Fractionation of extractable aluminium and biomass production in an acid soil treated with calcareous amendments

M. Vidal-Bardán* and E. Villa-Bermejo
School of Agricultural Engineering, University of León, Avenida de Portugal 41, 24071 León (Spain)

Abstract

A Typic Palexerult in the *raña* formations of the northern plateau in the province of León (Spain) was subjected to acidity correction field tests over a period of three years. The experimental crop was a local rye variety (*Secale cereale* L. var. Petkus) and the amendments included gypsum, dolomite, limestone and sugar foam waste, all at a 6,000 kg ha⁻¹ rate as CaCO₃. General analyses were integrated with specific tests for soluble and easily exchangeable Al forms (Al-CaCl₂) in addition to KCl, BaCl₂ and CuCl₂ extracted Al; adsorbed Al (NH₄AcO) and amorphous Al. Two types of multiple linear regression models (OLS) for production each year were established; some encompassed all studied variables and others the six Al forms only. As shown here, Al-KCl, Al-CaCl₂, base saturation and exchangeable Ca were the individual variables most strongly correlated with production, with $R^2 = 0.865$, within the topmost 12 cm of the soil layer receiving the calcareous amendments. A principal component analysis exposed a substantial share of pH-dependent charge in organic matter on the cation exchange capacity of the soil.

Additional key words: acidity; biomass; cation-exchange capacity; OLS equation; pH-dependent charge; pH increment.

Resumen

Fraccionamiento del aluminio extraíble y producción de biomasa en un suelo corregido con materiales calizos

Se han realizado unos ensayos de campo de tres años de duración encaminados a la corrección de la acidez de un Typic Palexerult de las formaciones de *raña* de la meseta norte de la provincia de León (España). El cultivo ensayado fue una variedad local (Petkus) de centeno (*Secale cereale* L.), empleándose como materiales calizos en la enmienda del suelo yeso, dolomita, caliza y espumas de azucarera, con dosis de 6.000 kg ha⁻¹ de CaCO₃ equivalente. Además de la analítica general, se determinaron las formas de Al solubles y fácilmente intercambiables (Al-CaCl₂), además del Al extraído con KCl, BaCl₂ y con CuCl₂ estimándose también el Al-adsorbido (NH₄OAc) y el Al-amorfo. Se establecieron dos tipos de modelos de regresión lineal múltiple (OLS) de la producción anual de biomasa; el primero incluye todas las variables analizadas, mientras que en el segundo únicamente se consideran las seis formas de Al analizadas. Se comprueba que el Al-KCl, el Al-CaCl₂, el porcentaje de saturación de bases y el Ca de cambio son las variables más correlacionadas con la producción, alcanzándose valores de $R^2 = 0.865$, al considerar únicamente los 12 cm superficiales del horizonte en el que incorporó la enmienda caliza. Finalmente, mediante el análisis de componentes principales, se deduce el importante efecto de la carga dependiente del pH de la materia orgánica en el aumento de la CIC del suelo.

Palabras clave adicionales: acidez; biomasa; capacidad de intercambio catiónico; carga dependiente del pH; pH incremento de pH; regresión.

Introduction

Xerults are Ultisols possessing a xeric moisture regime in the Soil Taxonomy (Soil Survey Staff, 1999) and the equivalents, among others, of Alumic Acrisols in the WRB classification (FAO, 1998). Knowledge with the agricultural management of Ultisols under a Mediterranean climate is more limited than that with...
the suborders Udults and Ustults, which are associated to regions with a subtropical (warmer, wetter) climate and more widely distributed worldwide (Soil Survey Staff, 1999). Palexerults are well represented in the central, western and northern regions of the Iberian Peninsula (Spain and Portugal). Most occur in old rañá surfaces. A rañá is a form of relief from the middle-upper Pliocene possessing a gentle slope and a continental detrital cover, and usually associated to quartzite ranges in nearby mountain range (Espejo, 1987).

Xerults are subject to two major agronomic shortcomings. One is the presence of argillic horizons with heavily altered minerals, a high content in exchangeable Al and, as a result, a low content in essential elements such as Ca, Mg, Cu, Zn and P. The content in phytotoxic Al$^{3+}$ increases and pH decreases (particularly in underlying Bt horizons) with increasing horizon depth in the soil profile (Boul et al., 1997). The other limiting factor for the suborder Xerults in relation to Udults is a result of its xeric moisture regime, which is highly typical of the Mediterranean region. This regime governs water availability during plant growth, which is hindered by a high water stress during the summer.

