



## RESEARCH ARTICLE

# A comparison of soil liming requirement methodologies in temperate, Northern European pedo-climates

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## Abstract

**Background:** Liming agricultural land is essential to optimise crop yield and soil nutrients. Despite the importance of pH management in agricultural soils, liming applications have been decreasing in the United Kingdom for decades. There is no comparison of contemporary and historical liming requirement (LR) methods for Northern European, temperate climate mineral soils high in organic matter (OM).

**Aims:** The aims of this research were to thoroughly comparatively analyse current methodologies and to ascertain which soil characteristics contribute to LR reactions.

**Methods:** Analysis compared methods for determining liming values common in the United Kingdom (Scottish Agricultural College [SAC] look-up chart, RothLime model), Europe and the United States (Shoemaker–McLean–Pratt, Sikora, Modified Mehlich buffers), and the 30-min calcium hydroxide titration developed by the University of Georgia.

**Results:** RothLime and SAC highly underestimated the LR value in acidic soils. The buffers highly over or underestimated LRs. The UGA titration method is a cheap, easy and accurate method which could be utilised for high OM soils but requires further calculation development. The characteristics most associated with soil–lime reactions in this experiment were measures of exchangeability (cation exchange capacity and loss on ignition, and by proxy, lime buffering capacity).

**Conclusions:** There is an opportunity to create buffer calculators and titration equations adapted to high OM soils. These are suggested for further development, through a larger diversity of UK soil types grouped by buffering capacity ranges. Including soil exchangeability factors in lime management calculations may contribute to more accurate values and therefore better resource management. Increasing LR accuracy for site-specific soil pH management, used in precision agriculture technologies, is a necessary tool for the conservation of natural resources like limestone, managing resource use efficiency, and for optimising yields.

## KEYWORDS

acidity, buffers, calcium hydroxide, RothLime, soil pH, titration

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## 1 | INTRODUCTION

United Kingdom climate policy aims to reduce agricultural sector emissions by 9% by year 2032, reaching this target will rely on precision soil management. The policy aims for improved on-farm nitrogen (N) fertiliser efficiency with variable rate fertiliser and lime spreading by utilising precision agriculture technologies (Scottish Government, 2018). To counteract natural and anthropogenic processes of soil acidification, soil pH is managed by the application of agricultural lime and lime applications have reduced nitrous oxide (N<sub>2</sub>O) and methane (CH<sub>4</sub>) emissions in soils (Abdalla et al., 2022). Though soil pH is historically a fundamental aspect of management, declining rates of agricultural lime applications have been observed in the United Kingdom (Farm Advisory Service (FAS), 2019; Goulding, 2016). Annual application rates were 6000–7000 kt year<sup>-1</sup> in 2003 but 2500 kt year<sup>-1</sup> in 2013 (Goulding, 2016). In Scotland, the percentage area of grassland treated with lime declined from 9% of total land area to 4.9% between 1998 and 2013 (PNMG, 2018). Liming requirement (LR) determination methods are used to recommend a quantity of lime to apply to a field, a key tool for precise nutrient management. Comparisons of methods used to determine LR values have been conducted in Ireland and Greece with emphasis on US, EU and UK methods, but such a comparison has not yet been performed on a diversity of United Kingdom soils that reflect temperate, Northern European pedo-climatic conditions (Barouchas et al., 2013; Tunney et al., 2010).

A range of lab-based methods, look-up charts and models are used to determine the amount of lime required to reach an optimum soil pH value. Lab methods include direct titration, incubation or measuring pH changes with a single or double buffer. Considered a precise measure of acidity, buffers are used to measure total exchangeable acidity, whereas soil pH measures active acidity (Godsey et al., 2007). Calculations commonly used to translate buffer pH measurements to LR values are calibrated for regional US soil characteristics and for different purposes (Godsey et al., 2007). The Shoemaker, McLean and Pratt (SMP) or Adams-Evans buffers are calculated to lime to a target pH (Adams & Evans, 1962; Shoemaker et al., 1961), whereas the Mehlich buffer determines the minimum amount of lime required to remove aluminium (Al<sup>3+</sup>) toxicity (Mehlich, 1976). Methods have been modified to produce effective alternatives that do not use environmentally harmful chemicals such as *p*-nitrophenol and potassium chromate. The Sikora buffer is an update to the SMP buffer, and Modified Mehlich (MM) is an update to the Mehlich buffer (Hoskins & Erich, 2008; Sikora, 2006).

Direct titration using a saturated calcium hydroxide (Ca(OH)<sub>2</sub>) solution is another method and can be referred to as the titration, single addition or the University of Georgia (UGA) method (Liu et al., 2004; Kissel et al., 2012; Sikora & Moore, 2012). Titration is a well-established method to predict LR values, this method measures the buffering reaction after the addition of an acid or a base to soil. This reaction is measured over a period of minutes to hours, days weeks or months. Initial research found equilibrium was reached within a 4-day period after addition (Dunn, 1943) and has since been updated (Kissel et al., 2012). An equation can be applied to a 30-min measurement to estimate a longer buffering reaction and a predictive equation was

created by UGA calibrated from US soils (Kissel et al., 2012; Sikora & Moore, 2012; Thompson et al., 2010). This method is a promising alternative method for routine lab use with some further evaluation required (Nguyen, 2023). Established charts (FAS, 2019) and proven models (Rothamsted Research, 2022) are widely adopted for rapid value estimates. There is little evidence of analysis and comparison of LR methods on United Kingdom and Northern European, temperate climate soils. In a period where soil acidity is a growing issue on agricultural land in the United Kingdom, this research aimed to (1) test the accuracy of current LR methodologies to determine the optimum methods for use and (2) to analyse the main soil characteristics that contribute to LR values (Goulding, 2016; Holland et al., 2018; PNMG, 2018). The methods compared were the Scottish Agricultural College (SAC) look-up table, the RothLime model, SMP, Sikora and MM buffers, the UGA titration method and calculations, and a modelled titration calculation.

