $LBC_{eq}$ : equilibrium lime buffer capacity; LR: lime rate required to achieve  $pH_W$  6.5; LEV: lime-equivalent value.

15 g OA  $kg^{-1}$  in this study), and  $LR_{inorg}$  is the amount of lime (i.e. inorganic source of  $CaCO_3$ ) needed in the combinations (g  $CaCO_3$   $kg^{-1}$  soil).

## 2.7 Calculating organic amendment in mixes

A 50 % : 50 % alkalinity-based mixture of selected OAs was prepared by combining rapidly decomposable organic material (wheat straw or faba bean straw) with resistant organic materials (compost or biochar) to test the effect of the mixture on soil pH over time. The amount of each amendment in the two-OA mix was calculated in order to contribute 50 % of the total alkalinity (i.e. 50 % of 2.81 g  $CaCO_3$   $kg^{-1}$ ) required to neutralise soil acidity and achieve  $pH_W$  6.5.

## 2.8 Validation experiments at different water contents

Incubation experiments were conducted at 60 %, 100 %, and 150 % of field capacity (FC) to validate whether the amounts of soil amendments calculated based on their titratable alkalinity could raise the soil pH to the desired level ( $pH_W$  6.5). The treatments included unamended or control soil, lime, OAs, OA mixes, and lime–OA combinations. The amendment rates calculated based on the method developed in this study are presented in Table S1 in the Supplement.

For the incubations at 60 % and 100 % FC, the amendments were mixed with 50 g soil (< 2 mm) in pots in replicates. The pots were incubated in a dark room with deionised water added on a weight basis. The pH was measured after 14, 30, 60, 90, and 120 d for incubation at 60 % FC and after 10, 20, 30, 40, and 50 d for incubation at 100 % FC until equilibrium pH was attained. For incubation at 150 % FC, 5 g of air-dried soil (< 2 mm) was weighed into 50 mL polyethylene plastic tubes and the amendments were mixed with the soil.

The treatments were incubated in a dark room at 150 % FC with the moisture content maintained on a weight basis. Soil pH was measured at 4, 6, 8, 10, and 12 d by adding the amount of deionised water needed to make a 1 : 5 soil-to-water ratio and shaking the suspension for 45 min.

## 2.9 Statistical analysis

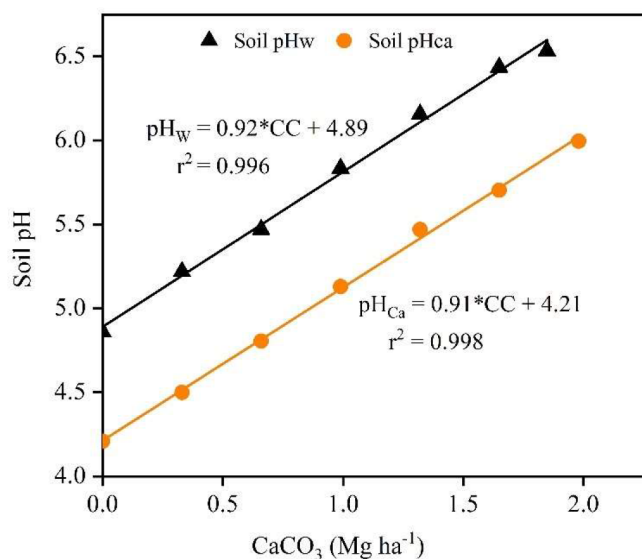
Regression curves for the titration points and equilibration experiments were fitted using OriginPro version 2022b (9.95). Mean comparisons of titratable alkalinity and other chemical properties of OAs were carried out using a one-way ANOVA. Mean comparisons of pH values measured over time during titration and equilibration experiments, as well as the rate of  $H^+$  neutralisation with incubation time, were undertaken using a one-way repeated-measures ANOVA. Changes in the pH values of amended soils over time for validation experiments conducted at different soil water contents were also analysed using a one-way repeated-measures ANOVA. The mean comparisons, correlation analysis, and tests of significance were conducted at  $p \leq 0.05$  using IBM SPSS version 28.0.1.0.

## 3 Results

### 3.1 Soil $pHBC_{30}$ and $LBC_{30}$

The regression lines between soil pH and incremental base additions (expressed as Mg  $CaCO_3$   $ha^{-1}$  equivalents; Eq. 3) were linear for titrations in water ( $r^2 = 0.996$ ) and 0.01 M  $CaCl_2$  ( $r^2 = 0.999$ ) (Fig. 2). The slopes of the regression lines were 0.92 and 0.91 pH ha Mg $^{-1}$   $CaCO_3$  for titrations in water and 0.01 M  $CaCl_2$  solution, respectively, with no significant difference between them ( $p = 0.231$ ).