The previous two limiting factors (i.e. an extreme acidity and prolonged drought in the soil moisture control section) require that soil management be aimed at two specific targets for success, namely: (a) reducing the contents in phytotoxic Al$^{3+}$ and (b) facilitating growth and expansion of the rhizosphere (particularly in the AB and Bt horizons). The essentially clayey nature of the argillic horizon can be useful towards increasing the soil water reserve in the root zone (Ritchey et al., 1995).

As a rule, the low solubility of the calcareous materials used for agronomic liming (limestone and dolomite, mainly) hinders efficient reduction of Al$^{3+}$ toxicity in sub-surface soil horizons (Reeve & Sumner, 1970, 1972). The low mobility of these materials can be overcome by replacing them with gypsum or sugar foam waste, which are more soluble and hence more efficient in reducing Al saturation in the Bt horizon (Shainberg et al., 1989). Specifically, gypsum is known to raise the Ca/Al ratio in soil (Lund, 1970; Noble et al., 1988; Kinraide et al., 1992) and also to facilitate the formation of Al$\text{SO}_4^-$ ion pairs (Pavan et al., 1982; Kinraide & Parker, 1987; Kinraide et al., 1992) in addition to boosting the self-liming effect reported by Reeve & Sumner (1970, 1972).

The primary aims of this field work were to identify the most suitable analytical variables for predicting biomass production in limed soil and establish their temporal changes, particularly as regards the exchange complex.

Material and methods

The experiment was conducted on an extremely acid Typic Palexerult from the Camposagrado rañá (N León, Spain) over a period of three cropping years (2007-08, 2008-09 and 2009-10). The average annual temperature, annual rainfall and evapotranspiration of the study area are 10.2°C, 667 mm and 640 mm, respectively. Based on these figures, the soil temperature and moisture regimes are Mesic and Xeric respectively.

The experimental crop was a local rye variety (Secale cereale L.; Petkus) and the calcareous amendments used included limestone, mineral gypsum (white alabaster variety), dolomite and sugar foam. The last is a waste from the beet sugar industry. All amendments were applied in April 2007 and buried at a depth of 20 cm at an identical rate (6000 kg ha$^{-1}$ as CaCO$_3$) (Vidal et al., 2003) for incorporation into the soil in a rotavator pass. The chemical composition of the four amendments was determined by atomic absorption spectrometry following alkaline fusion and digestion with HF and HClO$_4$ (Jackson, 1976). The experimental design was a randomized block with four replicates per treatment in addition to control plots. Each plot was 3 m × 5 m in size and separated from the next by a 2 m lane. The amount of seed per area unit employed in the rye field trial was 100 kg ha$^{-1}$. Background fertilizer was supplied in the form of an 8/15/15 complex to give 12.0 kg N ha$^{-1}$, 22.5 kg P$_2$O$_5$ ha$^{-1}$ and 22.5 kg K$_2$O ha$^{-1}$. The cover fertilizer consisted of 38 kg N ha$^{-1}$ as 33% calcium ammonium nitrate. This fertilization scheme coincides with the traditional choice of the local farmers. Broad-leaved adventitious plants were controlled with 40% MCPA at a rate of 1-L ha$^{-1}$ during the spring. Plants were harvested 16 cm aboveground with a self-propelled mower, the aerial (collected) portion constituting the total biomass production for each plot and the uncut portion stubble.

