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*Full Length Research Paper*

# Comparison of laboratory methods in predicting the lime requirement of acid soil in Wombera District, North western Ethiopia

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**Soil acidity is one of the major constraints of crop production in the Ethiopian highlands where precipitation is high enough to leach basic cations leaving the soil acidic. Liming is the major practice used to ameliorate the problem of soil acidity. Currently farmers use Ag lime in their fields to reduce soil acidity. However, the suitability of the existing methods of lime requirement (LR) has not yet being determined. Shoemaker-McLean-Pratt (SMP) single buffer, Adams-Evans buffer, Modified Mehlich buffer, titration with single addition of Ca $(OH)_2$, and exchangeable aluminum methods were evaluated using the $CaCO_3$ moist incubation method. The result revealed that the LR estimated by the buffer methods and titration highly correlated with the incubation LR since the correlation coefficient(r) was ≥ 0.98. The modified Mehlich buffer was better in predicting the LR on average, for target pH values of 5.5, 6.0, 6.5, 6.8 and 7.0 as r = 0.99 and as the standard error of estimate ($S_{y.x}$) 0.57, the minimum among the buffer methods. The modified Mehlich buffer should be calibrated further with the reference $CaCO_3$ incubation using a set of soils and a calibration equation to calculate the lime requirement.**

**Key words:** Acidity, lime requirement, calibration, buffer methods, incubation.

## INTRODUCTION

One of the major constraints of crop production in many regions of the world is soil acidity. Soil acidification or decrease in soil pH is primarily a natural process that is accelerated by human activities in crop production practices, as they use acid bearing fertilizers such as those containing nitrogen (Anderson et al., 2013). Soil acidity directly and indirectly influences the growth of plants; it affects the availability of plant nutrients, level of phytotoxic elements, and microbial activity and is a serious limitation to crop production in many regions of the world (Pagani and Mallarino, 2012).

Acid soils are found in large areas in both tropics and temperate regions. In Ethiopia, it is estimated that around 43% of the total cultivated land is affected by soil acidity (Yirga et al., 2019). Out of this percent, about 28.1% is dominated by strong acid soils (pH 4.1-5.5) (Yirga et al., 2019), and such soils are less fertile due to toxicity of Aluminum and Manganese and deficiencies of calcium, magnesium, phosphorus and molybdenum (Havlin et al. 2005). To correct this, acid soils are usually limed.

Soil pH referred to as the concentration of hydrogen ion in soil solution is necessary to determine whether to lime

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a soil or not. However, it is necessary to measure the buffer capacity of the amount of lime needed by the soil (Ketterings et al., 2010). Lime requirement is the amount of lime required to increase the pH of soil from an acidic condition to a value that is considered optimum for the desired use of the soil (Sims, 1996).

Various methods have been developed to estimate the LR of acidic soils: Soil-lime incubation, direct titration, use of buffer solution and exchangeable acidity/Al extracted with unbuffered salt. All the methods have their own strength and weakness. The soil-lime incubation with $CaCO_3$ is the ideal method to determine the LR of acid soils as it simulates field liming and is conducted in greenhouses or laboratories (Sims 1996). The method is commonly used to calibrate other methods (Hoskins and Erich, 2008; Shoemaker et al., 1961). However, it takes several weeks to complete the reaction between soil and lime applied and thus is not used for routine analysis (McLean, 1982). Buffer pH methods have been developed for a quicker assessment of LR. The Shoemaker-McLean-Pratt (SMP) buffer method (Shoemaker et al., 1961), Adams-Evans (AE) buffer method (Adams and Evans, 1962), and the modified Mehlich (MM) (Hoskins and Erich, 2008) are some of the commonly used buffer methods. All the methods relate the pH of the soil that is allowed to react with the buffer to the LR of a soil population.