The buffering capacity ( $pHBC_{30}$ ) of the acidic soil was estimated from the inverse slope of the titration curve and was 1.09 Mg  $CaCO_3$   $ha^{-1}$   $pH^{-1}$  in water and 1.10 Mg  $CaCO_3$   $ha^{-1}$   $pH^{-1}$  in 0.01 M  $CaCl_2$ , which are values equivalent to 14.54 and 14.66 mmol  $H^+$   $kg^{-1}$   $pH^{-1}$ , respectively. The  $pHBC$  is the amount of  $H^+$  that is consumed to raise the soil pH by one unit, whereas LBC is the amount of  $CaCO_3$  required to raise the soil pH by one unit. As a result,  $pHBC$  expressed as LBC is more convenient for calculating the amount of lime required to neutralise soil acidity. The average values of  $LBC_{30}$  calculated from  $pHBC_{30}$  were 727 mg  $CaCO_3$  (kg soil) $^{-1}$   $pH^{-1}$  for titration in water and 733 mg  $CaCO_3$  (kg soil) $^{-1}$   $pH^{-1}$  for titration in 0.01 M  $CaCl_2$ . The slightly higher  $pHBC_{30}$  in the 0.01 M  $CaCl_2$  solution than in water indicates that soil resistance to pH change increases when  $Ca^{2+}$  ions in the  $CaCl_2$  solution replace  $Al^{3+}$  and  $H^+$  ions in the soil exchange complex. The  $pHBC$  varied slightly across the pH values, indicating that the change in pH due to base additions is not uniform across the range of pH values.



**Figure 2.** Fitted linear regression lines for titrations to pH 6.5 in deionised water and pH 6.0 in 0.01 M CaCl<sub>2</sub> solution after 30 min. In the equations, “CC” is the CaCO<sub>3</sub> (Mg ha<sup>-1</sup>) consumed during titrations of the acidic soil with 0.022 M Ca(OH)<sub>2</sub>.

### 3.2 Equilibrium pH and lime buffer capacity

The pH<sub>w</sub> and LBC of soils amended with 0.5, 0.75, 1, 1.25, and 1.5 times the ETP of 0.022 M Ca(OH)<sub>2</sub> and incubated for 5 d using a 1 : 5 soil-to-deionised water ratio are shown in Fig. 3a and b. The pH<sub>w</sub> of unamended soil and soils amended with different rates of 0.022 M Ca(OH)<sub>2</sub> decreased nonlinearly with incubation time until it reached a relatively constant level. In contrast, compared with its initial pH, the pH<sub>w</sub> of the unamended soil increased slightly (by 0.11 units) by 72 h (Fig. 3a). To account for this change, which was not associated with base addition, the pH value for lime-amended treatments was corrected by subtracting the change in the pH of the control soil from the observed pH at the respective incubation time.

The soil pH<sub>w</sub> taken after 0.5 h for soil treated with 1 · ETP was similar to those of the 30 min titration. However, pH<sub>w</sub> values at 0.5, 24, and 48 h of incubation were significantly different from each other and from the rest of the incubation periods for all rates of Ca(OH)<sub>2</sub>. In contrast, the pH<sub>w</sub> values obtained after 72, 96, and 120 h of incubation were similar. This indicates that the acid–base reaction had reached equilibrium after 72 h of incubation. Based on this, the trends in the regression lines shown in Fig. 3a, and the quantity of protons neutralised over time (Fig. 3d), the equilibrium pH was calculated as the average of the adjusted pH values obtained after 96 and 120 h of incubation. For example, the equilibrium pH for 1 · ETP was 5.99 (i.e. (6 + 5.97)/2), implying that the 0.022 M Ca(OH)<sub>2</sub> equivalent determined during titration in water with a 30 min equilibration time only neutralised 69 % of the soil acidity when the target pH<sub>w</sub> was 6.5.

Furthermore, the LBC was calculated using Eq. (4) with the incubation time adjusted with respect to the control (unamended) soil, i.e. dividing the observed LBC by the correction factor (CF). The CF was calculated by dividing the difference in pH between a soil amended with “a” (the rate of 0.022 M Ca(OH)<sub>2</sub>) and the control soil “c” at a specific incubation time “t<sub>i</sub>” (e.g. 24, 48, 72 h) by the difference in pH between a soil amended with the same rate of Ca(OH)<sub>2</sub> and the control measured at 0.5 h of incubation.

The LBC had an increasing nonlinear trend with incubation time until equilibrium was reached, and values calculated for 72, 96, and 120 h incubations were not significantly different from each other (Fig. 3b) because LBC<sub>eq</sub> is obtained when the pH reaches equilibrium. By comparing the LBC values and the trends in the regression lines in Fig. 3b, the average LBC values obtained at 96 and 120 h incubations were used as LBC<sub>eq</sub> for the acidic soil in this study.