Control measurements were made by periodically sampling the Ap and AB horizons with a 10 cm Riverside drill. To this end, the Ap horizon was split into
two: Ap1 (0-12 cm) and Ap2 (12-25 cm). A total of six samplings were done over the studied period; three in March (shortly after establishment of the rye crop) and three after harvesting (October). The soil samples were air-dried and sieved through 2 mm mesh. Soil samples were analysed for pH in water and in 1 M KCl (1:2.5), electrical conductivity (EC) and organic matter (OM) (Walkley & Black, 1934). Soil texture was established according to Kilmer & Alexander (1949). Exchangeable bases were extracted with 1 M NH₄AcO at pH 7 and quantified by atomic absorption spectrometry (Ca and Mg) or flame emission spectrometry (Na and K). Cation-exchange capacity (CEC) was determined by titrating NH₄⁺ in a Kjeldahl autotitrator. Aluminium was extracted with 1 M KCl (Al-KCl) according to Lin & Coleman (1960) and determined by inductively coupled plasma atomic emission spectroscopy (ICP-AES). Soluble and easily exchangeable Al were extracted with 0.01 M CaCl₂ (Al-CaCl₂) according to Hoyt & Nyborg (1972), and so was Al-BaCl₂ with 0.1 M BaCl₂ (Peech et al., 1947). Aluminum bound to organic matter (Al-CuCl₂) and outer sphere complexes (Al-sorbed or Al extracted with 1 M NH₄AcO at pH 4) was also determined. Amorphous Al oxide and hydroxide, and amorphous aluminosilicates (if present) extractable with 0.2 M ammonium oxalate at pH 3 were also quantified. Amorphous Al oxide and hydroxide were calculated from ammonium oxalate extractable Al minus (Al-KCl + Al-CuCl₂ + Al-sorbed), following the sequential scheme of Soon (1993). Aluminium in the previous fractions was determined by ICP-AES. The experimental data were used to calculate the percent base saturation (BS), effective cation-exchange capacity (ECEC) and percent Al saturation (Al-SAT).

Results

The results of the chemical analyses of the liming materials are shown in Table 1. Dolomite was the amendment containing the greatest amount of Mg, and limestone and sugar foam those containing the most Ca. Also, Al was present in increased amounts in limestone and dolomite by virtue of their containing silicates. Unlike the other amendments, sugar foam waste contained 79 g kg⁻¹ OM with a C/N ratio of 11.0.

Tables 2 and 3 show the results of the physical and chemical characterization of the topmost three soil horizons. Texturally, the soil ranged from sandy loam in the Ap (umbric) horizon to sandy clayey loam in the AB (transition) horizon to clayey in the Bt (argillic) horizon.

Figure 1 shows the average total rye biomass production for each cropping year as obtained in the four replicates for the soil amended with limestone. The figure also shows the mean production per year for

| Table 1. Chemical composition of the calcareous amendments |
|---------------------------------------------------------|
| CaO (g kg⁻¹) | MgO (g kg⁻¹) | Na₂O (g kg⁻¹) | K₂O (g kg⁻¹) | Al (mg kg⁻¹) | Fe (mg kg⁻¹) | Mn (mg kg⁻¹) | Zn (mg kg⁻¹) | Cu (mg kg⁻¹) | OM (g kg⁻¹) |
|-------------|--------------|--------------|-------------|-------------|-------------|-------------|-------------|-------------|------------|
| Sugar foam  | 403.8        | 14.7         | 0.4         | 1.0         | 2,469       | 1,420       | 121         | 32          | 12.0       | 79         |
| Limestone   | 437.0        | 20.8         | 0.4         | 3.5         | 7,869       | 5,787       | 299         | 24          | 11.0       | 0          |
| Dolomite    | 311.0        | 184.0        | 1.2         | 3.5         | 9,529       | 10,483      | 361         | 26          | 12.0       | 0          |
| Gypsum      | 331.6        | 17.5         | 0.7         | 1.5         | 3,351       | 1,826       | 41          | 16          | 4.6        | 0          |
each treatment in addition to the standard error of mean (SE), which was the statistic used to quantify scatter and estimate the variability of the mean between samples. Biomass production was clearly greater in the third year; also, sugar foam waste was the most effective amendment (up to 6,008 kg ha⁻¹), followed by dolomite and limestone. Figure 2 lists the mean values of pH-water and pH-KCl (ΔpH) of the three subhorizons (Ap1, Ap2 and AB) by effect of the treatments.

The results for each cropping year were used to develop two different equations. One included all measured variables (12), whereas the other only considered the six Al forms corresponding to the sequential extractions (viz. Al-KCl, Al-CaCl₂, Al-CuCl₂, Al-BaCl₂, adsorbed Al and amorphous Al). Only the data for the Ap1 subhorizon were used in both the first model and that considering the six Al forms alone.