According to McLean et al. (1966), the SMP buffer method is accurate and well adapted to soils requiring > 4480 kg/ha, having pH <5.8, soil organic matter (OM) < 10% and having appreciable quantities of soluble aluminum. However, for soils with low LR (<4480 kg/ha), high pH (pH>5.8), and high organic matter (>10%), the results have been inaccurate (McLean et al., 1966). In comparing SMP with other buffer solutions, it is revealed that it is applicable to a broad group of soils having a wide range in LR values (McLean et al., 1977). The Adams and Evans (A-E) buffer for LR determination was developed to measure the LR of low activity clays and course- textured soils that are dominated by kaolinite and sesquioxide soil minerals (Adams and Evans, 1962).

Modified Mehlich buffer was developed by Hoskins and Erich (2008) to simulate the original buffer except barium chloride is replaced by calcium chloride in the buffer to remove the protocols for hazardous waste disposal due to the presence of barium. The authors reported that the modified Mehlich (MM) buffer is better than SMP during routine usage; it eliminates hazardous waste and has fewer adverse effects on electrode degradation.

Soil-base titrations procedure entails titrating (equilibrating) a soil suspension with a basic solution such as calcium hydroxide [$Ca(OH)_2$ ] to estimate the amount of lime required (Liu et al., 2005; Barouchas et al., 2013). The direct titration procedures initially developed by Dunn (1943) using multiple rates of 0.002 M $Ca(OH)_2$ took 4-days and were found to be too time-consuming for use in routine soil testing. However, 3-day incubation with a $Ca(OH)_2$ is a widely accepted reference method (McConnell et al., 1990; Owusu-Bennoah et al., 1995). However, the titration method was adapted and used as a routine soil testing procedure for LR determination; it became an alternative to the buffer method (Liu et al., 2005).

Calculation of the LR based on the amount of exchangeable aluminum extracted using unbuffered salt is used around the world. It has been suggested that the amount of lime sufficient to neutralize the exchangeable aluminum is adequate to avoid possible aluminum toxicity which is described as the main yield limiting factor in highly weathered acid mineral soils (Kamprath, 1970; Van Lierop, 1990). Accordingly, liming such soils containing sufficiently high level of aluminum to pH 5.5 can significantly increase crop yield (Van Lierop, 1990). The method is popular in areas where crop production is limited by highly acidic aluminous soils and the availability of limestone is limited (Sims, 1996). However, routine analysis of exchangeable Al for LR estimation is a challenge due to time and cost (Ketterings et al., 2010).

Soil testing laboratories in Ethiopia mainly use the SMP single buffer or the classical exchangeable acidity/Al method as a routine soil testing method to determine LR. Nevertheless, data on the suitability of these methods in predicting the LR of soils of Ethiopia in general and Wombera District in particular are lacking. With this, it is important to identify accurate and rapid LR determination method that better suits routine analysis. Therefore, this study aims to meet the following objectives: (1) To compare the LR determination methods (SMP, Adams-Evans, Modified Mehlich, $Ca(OH)_2$ titration, exchangeable Al) with the reference (greenhouse incubation) method; (2) To select the method that correlates better with the incubation LR for different target pH values.

## MATERIALS AND METHODS

### Description of the soil sampling site

Wombera District (Wereda) is located in Metekel Zone of the Benishangul Gumuz Regional State, North Western Ethiopia. The district is located within 9°56´24.33´´ and 11°8´17.09´´ north latitude and 35°9´3.49´´and 35°55´35.51´´east longitude. The agroecological zone (AEZ) of the area belongs to the tepid sub-humid mid highlands (Ministry of Agriculture, 2005). The mean annual rainfall of the area is 1550 mm. The average maximum and minimum temperatures are 27 and 12°C, respectively. According to BCEOM (1998), the soil class of the area is Rhodic Nitisols. The topography of the area is undulating or hilly. Mixed farming system is practiced by the local society of the study area that involves animal husbandry and crop production. The major crops grown are :tef (*Eragrostis* tef), niger seed (*Guizotia abyssinica*).Other crops grown are wheat (*Triticum aestivum* L.), barley (*Hordeumvulgare* L.), and potato (*Solanum tuberosum* L.) (Figure 1).