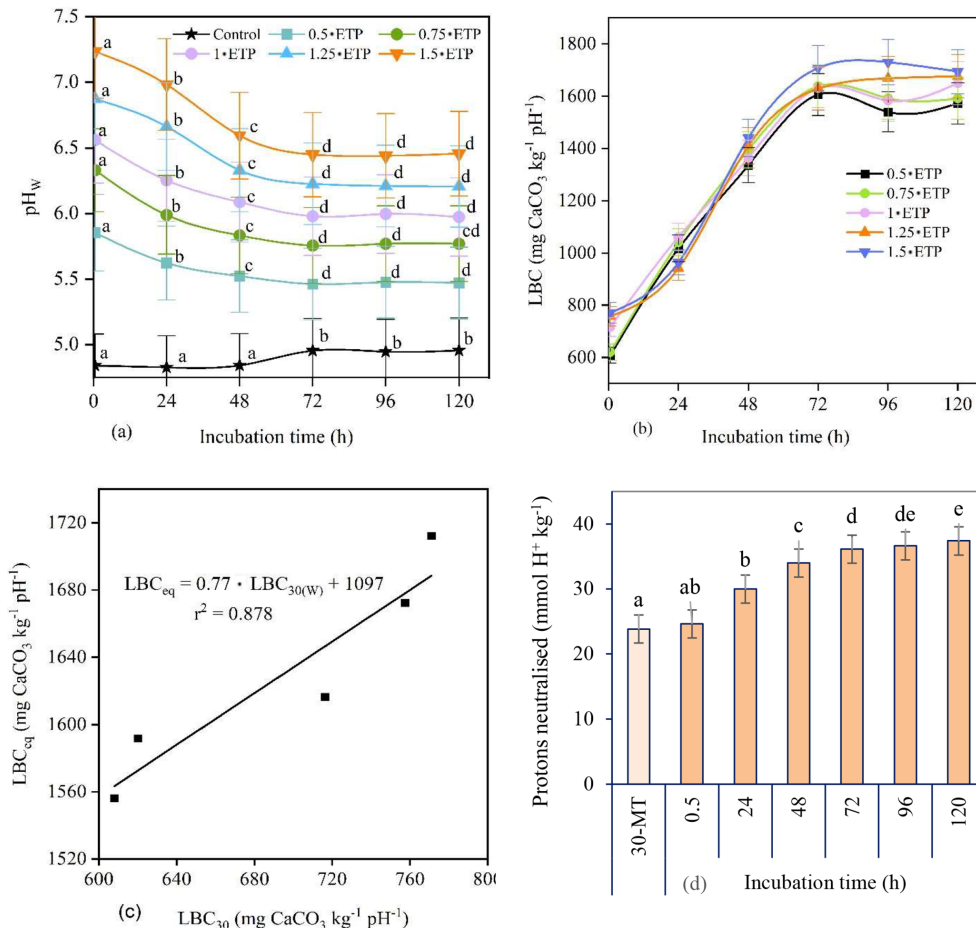
Subsequently, a regression equation was developed between adjusted LBC<sub>eq</sub> and LBC<sub>30</sub> (measured after 0.5 h), with different amounts of Ca(OH)<sub>2</sub> as the covariate (Fig. 3c). The LBC<sub>eq</sub> for the soils incubated with base in deionised water was then calculated from the LBC<sub>30</sub> value. For example, for an LBC<sub>30</sub> of 727 mg CaCO<sub>3</sub> kg<sup>-1</sup> pH<sup>-1</sup>, the LBC<sub>eq</sub> was 1657 mg CaCO<sub>3</sub> kg<sup>-1</sup> pH<sup>-1</sup>. This relationship between LBC<sub>30</sub> and LBC<sub>eq</sub> can be used to calculate LBC<sub>eq</sub> from LBC<sub>30</sub> data for similar soils.

### 3.3 Proton concentrations and lime requirements

The cumulative concentrations of protons neutralised by the added base during the 5 d incubation with 1 · ETP are shown in Fig. 3d. Thus, 1 · ETP incubated for 120 h neutralised 37.39 mmol H<sup>+</sup> kg<sup>-1</sup> soil (Eq. 5) and raised the pH<sub>w</sub> from 4.84 to 5.99, thus neutralising 36 % more protons than the initial 30 min titration (30-MT) (Fig. 3d). To raise the soil pH<sub>w</sub> to 6.5, 55 mmol H<sup>+</sup> kg<sup>-1</sup> must be neutralised. This corresponds to the total active acidity, as a base (OH<sup>-</sup>) neutralises not only indigenous H<sup>+</sup> but also H<sup>+</sup> produced by the hydrolysis of Al<sup>3+</sup>. Therefore, 2751 mg CaCO<sub>3</sub> kg<sup>-1</sup> soil was needed to raise the pH<sub>w</sub> to 6.5. For the lime used in this study (98 % effective neutralising value), that would be 2807 mg CaCO<sub>3</sub> kg<sup>-1</sup> soil (Eq. 6).

### 3.4 Effect of the alkalinity of organic amendments on soil pH

The titratable alkalinity of OAs ranged from 12 cmol H<sup>+</sup> kg<sup>-1</sup> for wheat straw to 357 cmol H<sup>+</sup> kg<sup>-1</sup> for compost (Table 3). Titratable alkalinity of blended poultry litter and biochar was about half that of compost (Table 3). The titratable alkalinity of OAs was significantly correlated with the EC of the amendments ( $r^2 = 0.69^{**}$ ,  $p = 0.004$ , where “\*\*” represents significance at the 0.01 level) (Table S2). In addition, alkalinity was moderately correlated with the inherent pH of OAs ( $r^2 = 0.60^*$ ,  $p = 0.018$ ,