## 2 | MATERIALS AND METHODS

### 2.1 | Soil collection and characterisation

Forty-three soils were collected across Scotland representing 43.9% of combined grassland and arable land area by Soil Association (Appendices A–C). Field sites were selected if from permanent pasture or grassland within a rotation with a pH value below target for grassland and were selected using the James Hutton Institute Land Cover of Scotland 1988 survey data (Scottish Government, 2021). FAS recommend the optimum pH value range for mineral grassland soils is 6.0–6.2; therefore, 6.2 was used as the target pH value (FAS, 2019). Liming and soil sampling depth recommended is 20 cm for arable/cultivated soils and 7 cm for grassland soils (FAS, 2019); thus, a 7–20 cm sampling depth was used in sample collection. Approximately 5 kg of each soil was collected using a spade and bucket. Soils were oven dried at 60°C for a minimum of 3 days and tested for stable weight on the third day. Soils were then sieved to 2 mm and homogenised.

Of the 43 soils, three were excluded for high pH values after initial lab analysis. Compulsive and effective cation exchange capacity (C-CEC, E-CEC) (pH 7.0 C-CEC, pH of soil E-CEC) were determined using ammonium acetate and sodium chloride (Kissel et al., 2012). Base cations (Ca, Mg, Na and K) were measured by atomic absorption spectroscopy (novAA 800, Analytik Jena). Exchange capacity was measured as NH<sub>4</sub>-N by flow injection analysis (Foss FIAstar 5000). Base saturation was calculated as the percent of total exchange capacity filled by the sum of cumulative base cations. Loss on ignition (LOI) was determined by the difference in soil sample weight before and after ignition (500°C overnight) and calculated as a percent. Soil pH was measured after end-over-end shaking for 1 h and resting for 1 h using a 1:2.5 soil to 0.01 M CaCl<sub>2</sub> ratio in a 50-mL centrifuge tube (2-Star Benchtop pH Meter, Thermo Scientific). Elemental analyses of carbon (C) and nitrogen (N) were measured using total combustion (FlashEA 1112 NC, Thermo Fisher Scientific). Soil organic matter (OM) was estimated by multiplying percent C by 1.724 (Pribyl, 2010).

## 2.2 | Ca(OH)<sub>2</sub> titration method

### 2.2.1 | Assay 1: four additions of Ca(OH)<sub>2</sub>

A standard method for making the saturated Ca(OH)<sub>2</sub> solution was used (Kissel et al., 2012). Soil-solution pH was measured in a 1:1 suspended mixture (soil:CaCl<sub>2</sub>, 20 g:20 mL in a 100-mL glass jar) using a magnetic stirrer during initial CaCl<sub>2</sub> mixing, Ca(OH)<sub>2</sub> solution addition and each pH measurement (Thermo Scientific Orion3 Star Benchtop pH meter). Soil-solution mixtures were measured to the nearest 0.01 pH value. In this procedure, an initial pH was measured (pH<sub>1</sub>) 30 min after soil and CaCl<sub>2</sub> were mixed, then a 2.4 mL saturated Ca(OH)<sub>2</sub> solution was immediately added to the mixture while stirred. At the time of saturated solution addition, soils were stirred for approximately 30 s and were set aside for 30 min before the second pH measurement (pH<sub>2</sub>) was taken while stirred. After pH<sub>2</sub> was measured, the same process was repeated twice (pH<sub>3</sub> and pH<sub>4</sub>). Three replicates each of both Ca(OH)<sub>2</sub>-added soils (limed) and control soils were performed for each soil. Calculations of the LR were based exclusively on the lime treatment response, that is the control soil change in pH over the time was subtracted from the lime-treated soil change in pH. This was to isolate the treatment pH response and to remove the natural soil pH response to incubation from further calculations. In the first of the three titration analyses, three replicates of sand with and without Ca(OH)<sub>2</sub> solution were run alongside the soils. The sand check as a soil standard was not deemed necessary after the first titration batch, the soils responded well to the method and the sand did not buffer the added Ca(OH)<sub>2</sub> solution.

Following the UGA methodology, a final LR was determined by first translating the difference in pH between pH<sub>2</sub> and pH<sub>1</sub> into a lime buffering capacity (LBC) value at 30 min (LBC<sub>30 min</sub>, Equation 1) (Kissel et al., 2012; Sikora & Moore, 2012; Thompson et al., 2010). LBC is the measure of acidity to be neutralized to raise the soil pH by 1.0 unit in mg CaCO<sub>3</sub>/kg soil (Kissel et al., 2009). In Equation (1), *V* is volume of the solution used (2.4 mL), *M* is molarity of the solution (saturated Ca(OH)<sub>2</sub> at 0.023 M) and *EQ* is equivalent weight of CaCO<sub>3</sub> (100 mg CaCO<sub>3</sub> mmol<sup>-1</sup>), soil weight (0.02 kg) and pH<sub>1&2</sub>. This LBC<sub>30 min</sub> value is translated into an estimate for an equilibrium LBC value, categorised by Equation (2) or C for LBC values either less than or greater than 250 mg CaCO<sub>3</sub> (LBC<sub>equilibrium</sub>). The LR value suggested for field application (t acre<sup>-1</sup> 100% effective neutralizing value [ENV] CaCO<sub>3</sub>) is calculated by translating Equation (2) or (3) to Equation (4), determining how far the initial pH (pH<sub>1</sub>) was from the target field pH (pH<sub>optimum</sub>). The initial pH (pH<sub>1</sub>) was measured in 0.01 M CaCl<sub>2</sub> (Liu et al., 2005). As the target pH for grassland mineral soils is 6.2 measured in water, pH<sub>optimum</sub> used in Equation (4) was 5.6, using the commonly used 0.6 value to represent the difference between CaCl<sub>2</sub> and water pH measurement methods (Kissel et al., 2009). This method was developed in the state of Georgia in the United States and the Equation 5 value is in lb acre<sup>-1</sup>: Equation (4) × 2 to equate ppm to pp2m to estimate 2 million pounds of soil in a 6-in. plough depth per acre and 1.5 to equate pure CaCO<sub>3</sub> to agricultural lime which has reduced particle reactivity. Equation (5) is calculating a 6 in. (15.24 cm) ploughing

depth and must be adjusted for a different depth. Equation (6) converts Equation (5) into t ha<sup>-1</sup> for UK use: lb acre<sup>-1</sup> divided by 2000 for t acre<sup>-1</sup>, multiplied by 2.24 to get t ha<sup>-1</sup>.