### Soil sampling

Prior to soil sampling, farm plots having severe soil acidity problem

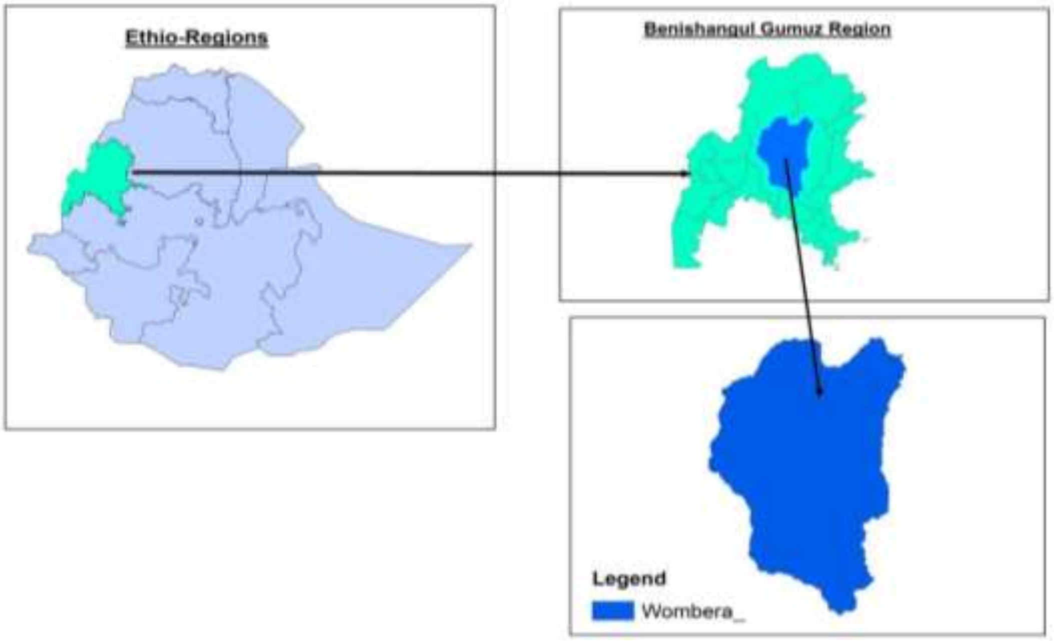

**Figure 1.** Location Map of Wombera Wereda.

(low pH) were screened from those that have less or no soil acidity problem. Following the selection of the farm plots, soil samples were collected from 0-20 cm depth at random to make one composite sample and a handheld auger was used to collect the samples. The composite soil sample was taken to the National Soil Testing Center where physicochemical analysis and incubation experiment was conducted. The composite soil sample was air dried ground and sieved to pass through a 2 mm sieve for physicochemical analysis and soil-lime incubation experiment. For total nitrogen and organic carbon determination, the soil was further pulverized and passed through 0.5 mm sieve.

### Physicochemical analysis

The soil physicochemical properties were measured prior to incubation and lime requirement determination. Soil particle size analysis was determined following Bouyoucos hydrometer (Bouyoucos, 1951). Soil pH in water (pH $_w$) and pH KCl were determined in a 1:2.5 suspension according to Van Reeuwijk (2002). Soil organic carbon (OC) was determined by the chromate acid oxidation method (Walkley and Black, 1934) and soil OM was calculated by multiplying percent OC by a factor of 1.724. Cation exchange capacity (CEC) was measured using the ammonium acetate method. Exchangeable acidity was determined by saturating the samples with 1 M KCl solution and titrated with 0.02 M NaOH as described by Rowell (1994). The exchangeable Al was determined from the same extract by applying 1 M NaF which formed a complex with Al and released NaOH; and then the NaOH was back titrated with a standard solution of 0.02 M HCl.

### Lime requirement methods

#### *Incubation method*

Three kilograms of soil was incubated in a greenhouse for a period of one month with different rates of analytical grade $CaCO_3$ including no liming (control). The rate of $CaCO_3$ to achieve target pH of 5.5 was first determined based on the soil-buffer pH measured and the corresponding LR value obtained from the adapted SMP table (Van Lierop, 1990). The rate of $CaCO_3$ was converted from tons/ha to gm/kg assuming the soil bulk density to be 1.4 gcm$^{-3}$ and incorporation depth to be 20 cm.