**Figure 3.** (a) The pH<sub>w</sub> of unamended (control) and amended soils with incubation time. (b) Relationship between the lime buffer capacity (LBC, mg CaCO<sub>3</sub> kg<sup>-1</sup> pH<sup>-1</sup>) and incubation time. The LBC was calculated for different rates of 0.022 M Ca(OH)<sub>2</sub> using Eq. (4). The amended soils received 0.022 M Ca(OH)<sub>2</sub> at rates of 0.5, 0.75, 1, 1.25, and 1.5 · ETP, where the equivalent titration point (ETP) is the volume of 0.022 M Ca(OH)<sub>2</sub> consumed during a 30 min titration in water to raise the soil pH<sub>w</sub> to 6.5 (i.e. 2.8 mL). (c) Relationship between the corrected LBC<sub>eq</sub> and the LBC<sub>30</sub> of soils amended with different rates of 0.022 M Ca(OH)<sub>2</sub> converted to CaCO<sub>3</sub> equivalent. LBC<sub>eq</sub>: equilibrium lime buffer capacity (i.e. after 96–120 h); LBC<sub>30</sub>: lime buffer capacity determined after 30 min titration in water. (d) Proton concentration neutralised by 0.022 M Ca(OH)<sub>2</sub> added at a rate of 1 · ETP during a 30 min titration (30-MT) and 5 d incubation in water. The term 30-MT represents the time required to complete a titration to achieve pH<sub>w</sub> 6.5 with a 30 min interval between each base addition, whereas 0.5 h refers to the initial time in the 5 d incubation. Pairwise comparisons using a repeated-measures ANOVA are shown, and the different letters indicate significant differences ( $p \leq 0.05$ ) over time.

where “\*\*” represents significance at the 0.05 level). The LEV or CaCO<sub>3</sub> content of each OA added at 1.5 % (*w/w*) (i.e. 15 g kg<sup>-1</sup> soil) (Eq. 7) varied with the titratable alkalinity of the OA (Table 3). The amounts of OAs required to neutralise soil acidity and achieve a pH<sub>w</sub> of 6.5, which was calculated based on the LEV of the amendments and the LBC<sub>eq</sub> of soil (Eq. 8), was inversely related to the alkalinity content of the amendments (Table 3).

Changes in soil pH ( $\Delta$ pH) caused by all OAs were significantly different at all water contents (60 %, 100 %, and 150 % FC). Incubation with faba bean and wheat straw at 60 % FC increased the pH<sub>w</sub> of the soil from 4.84 to 6.53 and 6.42, respectively (Fig. 4a). For the less biodegradable

or resistant OAs (ROAs) (i.e. blended poultry litter, compost, and biochar), the soil pH at 60 % FC was lower than for crop straws (Fig. 4a). Biochar resulted in a higher soil pH<sub>w</sub> (6.27) than the other ROAs.

At 100 % FC, faba bean and wheat straws also resulted in a soil pH higher than ROAs, with mean pH<sub>w</sub> values of 6.55 and 6.46, respectively (Fig. 4b). At this soil moisture content, ROAs such as biochar and compost increased the soil pH<sub>w</sub> from 4.84 to 6.33 and 6.16, respectively, thus resulting in higher pH values than at 60 % FC (Fig. 4a). The rate of change in soil pH over time ( $\Delta$ pH/time in days) was also rapid and higher than at 60 % FC. For instance, the rates of pH change for faba-bean-



**Table 3.** Titratable alkalinity of organic amendments and the amount of lime combined with the organic amendments added at 1.5 % to raise the soil pH<sub>W</sub> from 4.84 to 6.5. Different letters indicate significant differences ( $p \leq 0.05$ ).

Type of organic amendment	Titratable alkalinity (cmol H <sub>eq</sub> <sup>+</sup> kg <sup>-1</sup> OA)	LEV <sub>OA</sub> (g CaCO <sub>3</sub> kg <sup>-1</sup> )	Calculated rates of OA (g kg <sup>-1</sup> soil) to achieve pH <sub>W</sub> 6.5	LEV <sub>OA</sub> (g CaCO <sub>3</sub> (15 g OA) <sup>-1</sup> )	Amount of lime (g CaCO <sub>3</sub> kg <sup>-1</sup> soil) required with 1.5 % OA to achieve pH <sub>W</sub> 6.5
Wheat straw	11.7±5.2 <sup>a</sup>	5.8±2.5	471.1±7.3	0.09±0.04 <sup>a</sup>	2.72±0.04 <sup>a</sup>
Faba bean straw	43.0±3.6 <sup>b</sup>	21.5±1.8	127.8±6.8	0.32±0.03 <sup>b</sup>	2.49±0.03 <sup>b</sup>
Blended poultry litter	176.0±4.6 <sup>c</sup>	88.1±2.3	31.2±0.8	1.32±0.04 <sup>c</sup>	1.49±0.04 <sup>c</sup>
Biochar	168.7±1.5 <sup>c</sup>	84.4±0.8	32.6±0.3	1.27±0.02 <sup>c</sup>	1.54±0.01 <sup>c</sup>
Compost	357.0±5.5 <sup>d</sup>	178.7±2.7	15.4±0.2	2.68±0.04 <sup>d</sup>	0.13±0.04 <sup>d</sup>

OA: organic amendment; LEV: lime-equivalent value. The sum of CaCO<sub>3</sub> of OA (column 5) and the amount of lime (column 6) is equal to 100 % CaCO<sub>3</sub> equivalent (2.81 g CaCO<sub>3</sub> kg<sup>-1</sup> soil for the soil used in this study).

straw-amended and biochar-amended soils were 0.22 and 0.13 pH units d<sup>-1</sup> at 100 % FC, whereas these values were 0.11 and 0.07 pH units d<sup>-1</sup> at 60 % FC, respectively. Faba-bean-straw-amended soil reached an equilibrium pH after 30 d of incubation at 100 % FC but after 90 d at 60 % FC (Fig. 4a, b). The remaining soil amendments had a similar decrease in the equilibrium period with increasing soil water content. This could be due to the increased availability of both water and air for biodegradation of OAs at 100 % FC, resulting in higher alkalinity generation and the neutralisation of acidic soil.