$$LBC_{30\text{min}} = (V \times M \times EQCaCO_3) / (\text{soilwt} / (pH_2 - pH_1)) \quad (1)$$

$$LBC_{30\text{min}} < 250 : LBC_{\text{equilibrium}} = (LBC_{30\text{min}} \times 3.67) - 188.3 \quad (2)$$

$$LBC_{30\text{min}} \geq 250 : LBC_{\text{equilibrium}} = LBC_{30\text{min}} \times 2.90 \quad (3)$$

$$LRmgCaCO_3kg^{-1}\text{soil} = LBC \text{ Equation}(3)\text{or } (4) \times (pH_{\text{optimum}} - pH_1) \quad (4)$$

$$LRlbacre^{-1} = \text{Equation}(4) \times 2 \times 1.5 \quad (5)$$

$$LRtha^{-1} = (\text{Equation}(5)/2000) \times 2.24 \quad (6)$$

### 2.2.2 | Assay 2: 96-h equilibration

The 96-h equilibration method followed the same pH<sub>1</sub> and pH<sub>2</sub> steps of the four aliquot additions but instead of further lime additions, the pH<sub>2</sub> value was measured repeatedly over a 96-h period to determine when the pH value equilibrated. Time points were as follows: 2, 4, 8, 12, 24, 36, 48, 60, 72, 84 and 96 h (Thompson et al., 2010). The soils were stored in an incubator at 25°C between measurements. This method was performed three independent times. Output data was converted to 100% ENV CaCO<sub>3</sub>. Equilibrium was the point at which the pH value stopped increasing within the 96-h period.

### 2.2.3 | Assay 3: UGA and modelled calculations

The average of the 72–96 h LR values of the three measurement events in the 96-h equilibration is considered the ‘titration’ method hereafter. A model equation specific to the chosen soils was created by regressing the average LBC’s from 72 to 96 h of the three repeated events against the 30 min LBC values. The regression equation was applied to the 30 min LBC values from the initial four aliquot assay experiment for each soil ( $y = (LBC_{30\text{min}} \times 1.3543) + 431.19$ ).

## 2.3 | SMP, Modified Mehlich, Sikora

The methods for preparation of the SMP, MM and Sikora buffers followed standard procedures (Sikora & Moore, 2012). The SMP buffer method contained 0.013 M *p*-nitrophenol, 0.015 M potassium chromate, 0.36 M calcium chloride, 0.0126 M calcium acetate and 0.019 M triethanolamine at a pH value of 7.5 (Sikora & Moore, 2012). The Sikora buffer method contained 2 M potassium chloride, 0.89 M acetic acid, 0.031 M MES, 0.014 M imidazole and 0.07 M triethanolamine at a pH value of 7.7 (Sikora & Moore, 2012). The MM buffer method

contained 0.043 M acetic acid, 0.034 M triethanolamine, 0.8 M ammonium chloride, 0.16 M calcium chloride dihydrate and 0.06 M disodium glycerophosphate at a pH value of 6.7 (Sikora & Moore, 2012).

Sikora and MM buffer methods used  $10 \pm 0.01$  g of soil, and SMP used  $5 \text{ g} \pm 0.01$  of soil using 50 mL plastic centrifuge tubes. The pH measurements were at 1:1 soil:H<sub>2</sub>O ratio, shaken vigorously for 15 s and allowed to sit for 15 min before the pH value was measured while swirling. After the first pH measurement, 10 mL of the appropriate buffer solution was added to the sample. For the Sikora and SMP buffers, samples were placed on the end-over-end shaker for 15 min and rest for 15 min before the second pH measurement. For MM, the mixture was gently stirred for 5 s and rest for 30 min before the second pH measurement.

Calculations for the buffers were performed using both US state-specific calculators and original author calculations (Mehlich, 1976; Shoemaker et al., 1961; Sikora, 2006; Sikora & Moore, 2012; Washington State University, 2022). The MM, SMP and Sikora original author LR rates were calculated using tables adapted by Sikora and Moore (2012) (Appendix D; Shoemaker et al., 1961). The LR values in  $\text{t acre}^{-1}$  were calculated for each soil for both pH 6.0 and 6.4, converted to  $\text{t ha}^{-1}$ , these values were then averaged to represent the target pH value 6.2. Output data was converted to 100% ENV CaCO<sub>3</sub>.

## 2.4 | RothLime

The model required input of soil pH (H<sub>2</sub>O 1:2.5), target pH (6.0 for grassland, 6.5 for arable), liming material and soil type (peats, organic, medium/clay, light and sand). The output value is  $\text{t ha}^{-1}$  of the selected lime material. In this analysis, the data for each soil was input into the model and the LR recorded. The target pH was manually input at 6.2. As a number of the soils chosen in this analysis and the scope of this research aims to include fields that are either permanent grasslands or grasslands within an arable rotation, values of both grassland and arable soils were recorded and then averaged. Output data was converted from 50% ENV ground limestone or chalk to 100% ENV CaCO<sub>3</sub>.

## 2.5 | SAC chart (look-up table)

The SAC chart method (a look-up table) required inputs of initial soil pH (1:2.5 H<sub>2</sub>O) and the choice of either grassland or arable management (Appendix E). The values of both grassland and arable soils were recorded and averaged. Output data was converted from 50% ENV ground limestone or chalk to 100% ENV CaCO<sub>3</sub>.

## 2.6 | Statistical analysis

Statistical analyses were performed using Minitab 19.2020.1 and R 4.1.0 (R Core Team, 2022). The packages 'randomForest' (Liaw & Wiener, 2002) and 'ranger' (Wright & Ziegler, 2015) were used to carry out the random forest (RF) analysis, whereas 'tidyverse' (Wickham et al., 2019)

and 'ggplot2' (Wickham, 2016) were used to format the data and create graphs. The packages 'broom' and 'styler' were also used to format the data. In this analysis, LBC was considered the main soil response variable responsible for calculating the LR as it is a value determined by the combination of site-specific soil characteristics. The LBC was therefore the response variable utilised in the RF. The RF analysis was performed to determine the main contributing factors to the measure of LBC within a soil using routinely measured soil characteristics. The RF variables included: LR ( $\text{t ha}^{-1}$ ), compulsive and effective CEC (C-CEC, E-CEC) and base saturation (C-BS, E-BS), percent clay, sand and silt, percent LOI, percent OM (percent C  $\times$  1.725), percent N, C:N ratio and initial pH (CaCl<sub>2</sub>). Due to missing data points for five soils in various categories of soil characteristics, the RF analysis was performed on 38 of the 43 soils as 'ranger' does not recognise NA values.

Soil liming results were regressed to determine the variability of the liming methods. Regressions were also performed in a stepwise approach to determining a Scotland-specific equation to apply to the 30 min method to estimate full equilibration values. Regression figures were created with Sigmaplot 14.0.