The amounts of $CaCO_3$ added in the incubation experiment were: 11.4, 17.1, 22.8, 28.5, 34.2 and 39.9 gm. Each of the lime rates were replicated three times. The various rates of $CaCO_3$ were first mixed thoroughly with the dry prepared soil using a spatula. A polyethylene pot was used in the incubation experiment (Figure 2). The mixtures were incubated while maintaining the field capacity. The temperature of the greenhouse ranged from 25-27°C. After the incubation period, the mixtures were air dried and the pH was determined in a 1:2.5 (soil-water) suspension.

### SMP single buffer method

The SMP buffer solution preparation and measurement of soil-buffer pH were done according to Watson and Brown (1998). The LR corresponding to the soil-buffer pH value for different target pH values was obtained from the table prepared by Shoemaker et al. (1961).

### Adams-Evans buffer method

The Adams-Evans buffer solution preparation, buffer pH measurement procedure, and determination of the LR were made as described by Sims (1996).

### Modified Mehlich buffer Method

The buffer solution preparation and determination of the LR in ton/ha were done using equations adapted from Sikora and Moore (2014).

**Figure 2.** Greenhouse incubation experiment.

### Single addition of Ca $(OH)_2$ method

Soil pH measurements and titration were performed in 1:1 soil-water ratio. The soil $Ca(OH)_2$ mixture was shaken for 5 min on an end to end shaker and left to stand for 25 min before taking the soil pH measurement (Liu et al., 2005). Successive 3 ml aliquots of 0.022 M Ca $(OH)_2$ were added and pH was measured until it reached 7.30. All determination was made in triplicate. Calculation of the titration slope and determination of the LR for different target pH were determined using the formula described by Liu et al. (2005) as follows.

The slopes of the titration curve were estimated using consecutive two pH readings [before and after the addition of 3 ml of Ca $(OH)_2$], and the LR was calculated using the equation;

$$LR = \frac{((Target\ pH - initial\ \ pH))}{b} \times \mathrm{f} \tag{1}$$

Where b is the slope of the relationship of pH vs. $Ca(OH)_2$ added and f is the factor.

The calculation of the titration slopes for the different target pH values were determined using a series of conversion equations adapted from Liu et al. (2005).

### Exchangeable aluminum method

The exchangeable Al determined withthe unbuffered salt was used to estimate the LR that would possibly increase the pH of the soil to 5.5. Accordingly, the LR for 20 cm soil depth and bulk density of 1.4 $gcm^{-3}$ was calculated by converting meq/100 g soil of exchangeable Al to kg/ha as illustrated in Equation 2.

$$LR = \frac{2.8{\times}10^{6}\ \ kg\ soil}{ha} \times \frac{meqAl}{100g\ soil} \times \frac{0.05gCaCO\,3}{meq} \tag{2}$$

### Data analysis

Duncan’s mean separation test was conducted to determine the completion of the incubation period using SAS/STAT User’s Guide (2008). Linear regression and correlation analysis were generated using Microsoft Excel (Microsoft Corporation, 2007).

## RESULTS AND DISCUSSION

### Physicochemical characteristics of the soil

The textural class of the soil is clay as illustrated by the high clay content (48%).The pH $H_2O$ and pH KCl were 5.2 and 4.14 respectively. The percent of the organic carbon content (5.00) and CEC (29.52 meq/100 g) of the soil are rated medium and high according to Landon (1991). The high clay content of the soil could be the reason for the high CEC value. The delta pH value of the soil was negative (-1.06), indicating that the soil had a net negative charge (CEC) in the clay colloid (Table 1).