Soils incubated with ROAs for 12 d at 150 % FC (submerged conditions) had a higher soil pH than those incubated with faba bean and wheat straws (Fig. 4c). Biochar, compost, and blended poultry litter raised soil pH<sub>W</sub> to 6.44, 6.40, and 6.39, respectively. On the other hand, wheat and faba bean straws resulted in lower pH values (5.75 and 5.93, respectively) at 150 % FC than at the lower water content values. For all soil amendments, the rate of change in soil pH at 150 % was greater than at 100 and 60 % FC. For example, at 150 % FC, faba-bean-straw-amended and biochar-amended soils reached an equilibrium pH after 12 and 10 d (Fig. 4c), respectively, and their corresponding rates of change in pH were 0.48 and 0.64 pH units d<sup>-1</sup>.

The magnitude of change in pH or alkalinity production by straws decreased above 100 % FC, whereas that of lime and less biodegradable OAs linearly increased with soil water content. The correlation between the titratable alkalinity of amendments and changes in soil pH increased significantly with soil water content, with  $r^2$  values of 0.72\*\*, 0.75\*\*, and 0.82\*\* at 60 %, 100 %, and 150 % FC, respectively (Table S2).

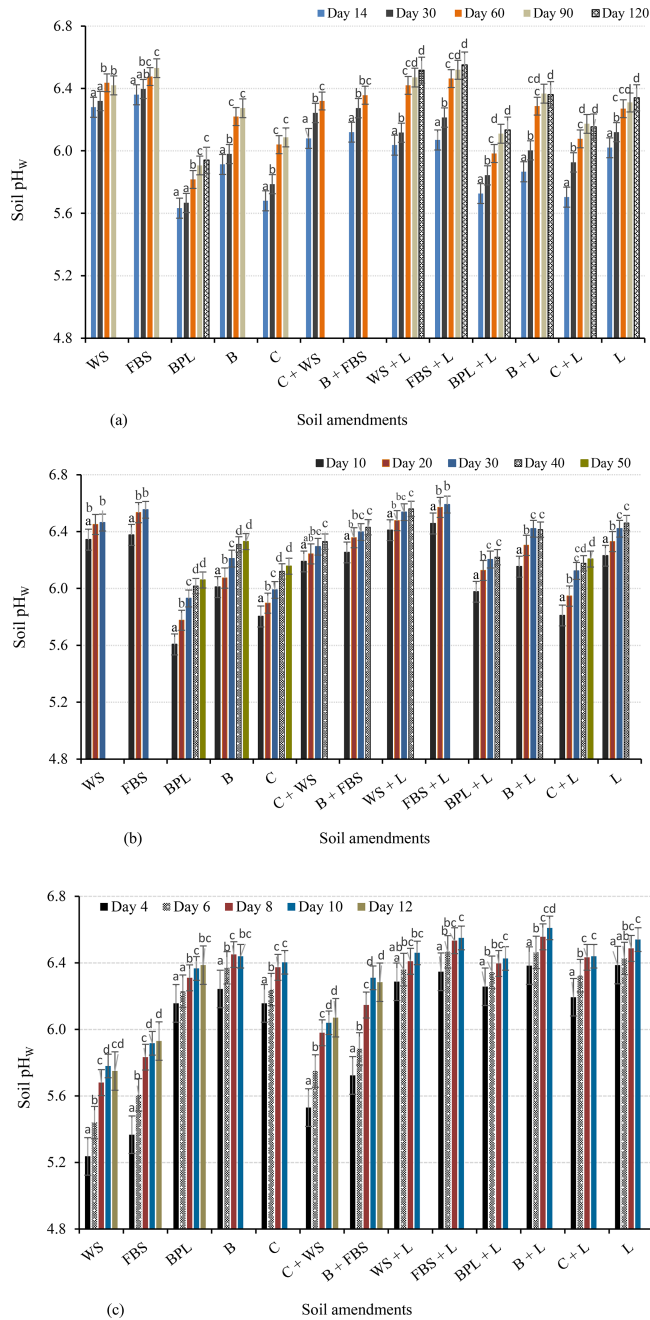
The alkalinity-based mixture of wheat straw–compost or faba bean straw–biochar that aimed to generate 50 % : 50 % total alkalinity was calculated as 235.6 g kg<sup>-1</sup> wheat straw + 7.7 g kg<sup>-1</sup> compost or 63.9 g kg<sup>-1</sup> faba bean straw + 16.3 g kg<sup>-1</sup> biochar, respectively. The mixes of resistant and easily biodegradable OAs are based on their alkalinity

to provide the required 100 % CaCO<sub>3</sub> to neutralise soil acidity. The 50 % : 50 % alkalinity-based mixture of resistant and easily decomposable OAs produced soil pH values that were intermediate between the individual amendments. At 60 % FC, the wheat straw and compost mixture resulted in a soil pH<sub>W</sub> of 6.32, which was 0.23 units higher than the pH changes caused by compost but 0.10 units lower than with wheat straw (Fig. 4a). The faba bean straw and biochar mixture increased pH<sub>W</sub> to 6.36, resulting in values that were higher than biochar but lower than faba bean straw. A similar trend was observed at 100 % FC, although with slightly higher equilibrium pH values of 6.33 (compost–wheat straw mix) and 6.43 (biochar–faba bean straw mix) (Fig. 4b). This shows that the pH changes by alkalinity-based OA mixes can be predicted from pH changes by individual OAs.