After analysis, three soils were excluded, and the following data represents the final 40 soils analysed. In a number of instances in soil collection, a set of soils were collected from multiple fields within a given farm. This occurred if fields were under different management or had differing soil associations. Those excluded were similar in pH to their site counterparts.

## 3 | RESULTS

### 3.1 | Ca(OH)<sub>2</sub> titration method

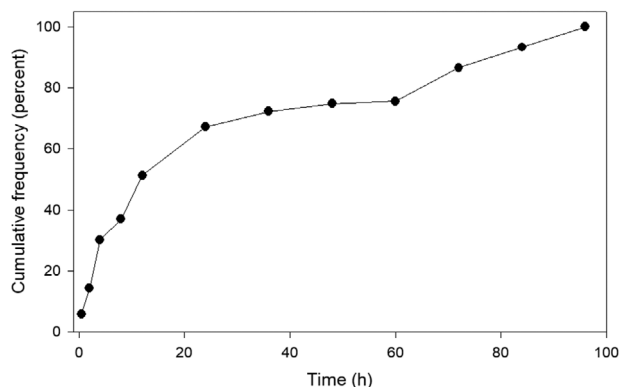
#### 3.1.1 | Assay 1: four additions of Ca(OH)<sub>2</sub>

The four-point pH response to the four measurement points indicated a linear response of  $R^2 \geq 0.97$  for the 40 soils measured.

#### 3.1.2 | Assay 2: 96-h equilibration

Equilibration of soils appeared to fall into two groupings in the 96-h period. By 24 h, 50% of the soil samples had reached an equilibrium pH value (the point at which soil reached approximately 100% of their total equilibrium LBC), there was no further increase in the proportion of soils reaching equilibrium until 72 h. Those remaining samples reached an equilibrium pH value within the time frame of this incubation experiment between 72 and 96 h (Figure 1). The time period of estimated equilibration for all samples was between 72 and 96 h.

The initial pH values (Table 1(A)), the pH values 30 min after lime addition (Table 1(B)), and the LR values 72–96 h were regressed within their groups (Table 1(C)) to check for potential variances among the three testing events. The pH values were strongly correlated in the first, second and third events for the initial pH (all  $R^2 > 0.99$ ) and 30 min after lime addition (all  $R^2 > 0.97$ ). The calculated LR at 72–96 h regres-



**FIGURE 1** Cumulative frequency of the time points at which the 40 soil samples reached 100% of their total equilibrium lime buffering capacity (LBC) within the 96-h time period.

**TABLE 1** (A) Correlations of 1st, 2nd and 3rd replicated measurement of initial pH  $R^2$  value; (B) 30 min pH  $R^2$  value; (C) 72–96 h liming requirement (LR)  $R^2$  value; (D) 30 min LR  $R^2$  value.

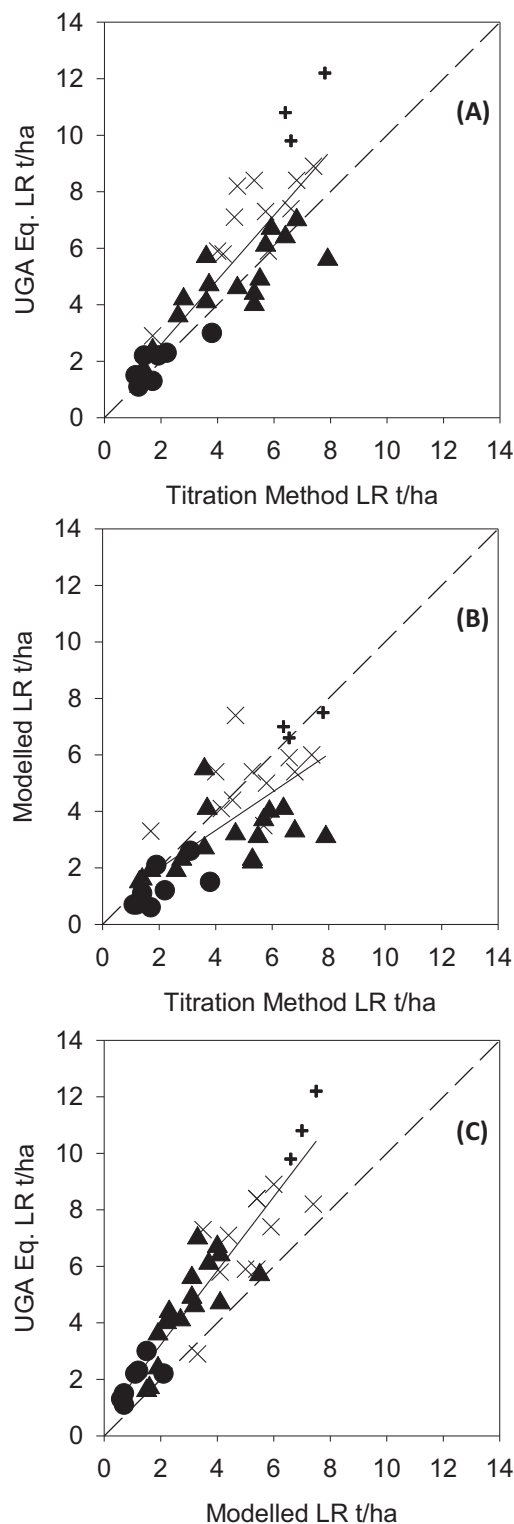
(A)	1st incubation initial pH	2nd incubation initial pH
2nd incubation initial pH	0.991	
3rd incubation initial pH	0.995	0.995
(B)	1st incubation 30 min pH	2nd incubation 30 min pH
2nd incubation 30 min pH	0.973	
3rd incubation 30 min pH	0.982	0.987
(C)	1st incubation 72–96 h LR	2nd incubation 72–96 h LR
2nd incubation 72–96 h LR	0.558	
3rd incubation 72–96 h LR	0.566	0.618
(D)	1st incubation 30 min LR	2nd incubation 30 min LR
2nd incubation 30 min LR	0.902	
3rd incubation 30 min LR	0.854	0.862

Note: All correlations are significant at a  $p$ -value of  $\leq 0.05$ .

sions for the three events were  $R^2 = 0.55$ – $0.62$ . The 30 min pH values were calculated up to LR values, and the events were regressed for correlation (Table 1(D)). The LR  $R^2$  values ranged from 0.85 to 0.90 for LR values compared to 0.97 to 0.98 for the pH values of the same data.