### Lime incubation study

The amount of $CaCO_3$ applied in the incubation study resulted in an increase in soil pH value of at least 6.37 (Figure 3). Accordingly, the incubation study can be used to calibrate other methods. The decrease in pH of the control (no treatment) soil (5.15) from the pre-incubation pH level (5.2) could have been due to nitrification effect, which is noted as one of the main sources of soil acidity. The quantity of lime added versus pH response was plotted and fitted by non-linear (polynomial) regression. The lime requirement for the target pH values (5.5, 6.0, 6.5, 6.8 and 7.0) was calculated using the equation. The amount of lime required for the corresponding pH values was 2.72, 7.18, 12.52, 16.47, and 19.68 tons of lime per hectare. The graph clearly indicates that there was a gradual increase in the pH of the soil, as lime application increased up to pH 7.4 from where, it started to decrease. Similar results were reported by Shoemaker et al. (1961) and Hoskins and Erich (2008).

**Table 1.** Physicochemical properties of the soil.

| Sand (%) | Silt (%) | Clay (%) | OC (%) | pHW | pH KCl | ∆pH | EC ($dsm^{-1}$) | CEC (meq/100 g) | Ex. Acidity (meq/100 g) | Ex. Al (meq/100 g) |
|---|---|---|---|---|---|---|---|---|---|---|
| 16 | 36 | 48 | 5.00 | 5.20 | 4.14 | -1.06 | 0.079 | 29.52 | 2.33 | 2.19 |