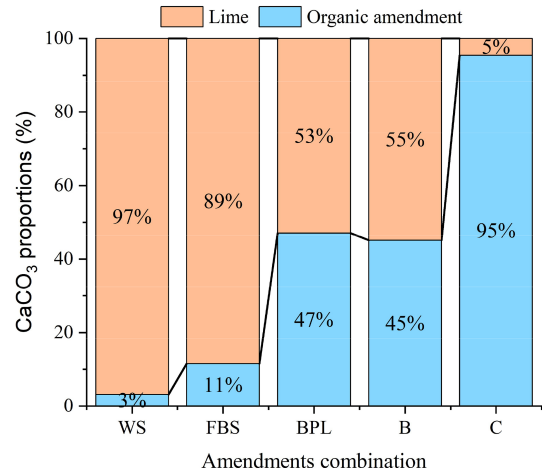
### 3.5 Effect of lime–organic amendment combinations

The amount of lime required in the lime–OA combinations was calculated using Eq. (9). When the OAs were added at 15 g kg<sup>-1</sup>, the amount of lime needed to ameliorate acidic soils in combined lime–OAs was inversely related to the titratable alkalinity of the OAs. With increasing alkalinity of the OAs from 5.8 g CaCO<sub>3</sub> kg<sup>-1</sup> (wheat straw) to 178.7 g CaCO<sub>3</sub> kg<sup>-1</sup> (compost), the lime requirement in lime–OA combinations decreased from 2.72 (wheat straw) to 0.09 g CaCO<sub>3</sub> kg<sup>-1</sup> (compost) (Table 3, Fig. 5). Compost addition, for example, reduced the lime requirement by 95 % compared with lime alone. Wheat straw with very low alkalinity, on the other hand, reduced lime requirement by only 3 % compared with lime alone.

Lime added alone increased the soil pH by 0.20 units when the soil moisture content increased from 60 % to 150 %. Combinations of lime and OAs resulted in a relatively higher soil pH than OAs alone at all incubation moisture levels (Fig. 4a, b, c). Combined additions of faba bean straw–lime resulted in a soil pH<sub>W</sub> of 6.55, followed by wheat straw–lime (pH<sub>W</sub> of 6.52), after 120 d of incubation at 60 % FC (Fig. 4a).



**Figure 4.** Changes in the pH<sub>w</sub> of soils incubated with individual amendments or combinations of different amendments (a) at 60% FC for 120 d, (b) at 100% FC for 50 d, and (c) at 150% FC for 12 d. Different letters indicate significant changes in the soil pH with incubation time. The final pH values represent the equilibrium pH of the treatment. WS: wheat straw; FBS: faba bean straw; BPL: blended poultry litter; B: biochar; C: compost; L: lime.



**Figure 5.** Proportions of alkalinity generated by organic amendments (OAs) added at 15 g kg<sup>-1</sup> and the lime required to achieve 100% CaCO<sub>3</sub> equivalent in combined lime-OA applications. WS: wheat straw; FBS: faba bean straw; BPL: blended poultry litter; B: biochar; C: compost.

At 100% FC, the faba bean–lime and wheat straw–lime combinations generated a soil pH<sub>w</sub> of 6.59 and 6.56 within 30 and 40 d of incubation, respectively (Fig. 4b). At 100% FC, lime combined with biochar, blended poultry litter, and compost increased the soil pH<sub>w</sub> to 6.41, 6.22, and 6.21, respectively. At 150% FC, the combined lime–ROA treatments increased the soil pH<sub>w</sub> to close to 6.5 (Fig. 4c).

## 4 Discussion

Our findings show that the laboratory method developed based on the titratable alkalinity of OAs and standard soil parameters (pH and LBC) can be used to accurately calculate OA rates and lime–OA combinations for the amelioration of acid soils.

### 4.1 Optimum application rates of soil amendments

The acidity in the soil that had to be neutralised to achieve the target pH<sub>w</sub> of 6.5 was estimated by determining an equilibrium LBC. In this study, an equilibrium pH of 5.99 was reached after 96–120 h at the equivalent CaCO<sub>3</sub> rate determined during titration, giving an LBC<sub>eq</sub> of 1657 mg CaCO<sub>3</sub> kg<sup>-1</sup> pH<sup>-1</sup>. Lime requirement was then calculated by multiplying this LBC<sub>eq</sub> by the required change in soil pH. However, it should be noted that the time required to reach the equilibrium pH depends on the pH<sub>HBC</sub> of the soil (Jalali and Moradi, 2020; Kissel et al., 2005; Wang et al., 2015). Hence, it is important to check this equilibrium time when using this method for different soils. LBC estimates based on 30 min titration (LBC<sub>30</sub>) did not represent the total concentrations of soil acidity that had to be neutralised to bring the pH to the desired level, as the acid–base neutral-

sation reactions do not attain equilibrium in this short time period (Barrow and Cox, 1990; Kissel et al., 2007; Liu et al., 2004). However, we showed that a linear regression between  $LBC_{eq}$  and  $LBC_{30}$  can be used for rapid assessment, once this relationship is developed for a particular soil, without the need to conduct a long-term incubation each time. The results showed that the percent by weight ( $w/w$ ) of OAs needed to achieve a soil  $pH_W$  of 6.5 is inversely related to the titratable alkalinity of those amendments. The titratable alkalinity of OAs increased in the following order: wheat straw < faba bean straw < biochar < blended poultry litter < compost (Table 3). The optimum application rates of the amendments decreased in the following order: wheat straw (47.1 %) > faba bean straw (12.8 %) > biochar (3.3 %) > blended poultry litter (3.1 %) > compost (1.5 %).