### 3.1.3 | Comparison of the LR based on equilibrium versus the UGA equation modelled LR

LRs calculated from Equation D using  $LBC_{30\text{ min}}$  values (hereafter referred to as UGA Equation) were regressed against the titration method LR's (Figure 2A). The UGA equations overestimated the LR of these soils but were well correlated ( $R^2 = 0.72$ ,  $p < 0.001$ ). Modelled LR's were regressed against titration LR's (Figure 2B), the modelled val-



**FIGURE 2** (A) Regression of titration method liming requirement (LR) results against the UGA adjustment equation for equilibrium compensation ( $R^2 = 0.72$ ,  $p < 0.001$ ), (B) equilibrium LR from the 72–96 h average period regressed against the modelled LR ( $R^2 = 0.52$ ,  $p < 0.001$ ) and (C) UGA equation calculated LR from regressed against the modelled LR ( $R^2 = 0.85$ ,  $p < 0.001$ ). Legend symbols designating initial soil pH values recorded in  $CaCl_2$ : + = pH 3.5–4.0, x = pH 4.0–4.5, ▲ = pH 4.5–5.0, ● = pH 5.0–5.5, dash line is the 1:1 line and the solid line is best fit.

ues better predicted the value range, but values were less correlated ( $R^2 = 0.52, p < 0.001$ ) (Figure 2B). The modelled LR was then regressed against the UGA equations which were well correlated in their values but the UGA equation overestimated LR's ( $R^2 = 0.85, p < 0.001$ ) (Figure 2C). Full comparison of regressions found in Appendix F.

### 3.2 | Buffers

LR values of each buffer were regressed against titration values. The MM buffer underestimated the LR for the original calculations ( $R^2 = 0.30, p = 0.003$ ; Figure 3A). The LR values were highly overestimated for the original SMP buffer calculations ( $R^2 = 0.15, p < 0.001$ ; Figure 3B) and the Sikora buffer original calculations ( $R^2 = 0.27, p < 0.001$ ; Figure 3C). Summary table of regressions can be found in Appendix F.

### 3.3 | RothLime, SAC chart

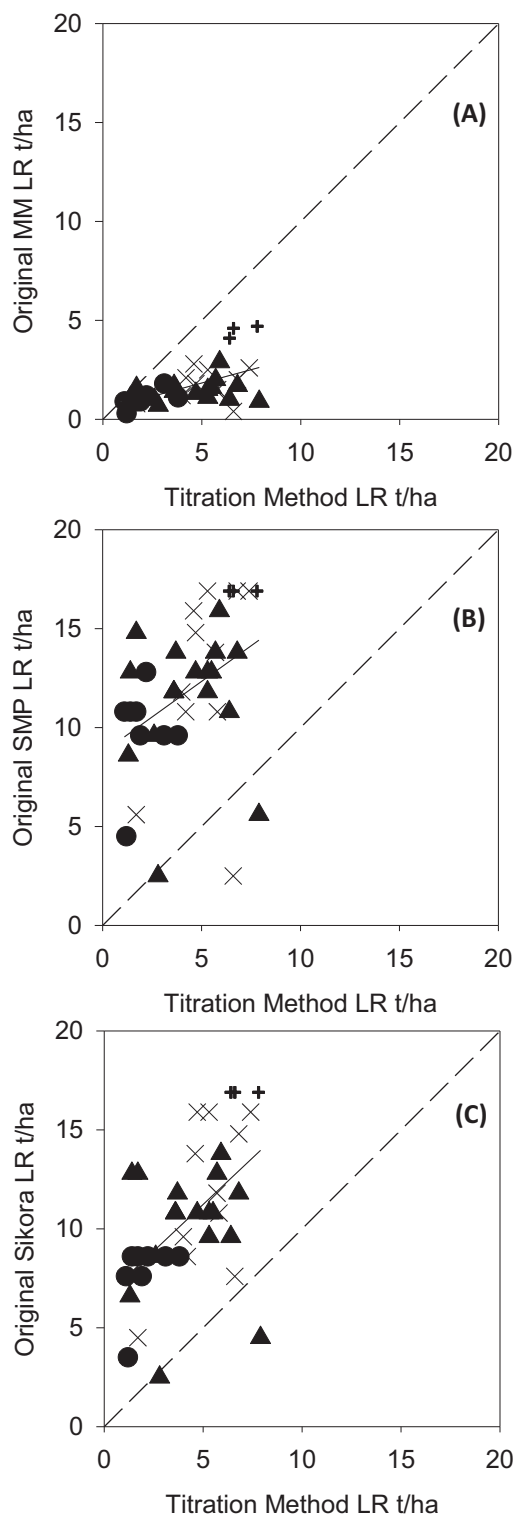
The output data from Rothlime and the SAC chart are defined values rather than continuous data points. The RothLime model values were poorly correlated to the titration values ( $R^2 = 0.33, p < 0.001$ ; Figure 4A). RothLime overestimated LR values when soils were initially high in pH. The SAC chart values were moderately correlated to the titration method values ( $R^2 = 0.43, p < 0.001$ ). Lower initial pH values resulted in underestimated LR's (Figure 4B).

### 3.4 | Soil characteristics

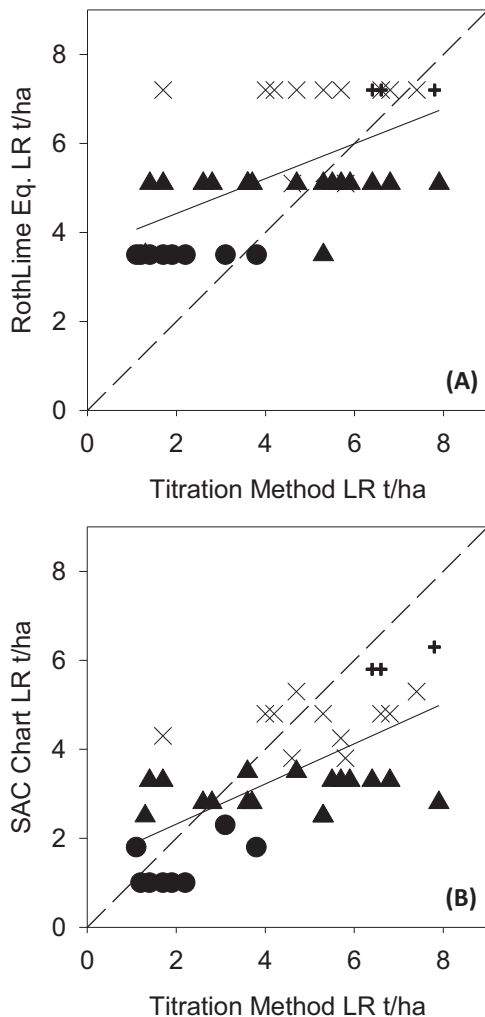
The relationships between LBC and soil characteristics were analysed using RF. The number of trees providing the lowest error rate was 9, with an average LBC error of 489.54. The first three variables of importance were C-CEC, E-CEC and LOI of the 14 variables analysed (Figure 5). The least important variables were sand, clay and silt.