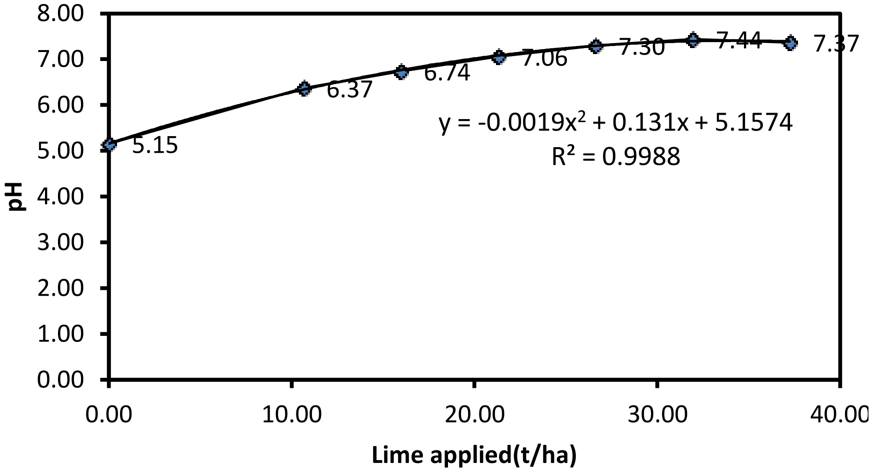

**Figure 3.** Lime response curve of the incubation experiment.

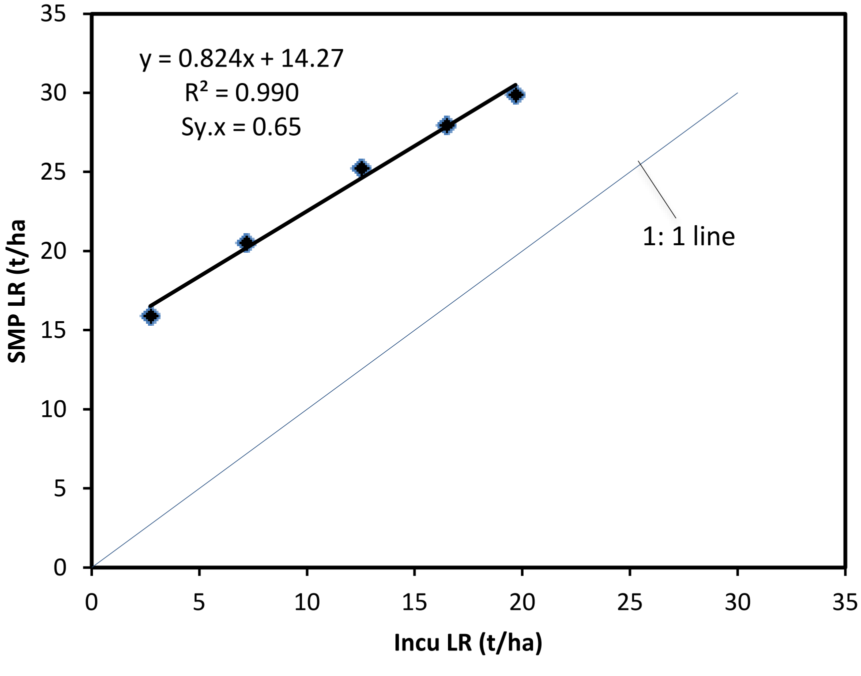

**Figure 4.** SMP versus Incubation LR in tons/ha.

## SMP buffer indicated vs. calcium carbonate incubation measured LR

The LR estimated using the SMP buffer pH value and incubation highly correlated ($r^2 = 0.99$). Nevertheless, the SMP overestimated the LR for all target pH values as indicated in Figure 4. The t-statistics in the regression analysis revealed that the slope of the regression equation was significantly different from one (*p*= 0.00041), and the intercept was also significantly different

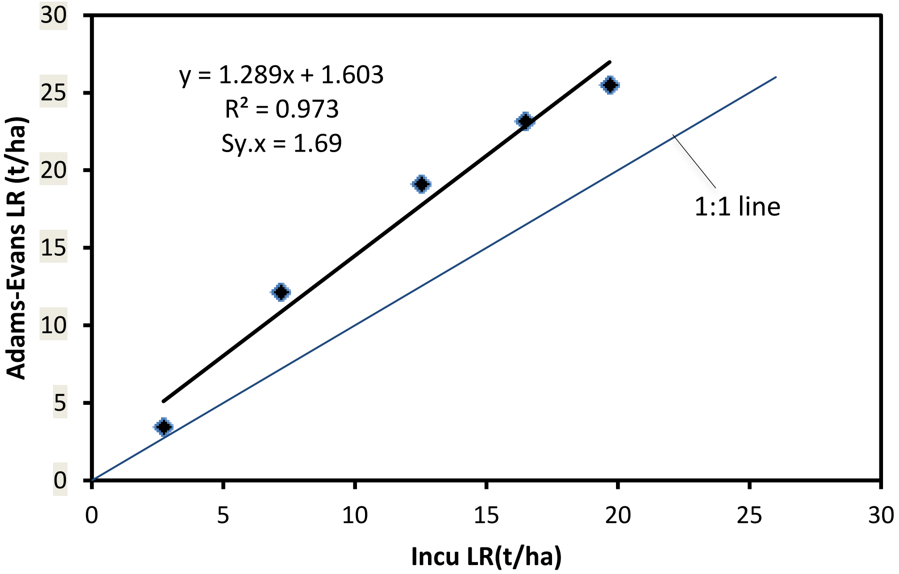

**Figure 5.** Incubation LR versus Adams-Evans estimated LR.

from zero (p = 0.0001). This indicates that, the SMP overestimated the LR in both the low and high LR values. Machacha (2005) found that the SMP single buffer method showed a high correlation (r= 0.80) between the LR predicted by the buffer method compared to the incubation LR; though it overestimated the actual LR for target pH of 6.5.

### Adams evans buffer estimated LR vs. incubation LR

The Adams-Evans buffer estimated LR was also highly correlated with the incubation LR, as indicated by the high correlation ($r^2$= 0.97) value (Figure 5). The t-statistics calculated in the regression analysis indicated that the slope of the regression equation (1.289) was significantly different from one (p=0.00187). However, the intercept (1.603) was not significantly different from zero (p = 0.398). This implies that the Adams-Evans buffer LR was relatively close to the incubation LR at low LR values (Figure 5). Nevertheless, as the LR value increases, the Adams-Evans estimated LR departs from the incubation LR. The standard error of estimates for the Adams-Evans was higher than the SMP. Thus, the regression equation poorly explains the variability in LR as compared to the SMP.