#### 4.2 Effect of different organic amendment properties on the soil pH

Alkalinity content has been previously found to be a primary measure of the acid-neutralising capacity of OAs (Noble et al., 1996; Noble and Randall, 1998). This was confirmed in the present study by the significant correlation between the soil pH and titratable alkalinity of OAs at various soil water contents. Differences in the alkalinity content of OAs has previously been found to be due to differences in the excess cation content and other physicochemical properties which are influenced by soil, climate, and management practices (Noble and Randall, 1998; Slattey et al., 1991).

Previous studies have also shown that OAs generate alkalinity in the form of hydroxyl ( $-OH$ ) and carboxyl ( $-COOH$ ) surface functional groups, carbonates, organic anions (e.g. oxalate and malate), inorganic anions such as sulfate ( $SO_4^{2-}$ ), phosphate ( $PO_4^{3-}$ ), silicate ( $SiO_4^{4-}$ ), and iron hydroxides ( $FeO-O-$ ) (Cai et al., 2020; Dai et al., 2017; Fidel et al., 2017; Li et al., 2022; Sakala et al., 2004). These groups detoxify  $H^+$ ,  $Al^{3+}$ , and other acid-forming ions through neutralisation and association reactions. Different OAs have different types and numbers of these groups.

In our experiments, the magnitude of the soil pH changes at the same soil water content was not consistently related to the titratable alkalinity of the OAs added. This suggests that other physicochemical properties of OAs (e.g. inherent pH, carbonate content, and C/N ratio) may significantly influence soil pH changes. Biochar, for example, was added at a rate of 100 %  $CaCO_{3eq}$ , which was comparable to the rates for compost and blended poultry litter, but biochar increased soil pH more than these amendments. This might be caused by other properties of biochar such as a high inherent pH and solid-phase carbonate content following pyrolysis (Mosley et al., 2015) as well as oxygen-rich surface functional groups and a large specific surface area, which leads to high surface adsorption of protons and  $Al^{3+}$  (Chintala et al., 2014; Cui et al., 2021).

We found that, when applied at similar  $CaCO_3$  equivalents, faba bean straw produced a higher soil pH than wheat straw at all soil water contents. This could be because faba bean straw has a lower C/N than wheat straw (Table 2) and, therefore, is decomposed more quickly and results in net ammonification during the mineralisation of organic N and a pH increase. Higher total N concentrations in legumes than in cereals likely lead to a higher N-cycling rates and a faster biological decarboxylation (Butterly et al., 2013), resulting in faster and greater soil pH increases.

#### 4.3 Impact of the soil water content on alkalinity production by organic amendments

The present study showed that changes in the soil pH by wheat and faba bean straws decreased as follows: 100 % FC > 60 % FC > 150 % FC. In contrast, the ROA soil pH increased with water content as follows: 60 % FC < 100 % FC < 150 % FC. When applied at similar  $CaCO_3$  equivalent, wheat and faba bean straws resulted in higher pH values than ROAs at 60 % FC. The more rapid soil pH changes with wheat and faba bean straws to  $pH_W$  6.53 and 6.42 compared with the other OAs at 60 % FC are likely due to the fact that crop straws decompose fast because microbial activity is high when oxygen availability is high (Grzyb et al., 2021; Jin et al., 2023), leading to the rapid release of available alkalinity. In contrast, the pH changes with ROAs were slow at 60 % FC. The lignin content of woodchips in blended poultry litter and the resistance of compost and biochar to further decomposition is likely to result in slow decomposition of these amendments, thereby reducing the release of organic anions. In addition, the water content at 60 % FC may not be high enough for the dissolution of soluble organic compounds and, thus, production of alkalinity in ROAs. As a result, soil amended with ROAs reached an equilibrium pH after 90–120 d at 60 % FC, indicating that ROAs should be added several months before planting to neutralise acidity.

We found that an increase in the soil water content from 60 % to 100 % FC reduced the time required for amended soils to reach equilibrium pH by nearly 2-fold. Thus, the soil water content should be considered when deciding on the application time and comparing the effects of amendments on soil pH (e.g. optimal application may be before a significant rainfall event).

At 150 % FC, amendment with ROAs resulted in a  $pH_W$  close to 6.5, likely because there was enough water for the dissolution of soluble organic/inorganic anions and carbonates. Even after the equilibrium pH was reached, undissolved/undecomposed ROA particles were found in the suspension, causing minor deviations in the soil  $pH_W$  from 6.5. However, it is unlikely that the entire acid-extractable alkalinity of the ROAs is released by water. Adeleke et al. (2017) suggested that organic anions that provide various functional groups (amino, carboxylic, phenolic) for surface adsorption

of  $H^+$  and  $Al^{3+}$  are weak acids and do not dissolve completely in water. Acid-extractable alkalinity determined by the reaction of organic materials with acids (e.g. HCl) over a long time can also lead to the increased solubility of inorganic alkali, exposing occluded inorganic alkali and occluded conjugate bases of functional groups (Fidel, 2012), which are not soluble in water. Nevertheless, water-insoluble fractions of acid-extractable alkalinity can increase the long-term buffering capacity of acidic soils (Fidel et al., 2017; Yuan et al., 2011).