### 3.5 | All methods

The LR values of  $\text{CaCO}_3 \text{ t ha}^{-1}$  for each method were compared (Appendix B). The titration method refers to the 72–96 h LR values averaged over the three data collection events. The titration ( $4.3 \text{ t ha}^{-1}$ ,  $\text{SD} = 2.0$ ), RothLime ( $5.3 \text{ t ha}^{-1}$ ,  $\text{SD} = 1.4$ ), SAC ( $3.3 \text{ t ha}^{-1}$ ,  $\text{SD} = 1.4$ ) and MM ( $1.6 \text{ t ha}^{-1}$ ,  $\text{SD} = 1.0$ ) methods yielded lower mean LR values across all the soils with less variability when compared to UGA ( $5.3 \text{ t ha}^{-1}$ ,  $\text{SD} = 2.8$ ) and Thompson equations ( $7.7 \text{ t ha}^{-1}$ ,  $\text{SD} = 4.9$ ), and Sikora ( $10.5 \text{ t ha}^{-1}$ ,  $\text{SD} = 3.7$ ) and SMP ( $11.7 \text{ t ha}^{-1}$ ,  $\text{SD} = 3.9$ ) buffers. The titration method values were regressed against the remaining methods (Appendix F). Though the values of the titration method were most comparable to the RothLime and SAC methods, the  $R^2$  correlations were 0.58 and 0.66 which were moderately correlated. Methods that resulted in high values with greater standard deviation compared



**FIGURE 3** (A) Regression of the titration liming requirement (LR) values against the original Modified Mehlich (MM) values ( $R^2 = 0.30, p = 0.003$ ). (B) Against the original Shoemaker, McLean and Pratt (SMP) values ( $R^2 = 0.15, p < 0.001$ ). (C) Against the original Sikora values ( $R^2 = 0.27, p < 0.001$ ). Legend symbols designating initial soil pH values recorded in  $\text{CaCl}_2$ : + = pH 3.5–4.0, x = pH 4.0–4.5, ▲ = pH 4.5–5.0, ● = pH 5.0–5.5, dash line is the 1:1 line and the solid line is best fit.



**FIGURE 4** (A) Regression of the titration liming requirement (LR) values against the RothLime model output LR ( $R^2 = 0.33$ ,  $p < 0.001$ ). (B) Against the Scottish Agricultural College (SAC) chart model output LR results ( $R^2 = 0.43$ ,  $p = < 0.001$ ). Legend symbols designating initial soil pH values recorded in  $\text{CaCl}_2$ : + = pH 3.5–4.0, x = pH 4.0–4.5, ▲ = pH 4.5–5.0, ● = pH 5.0–5.5, dash line is the 1:1 line.

to the titration method were more strongly correlated. Regressed against titration, the Sikora buffer had the highest correlative relationship ( $R^2 = 0.82$ ) followed by SMP ( $R^2 = 0.76$ ) and the UGA equation ( $R^2 = 0.72$ ) but highly over and underestimated LR values.

## 4 | DISCUSSIONS

### 4.1 | $\text{Ca}(\text{OH})_2$ titration

#### 4.1.1 | Four additions of $\text{Ca}(\text{OH})_2$

The strong regression in four aliquots for all soils (all  $R^2 \geq 0.97$ , not presented) confirmed that the two-point titration method matches the linearity observed in more than two aliquots (Liu et al., 2005). Previous work established that the two-point titration method gave adequate

resolution for and significantly correlated to the 3-day incubated values with 88% accuracy, though the incubation was performed in water and the titration was performed in  $\text{CaCl}_2$  ( $R^2 = 0.93$ ,  $p < 0.001$ ,  $n = 17$ ) (Liu et al., 2005). This research, in part, was a preliminary experiment to analyse the efficacy and applicability of the titration method for use on UK soils. In the work reported here, results confirm that the two-point titration method could be confidently used for routine LR testing on a range of soil characteristics. A conversion factor to compensate for equilibrium needs to be further developed.

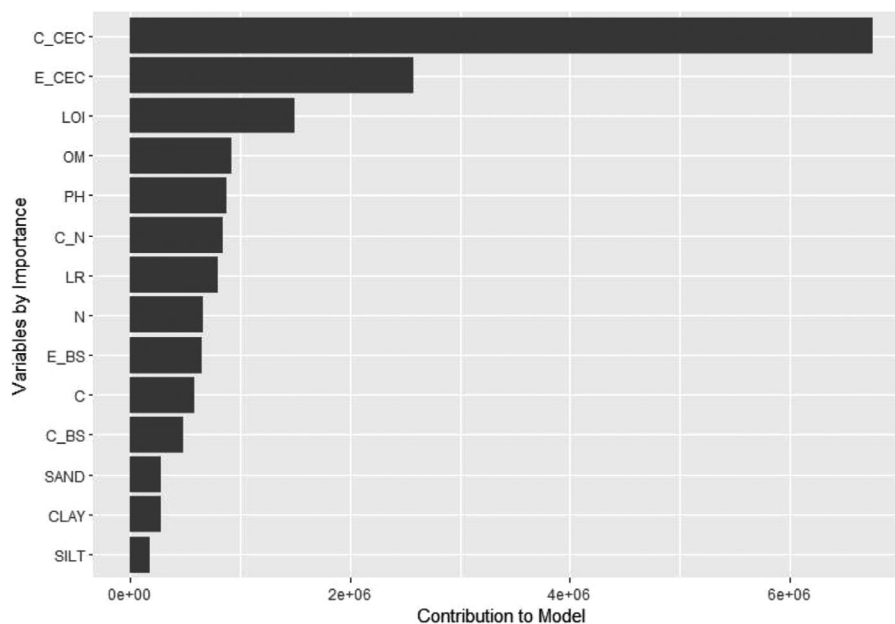
#### 4.1.2 | 96-h equilibration

Regression values between the first, second and third 72–96 h equilibration events suggest this method is approximately 50%–60% accurate when replicated. The reduction in replication resolution may be due to any number of factors such as OM variability or particle size settling in the larger dried soil sample source (5 kg) which was sub-sampled. Samples were held in an incubator between measurements, and it is unlikely that temperature played a role in variability.