If the average production (kg ha⁻¹) of the plots subjected to any of the four treatments is denoted by P, then the equations for the models including all

Table 2. General characterization of the studied soil

| Horizon | Depth (cm) | pH-water | pH-KCl | ΔpH | Sand (%) | Silt (%) | Clay (%) | OM (g kg⁻¹) | EC (dS m⁻¹) |
|---------|------------|----------|--------|-----|----------|----------|----------|-------------|-------------|
| Ap      | 0-25       | 5.0      | 3.9    | −1.1| 57.7     | 23.1     | 19.2     | 24.6        | 0.031       |
| AB      | 25-35      | 4.8      | 3.9    | −0.9| 54.3     | 18.4     | 27.3     | 6.5         | 0.035       |
| Bt      | 35-55      | 4.7      | 3.6    | −1.1| 42.5     | 11.2     | 46.3     | 6.8         | 0.052       |

Table 3. Characteristics of the exchange complex. Aluminium extracted by KCl and bases extracted by NH₄AcO. All values are in cmol(+) kg⁻¹

| Horizon | Ca     | Mg     | Na     | K      | Al     | CEC    | ECEC   | BS (%)   | Al-SAT(%) | Al-KCl/CEC |
|---------|--------|--------|--------|--------|--------|--------|--------|----------|-----------|------------|
| Ap      | 1.40   | 0.20   | 0.03   | 0.21   | 1.14   | 5.70   | 2.98   | 32.2     | 38.3      | 0.200      |
| AB      | 1.32   | 0.18   | 0.02   | 0.13   | 1.64   | 5.48   | 3.29   | 30.1     | 49.8      | 0.299      |
| Bt      | 1.87   | 0.30   | 0.03   | 0.12   | 3.10   | 9.45   | 5.42   | 24.7     | 57.1      | 0.328      |
studied variables are (Dw is Darwin-Watson statistic):

\[ P_{2007-08} = 3468.5 - 0.779\text{Al-KCl} \]
\( (R^2 = 0.606, F = 0.000, Dw = 1.423) \)

\[ P_{2008-09} = -2053.7 + 662.2\text{pH} \]
\( (R^2 = 0.706, F = 0.000, Dw = 1.641) \)

\[ P_{2009-10} = 2391.0 + 1.5\text{BS} - 0.678 \times \text{Ca} \]
\( (R^2 = 0.865, F = 0.000, Dw = 1.792) \)

And those for the models including the six Al forms only:

\[ P_{2007-08} = 3362.0 - 0.810\text{Al-CaCl}_2 \]
\( (R^2 = 0.656, F = 0.000, Dw = 2.218) \)

\[ P_{2008-09} = 466.4 - 726.4\text{Al-KCl} + 252.5\text{Al-amorphous} \]
\( (R^2 = 0.704, F = 0.000, Dw = 1.978) \)

\[ P_{2009-10} = 5374.5 - 0.858\text{Al-KCl} \]
\( (R^2 = 0.737, F = 0.000, Dw = 1.881) \)

Figures 3a and 3b show the factor saturation diagrams of the principal component analysis (PCA) for the first and third cropping year, respectively, and the four amendments in combination plus the control. PCA was applied to 12 analytical variables for the Ap1 subhorizon alone and included total biomass production in order to facilitate the analysis of temporal changes in the variables and their correlation with total annual biomass. The first two principal components (PCs) accounted for 69.6% of the total variance for the first year and 71.4% of that for the third. The first PC can be assigned to the axis reflecting pH and the factors governing it during the soil acidity correction process, whereas the second reflects the nature of the exchange complex in the Ap1 subhorizon and its changes.

Figure 4 shows the variation of the Al-KCl/CEC ratio as a function of pH-KCl for the two Ap subhorizons and the AB horizon in the control plots, which were only subject to the effect of the crop. The data correspond to the 24 samples from each of the three studied horizons (Ap1, Ap2 and AB) in the control plots, which were obtained in four replications of three yearly samplings; a total of 72 samples are thus included. Differences between horizons are clearly reflected in the substantial decrease in Al-KCl/CEC ratio observed in the Ap subhorizons and AB horizon.