However, wheat and faba bean straws induced lower pH at 150 % FC. The lower pH with wheat and faba bean straws at 150 % FC than at the lower water contents may be because undecomposed crop straws were added at high rates, resulting in a low oxygen content at this high water content due to low decomposition rates and, thus, the limited production of alkalinity. For example, Chen et al. (2018) showed that anaerobic conditions reduced the rate of straw decomposition by 30 % compared with aerobic conditions. Reduction in organic material decomposition is associated with a limited production of organic anions (Cai et al., 2020; Xu et al., 2006b). Furthermore, anaerobic conditions may have favoured the formation of acid-forming products, such as  $H_2S$ , and promoted the formation of protons from the acidic soil via the hydrolysis of  $Al(OH)_3$  and the dissolution of Fe hydroxy oxide clay minerals.

#### 4.4 Effect of mixing soil amendments based on alkalinity production

An alkalinity-based mixture of resistant and easily decomposable OAs produced soil pH changes that were intermediate (between the two amendments). The mixtures led to greater pH increases than ROAs alone but smaller pH changes than rapidly decomposable OAs alone. Previous studies have indicated that the nutrient release and decomposition patterns of mass-based OA mixtures resulted in nonadditive responses (Gartner and Cardon, 2004; Le and Marschner, 2018). However, there is no information on how alkalinity-based mixtures of OAs affect soil pH. Our findings show that for alkalinity-based OA mixtures, pH changes in the mixes can be predicted from the pH changes in the individual amendments. The pH values can be estimated as the sum of pH changes by individual amendments multiplied by their proportion of alkalinity in the mix. Hence, mixing of OAs based on their alkalinity and C/N ratio could promote an early pH increase due to the rapid production of alkalinity by easily decomposable amendments as well as a sustained increase later from the resistant amendments. Amendment mixtures may occur where crop residues are left in the field after harvest and are mixed with ROAs to generate the additional alkalinity required to neutralise soil acidity.

The results showed that lime–OA combinations induce a higher pH than individual OAs. For instance, the combined lime–ROA treatments increased the soil  $pH_W$  to about 6.5

at 150 % FC, which can likely be attributed to the increased alkalinity production for the reasons explained above. As lime dissolution in acidic soils increases with water content (Anderson et al., 2020; Naorem et al., 2022), the combined use of lime and ROAs could be a good option for managing acidic soils forming in coastal areas and wetlands, including acid sulfate soils. Mixtures of lime and wheat and faba bean straws also produced a higher pH than lime alone. This could be due to synergetic interactions between lime and OAs, leading to high alkalinity generation. Decomposition of OAs generates  $CO_2$  which leads to the dissolution of carbonate; liming, on the other hand, may increase OA decomposition by increasing the soil pH. However, the relative effectiveness of OAs with lime in ameliorating acidic soil is not simply the sum of their neutralising capacity (Butterly and Tang, 2018), likely due to the adsorption of cations (e.g.  $Ca^{2+}$ ) released from lime on the surfaces of OAs.

## 5 Conclusion

This study showed that the titratable alkalinity of OA and the LBC of acidic soils can be used to accurately calculate the amounts of OAs and the lime–OA combination ratio needed to neutralise soil acidity. The alkalinity production potential of soil amendments was significantly affected by the soil amendment type and soil water content. Crop straws decomposed faster to release more alkalinity under aerobic than anaerobic conditions. On the other hand, lime and ROAs, such as compost, biochar, and blended poultry litter, led to a higher pH in soils with a high water content. Alkalinity-based mixtures of two OAs produced soil pH changes that were intermediate between the individual amendments. Lime–OA combinations, in contrast, led to positive interactions, generating more alkalinity than individual amendments. The developed method is novel because it could help to reduce the time and cost associated with conducting field experiments to determine the optimum lime–OA rates for a specific soil. This could be particularly important in situations where lime sources are unavailable or need to be transported long distances. It could also allow flexibility in adjusting the amount of lime or OAs in combined applications based on their availability and cost. The method can be reproduced in different soils, as it only requires the measurements of standard soil parameters (soil pH and LBC) and the titratable alkalinity of OAs. The study also showed that uncertainty in achieving the desired soil pH from amendment calculations based on the titratable alkalinity could be due to the soil moisture following amendment, fractions of water-insoluble alkalinity, and technical errors in measuring soil  $LBC_{eq}$  and OA alkalinity.

**Data availability.** The data generated in this study are available from the corresponding author upon request.

**Supplement.** The supplement related to this article is available online at: <https://doi.org/10.5194/soil-10-33-2024-supplement>.

**Author contributions.** BI, LMM, and PM conceptualised the study. BI conducted soil sampling, performed the experiments, and drafted the manuscript with editorial contributions from the two co-authors.

**Competing interests.** The contact author has declared that none of the authors has any competing interests.

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