Most of the samples were equilibrated by 72–96 h, similar to past research (Thompson et al., 2010). When the 96-h equilibration procedure was applied to a range of North Carolina soils, the majority soil LBC values equilibrated to 95% of total LBC by 40 h (Thompson et al., 2010). It was speculated that outlier soils that did not reach 95% by 40 h had higher iron content which accounted for the continuous rise in pH over time (Thompson et al., 2010). By contrast, approximately 50% of soil LBC values equilibrated to 95% of total values by 24 h in this experiment. The remaining soils were equilibrated between 72 and 96 h. These differences between soils were not correlated to any single soil parameter and it would be speculated that these two groupings of soils could be due to iron content as seen in Thompson et al. (2010).

The 72–96 h equilibration period was determined to be the most equilibrated LR method by which to compare other methods. This was supported by previous work which used both a 96-h experiment and an incubation experiment to calibrate the calculations for equilibrium (Godsey et al., 2007; Kissel et al., 2007, 2012; Thompson et al., 2010). Five-day incubations were contrasted to the quick two-point titration method in previous research and LBC-regressed results were strongly correlated ( $R^2 = 0.91$ ,  $n = 67$ ) (Thompson et al., 2010). Results of our 5-day equilibration LR results regressed against the two-point titration results yielded a much weaker correlation. The poor correlation in the three comparative regressions for the 79–96 h period may be attributed to the small degree of change in pH and the subsequent increased degree in LBC error as a result. It is suggested for future development of the method that testing an increased volume of the saturated  $\text{Ca}(\text{OH})_2$  solution is necessary. An increased pH response between  $\text{pH}_1$  and  $\text{pH}_2$  would likely reduce the LBC error due to its reduced proportion of the total change in pH.

It has been reported previously that  $\text{LBC}_{30 \text{ min}}$  values ranged from 100 to 500  $\text{mg CaCO}_3 \text{ kg}^{-1}$  in soils from Georgia, USA (Kissel et al., 2012). For the Scottish soils used, the  $\text{LBC}_{30 \text{ min}}$  ranged from 369 to 2070  $\text{mg CaCO}_3 \text{ kg}^{-1}$  with a mean LBC of 1182  $\text{mg CaCO}_3 \text{ kg}^{-1}$ .



**FIGURE 5** The top 14 important soil characteristic variables in order of importance with the response variable of lime buffering capacity (LBC) from random forest analysis. Variables include compulsive and effective cation exchange capacity (C/E\_CEC), loss on ignition (LOI), organic matter (OM), soil pH in  $\text{CaCl}_2$  (PH), carbon to nitrogen ration (C\_N), liming requirement (LR), nitrogen (N), effective base saturation (E\_BS), carbon (C), compulsive base saturation (C\_BS), sand, clay and silt.

These differences would further indicate the requirement of creating equations specific to temperate, wet climate soils which are high OM (Poggio et al., 2013).

The average  $\text{LBC}_{30\text{ min}}$  values represented 57.4% of the equilibrated values. The underrepresentation of reduced values in the 30 min measurements as opposed to incubated or equilibrated measurements has been attributed to the slow release of  $\text{H}^+$  ions into the solution over time and under temperatures not reproducible in a 30-min period (Godsey et al., 2007; Liu et al., 2004). As compared to a 60-day incubation with  $\text{CaCO}_3$ , previous  $\text{Ca(OH)}_2$  two-point titration research measured an average of 45% of the LR when trying to achieve a target pH value ( $n = 16$ ) (Godsey et al., 2007). A conversion factor of 2.2 was applied to the two-point titration results in that experiment. Comparatively, a 1.74 conversion factor would be used in this research. In other work, two-point titration LR's regressed against 3-day incubation accounted for 80% of the LR by the quick procedure (Liu et al., 2004). The 57% (of two-point titration representing equilibration in 96 h) results in this experiment fell between 45% for 60-day incubation and 80% for 3-day equilibration recorded in previous literature.

#### 4.1.3 | UGA and modelled calculations

The UGA equation was closely correlated to the equilibration adjustment equation created in this experiment but the UGA equation overrepresented LR values. Low pH soils were especially overestimated by the UGA equation. The UGA equation also overrepresented LR values when regressed against the titration method LR's. The moderate correlation between the equilibration adjustment equation in

this experiment to the titration values indicates further development is required for an equilibration equation. The mean modelled LR and the titration method for the 40 soils were within similar ranges for the LR model and for the titration method.

By analysing soils from outside the United States but using equations calibrated for US-based soils, results from Equation (4) were not expected to yield exact correlations compared to previous literature but they were expected to be within range (Kissel et al., 2012). The main analysis of the UGA method outside of the United States was on Irish soils (Tunney et al., 2010). Tunney et al. (2010) acknowledged that they did not perform equilibration or incubation themselves but sent samples for analysis to the UGA lab and used UGA calculations. The equilibration assay of the UGA method when applied to non-Georgia soils is fundamental to ensure the correct equilibration values are being cross-checked against the two-point titration calculations.

In summary, the titration method is a potentially valuable laboratory alternative that could be utilised when precise LR values are required. The development of a more accurate equilibration equation specific to Northern European, temperate climates and soils high in OM would be suggested. In a following experiment, it would be suggested to develop this equation using a greater number of soils with a greater diversity of soil associations from across the United Kingdom. It would also be suggested that calculations be grouped into LBC groups more specific to high OM soils. The ranges utilised by the UGA calculations (Equations 3 and 4) were far too small ( $\text{LBC} < 250$  or  $\geq 250 \text{ mg CaCO}_3 \text{ kg}^{-1}$ ) which suggests the soils used for developing this method had very low buffering capacities due to low clay content and/or OM. Previous work on LBC method analyses utilised six points for the titration curve which showed an exponential soil pH response to  $\text{Ca(OH)}_2$  (Vogel et al., 2020).