### Modified Mehlich (MM) buffer estimated LR vs. incubation LR

Similarly, the modified Mehlich buffer estimated LR was highly correlated with the incubation LR ($r^2$ = 0.989). In contrast to the SMP and Adams-Evans, the modified Mehlich (MM) narrowly underestimated the LR for target pH of 6.5, 6.8 and 7.0 and slightly overestimated the LR for target pH of 5.5 (Figures 6 and 7). For target pH of 6.0, the incubation and modified Mehlich LR are similar. The standard error of estimates for the modified Mehlich (Sy.x=0.57) was relatively lower than the SMP and Adams-Evans (Figure 6). Accordingly, the regression equation for the modified Mehlich better explains the variability in LR than the two methods. Hoskins and Erich (2008) also found that the modified Mehlich predicted the LR better than the SMP for the target pH of 6.0.6.5 and 7.0. Wolf et al. (2008) reported that the modified Mehlich was better than SMP in predicting the LR for target pH of 6.5 and 7.0.

### Titration curve of the pH vs. $Ca(OH)_2$ added

The plot of soil pH as a function of $Ca(OH)_2$ added was linear (Figure 7). It shows an increase in pH of soil with the addition of $Ca(OH)_2$. Similar result was reported by Barouchas et al. (2013).

### LR calculated using Single Addition of $Ca(OH)_2$ vs. Incubation LR

As compared to the incubation, the calcium hydroxide titration greatly underestimated the LR for both the low and high LR values (Figure 8). The slope of the regression equation clearly showed that it measured about 11.6% of the incubation LR on average for the target pH values. The finding is similar to what Godsey et al. (2007) found. The authors reported that titration