It would therefore be suggested that any further investigation into the method using UK soils should increase the quantity of titration points to measure a wider soil pH response to lime additions. Incubations utilised to create a model to convert  $LBC_{30\text{ min}}$  to  $LBC_{\text{equilibrium}}$  should utilise at 5 days (120 h)  $\text{Ca}(\text{OH})_2$  incubation (Thompson et al., 2010). This is to avoid any ammonification or nitrification experienced in a longer term incubation, seen in Liu et al (2008).

## 4.2 | Buffers

The SMP buffer was created for Ohio soils but has since been adopted for routine use in other states in the United States and outside of the United States (Godsey et al., 2007; Shoemaker et al., 1961; Tunney et al., 2010). The updated version of the SMP buffer, the Sikora buffer, was adapted and calibrated using soils in Kentucky, USA (Sikora, 2006). The MM buffer was an update of the original Mehlich buffer and was calibrated for use in Pennsylvania, USA (Hoskins & Erich, 2008; Mehlich, 1976; Wolf et al., 2008). When regressed against titration values, both SMP ( $R^2 = 0.15$ ) and Sikora ( $R^2 = 0.27$ ) calculations highly overestimated the LR recommendations. When Irish soils were used to regress titration and SMP methods, values were highly correlated ( $R^2 = 0.97$ ) (Tunney et al., 2010). The differences in correlations between the Irish soils and those in this study may be attributed to the range of pH values, soil texture and other soil characteristics, the lack of an equilibration check with Irish soils or other unknown factors.

Of the buffers tested, the methods were not found to be applicable for use on Scottish soils. The buffer methods were easy to undertake and consistent in their pH measurements, but LR calculators are calibrated to US soils (specifically Ultisols [USDA] and highly weathered soils with low buffering capacities) (McFarland et al., 2020). It has been suggested that the use of buffers should reflect the accuracy of buffer performance on target soil groups and the accuracy of buffer calibrations when compared to incubation (McFarland et al., 2020). The SMP buffer had been used as the LR method in Ireland since 1965 with an Irish-specific calibration factor ( $\text{LR} = 0.8 \times \text{SMP}$ ) (Tunney et al., 2010). Even with a reduction of 20% in values by using the Irish conversion factor, those soils in the higher LR range would be reduced from 15.9 to 12.7 t ha<sup>-1</sup>. If the Sikora or SMP buffers were desired to be used in climates that result in soils with high OM contents, the establishment of a similar calibration factor would be recommended. The high soil OM content likely contributes to the significant differences observed between US-calibrated LR's and the titration LR's as OM increases the measure of exchange sites and therefore buffering capacity. There is scope for the development of buffer calibration for use in the United Kingdom.

## 4.3 | RothLime and SAC chart

RothLime and SAC results both under and overestimated LR values compared to the titration values. The RothLime model overestimated the amount of lime required, especially when the initial pH was >5.0. The SAC chart underestimated the amount of lime required, especially

when soils were initially <4.5. The RothLime over and underestimation was observed by Tunney et al. (2010) when regressed against SMP buffer results, similarly when more acidity needed to be ameliorated. From these results it would be estimated that soils with greater initial acidity may require a more precise LR determination method due to a range of potential factors. Those factors could be, beyond the higher rate of available H<sup>+</sup> ions, the strength of retention of these ions which are not possibly expressed in a simple model. As RothLime and SAC produce rounded values to make it easy for on-farm calculations, the precision of the LR is reduced. The rounding of values makes the SAC and RothLime methods accessible but potentially hinders accuracy in environments where precision agriculture technologies would be utilised.

## 4.4 | Soil characteristics

Since liming aims to buffer the acidic ions on the exchange sites within a soil, the estimation of an LR is correlated to the measure of exchange sites. Exchange sites are determined by the surface area of soil (particle size from texture), the addition of further sites via OM, and exchangeable/nonexchangeable acidity within the soil. The main contributing soil factors to final LR values in previous research were OM (from the number of exchange sites it provides), the exchangeable and nonexchangeable Al<sup>3+</sup> content and pH-dependent exchange sites on clay (and therefore the proportion of sand) (Curtin et al., 2011; McFarland et al., 2020; Pionke et al., 1968; Ross et al., 1964; Ruehlmann et al., 2021). Stepwise multivariate analysis using OM, extractable Al<sup>3+</sup> or soil pH was able to predict LR to 72% accuracy compared to 90 days soil-lime incubations (McFarland et al., 2020).

The exchange capacity (C/E-CEC) and LOI variables were the top three variables with the greatest contribution to the analysis for the measure of LBC in the selected soils. Such variables are related to soil buffering capacity and the LBC measurement may be functionally used in place of combinations of other measures of exchangeability in future analysis. There is scope to utilise the LBC value as a proxy method for the various other methods. Variables contributing to the exchangeability of a soil could be added to the LR methods that exclude this information. Soil textures, measured through sand, clay and silt, were the least important variables in the RF analysis. Not anticipated in these results was that the per cent clay content of the soil not falling within the top five important variables range, though it is understood that OM has a greater effect on soil buffering capacity than clay content or type of clay (Nelson & Su, 2010; Vogel et al., 2020). This effect is due to the greater surface area supplied by OM and the pH-dependent variable charges of OM (Nelson & Su, 2010; Vogel et al., 2020).

## 5 | CONCLUSIONS AND RECOMMENDATIONS

Current LR methods used in Scotland and the United Kingdom underestimate the LR's of acidic UK soils which are critically historically under-managed. Agricultural requirements to reach UK climate targets include precision resource management and determining the accuracy

of amendment recommendation methods is a vital role in assuring best-practice. In instances where greater precision soil pH management is required, analyses beyond the common LR methods (SAC chart, RothLime) should be explored, such as the two-point  $\text{Ca}(\text{OH})_2$  titration method. The LBC value may be utilised in future analysis as an effective substitute for an overarching measure of exchangeability. If that is not desired, additional measurements of buffering capacity such as CEC and LOI could be added to the LR methods that exclude exchangeability information. The two-point  $\text{Ca}(\text{OH})_2$  titration method has scope to be an accurate, cheap and speedy alternative to current methods. A region-specific equilibration adjustment equation must be developed as this research was a thorough preliminary method analysis to assess the applicability of the method to UK soils. This development would require a greater number of soils from a greater diversity of soil types from the United Kingdom. It would be suggested that calculations based on a greater number of soils be grouped by buffering capacities ranges, similar to the UGA calculations.

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## DATA AVAILABILITY STATEMENT

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

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