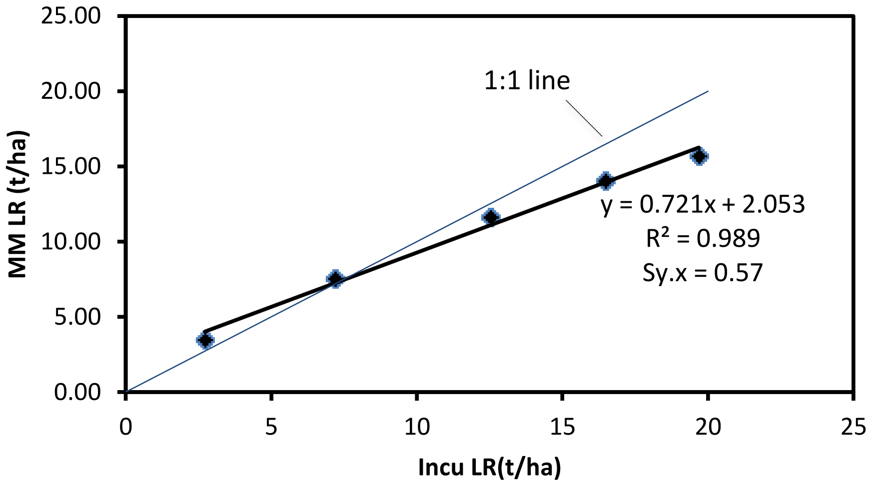

**Figure 6.** Modified Mehlich versus incubation LR in tons/ha.

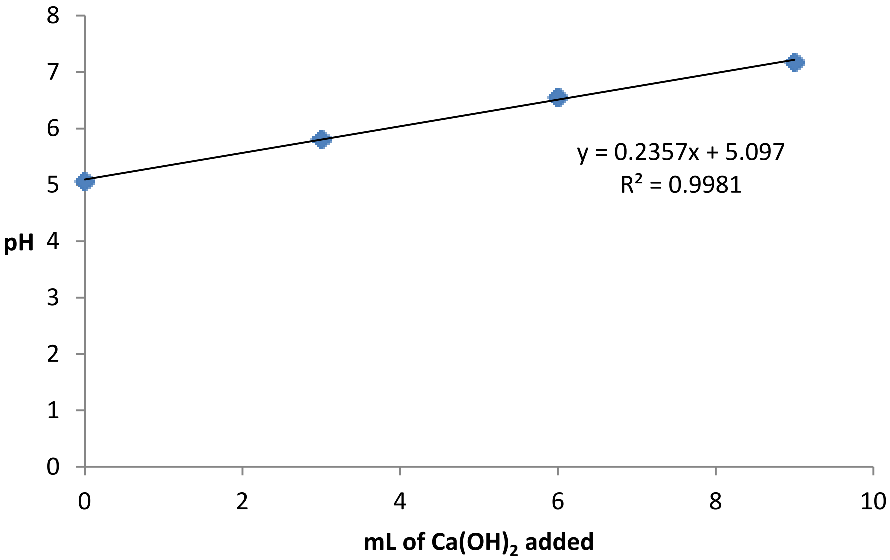

**Figure 7.** Titration curve displaying the amount of added Ca $(OH)_2$ vs. pH.

underestimated the 60-day incubation LR and measured only 45% of it. In contrast, Liu et al. (2004) found that titration with $Ca(OH)_2$ measured 80% of the LR from a 3-day incubation. The disagreement in the findings could be the high clay and CEC content of the soil used in this study which would give the high buffering capacity to the soil compared to the soil used in a 3-day incubation period.

## The exchangeable aluminum method for LR estimation of LR

Based on the exchangeable Al, the LR was calculated using Equation 2; it is about 3.066 kg/ha or 3.07 ton/ha, and is the equivalent lime amount required to neutralize the exchangeable Al. According to Kamprath (1970), liming equivalent amount of exchangeable Al resulted in the neutralization of most of the exchangeable Al of the acid soils and raised their pH near 5.5. If we compare the exchangeable Al equivalent LR (3.07 ton/ha) with the incubation LR (2.72 ton/ha) assuming the 3.07 ton/ha would bring the soil pH to 5.5 from the initial pH of 5.2, it is a little bit higher than the incubation LR, though the difference is small (0.25 tons/ha). For target pH of 6.0, 6.5, 6.8 and 7.0, comparing the method with the incubation or other LR methods is impossible as the LR is only based on neutralizing the exchangeable Al.

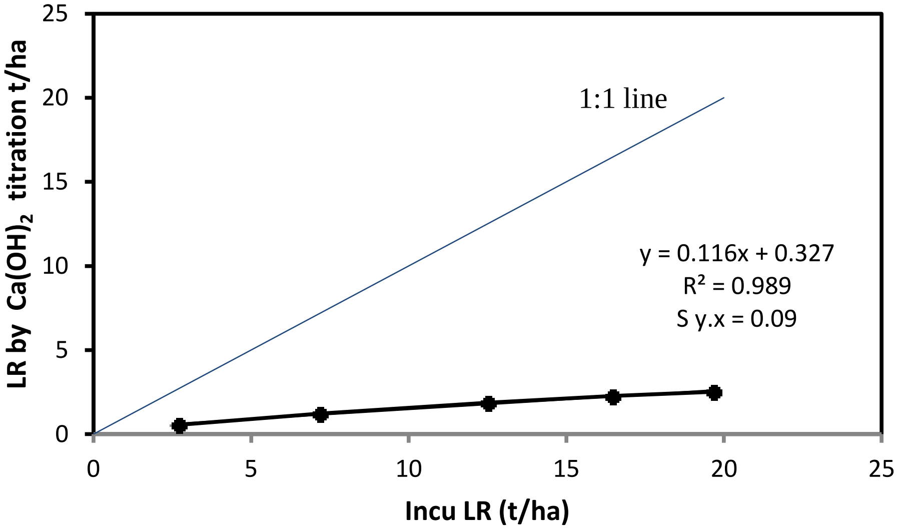

**Figure 8.** Relationship between the LR estimated by Ca $(OH)_2$ and incubation.

## Conclusion

All the methods compared with the incubation LR except the exchangeable acidity method showed a high correlation as the $r^2$ values illustrated. Of the methods compared, the Modified Mehlich buffer was found to be closer in estimating the LR to the reference, incubation LR as Figure 6 depicts. However, further calibration of the method using a set of soils would be necessary to derive a calibrated equation to calculate LR.

## CONFLICT OF INTERESTS

The authors have not declared any conflict of interests.

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