**Discussion**

The differences between the pH values in KCl and water (\( \Delta \text{pH} \)) were negative and amounted to approximately one unit (Table 2), which suggests the presence of net charge in the studied soil. Also, the relationship between pH and the degree of base saturation (BS) was very similar to that of kaolinite, which was the dominant mineralogical species (65%), followed by illite (20%) and vermiculite (5%) —the two minor species (Villa, 2005). Villa (2005) previously found gibbsite and goethite contents to increase with increasing horizon depth, and hematite to exhibit the opposite trend. As can be inferred from the Al-SAT values of Table 3, aluminum was the dominant cation in the exchange complex; thus, Al-SAT accounted for 38.3%,
49.8% and 57.1% in the Ap, AB and Bt horizon, respectively. All three horizons clearly exceeded the accepted phytotoxicity threshold for wet Spanish regions, which, according to Mombiela & Mateo (1984), is an Al-SAT level exceeding 20%. Although variable charge associated to organic matter in a highly acid soil is usually low (Thomas & Hargrove, 1984), we used the Al-KCl/CEC ratio as an estimate of pH-dependent charge in the soil organic fraction. As can be seen from Table 3, the Ap (umbric) horizon had a higher CEC and a lower exchangeable Al concentration than the AB horizon; as a result, the former exhibited a higher Al-KCl/CEC ratio. Likewise, the umbric horizon contained more OM and less clay than the AB and Bt horizons; therefore, variable, pH-dependent charge was a result mainly of organic matter in the Ap horizon. The superiority of this agro industrial waste as an amendment is well documented (Espejo et al., 1991) and has been ascribed to its increased content in active, readily dissolved limestone, and also to its fine texture—and increased surface/weight ratio as a result (Peregrina et al., 2006). The increased total biomass production in the third year was due not only to the effect of the liming amendments—and hence, to the increased soil pH—observed in the plots receiving the liming amendments (Fig. 2), but also to the improved agroclimatic conditions and soil water balance. Thus, precipitation was 548.7 mm the first year (2007-08) and 958.5 mm the third (2009-10), the latter exceeding the mean annual rainfall for the study area (667 mm).

Only the regression equations for the Ap1 subhorizon are discussed since the highest $R^2$ values were invariably obtained in the topmost 12 cm of soil. Including the Ap2 subhorizon and/or the AB horizon in the calculations led to poorer multiple linear regression fitting, as well as to lower proportions of explained variance. These results suggest that the liming amendment and its final incorporation into the soil after three years were only effective within the topmost 12 cm.

The explained variance, $R^2$, increased gradually each year with both the 12-variable model and the 6-variable model, and peaked in the third year, once the liming material started to act and the acid correction effect on the topmost 12 cm of the Ap1 subhorizon increased. All $F$ values were significant at the $p = 0.05$ level, which confirms the linear relationship between biomass production and the variables included in each regression model. Also, the residuals were independent, as reflected in Dw, which ranged from 1.4 to 2.2 (Dw is close to 2 for random, independent errors). Similarly apparent was the strongly adverse effect of the content in exchangeable Al (Al-KCl) during the first year, which was the sole variable present in the $P_{2007-08}$ linear regression equation corresponding the first group of OLS equations. The results for the second group confirmed that the most phytotoxic Al form was Al-CaCl$_2$ (i.e. soluble, readily exchanged aluminium) the first year ($P_{2007-08}$), but residual Al-KCl the second ($P_{2008-09}$) and third ($P_{2009-10}$). The beneficial effect of the transformation of displaced Al (Al-KCl) to Al-amorphous forms also reflected in the second group of OLS equations for the second year ($P_{2008-09}$), where Al-amorphous exhibited a positive regression coefficient. Finally, the negative sign of the regression coefficient for exchangeable calcium in the first group of equations for the third year ($P_{2009-10}$) can be ascribed to incomplete incorporation of the amendment into the soil and its partial dissolution during the time span of the experiment. Therefore, total biomass production was closely related to changes in Ap1 in the umbric horizon and scarcely influenced by the other horizons.

The multivariate analysis allowed the changes in Al forms obtained from the extraction sequence, and those in the soil exchange complex, to be interpreted. A comparison of Figs 3a and 3b, which show the final results for ACP in the first and second cropping year,
respectively, reveals that biomass production, pH\text{water} and BS saturated factor 1 very strongly and in the same direction; also, they were associated to a high content in exchangeable Al (Al-KCl and Al-CaCl$_2$). Likewise, amorphous Al and organically bound Al (Al-CuCl$_2$) exhibited substantial changes in factor 1. This can be ascribed to the combination of a reduced adverse, phytotoxic effect of Al-CuCl$_2$ and a simultaneous increase in amorphous Al during the third cropping year. Also, a comparison of the two figures reveals that Al-KCl/CEC, pH\text{water}, and presence of net charge ($\Delta$H) saturated factor 2 differently. Thus, only during the third year did $\Delta$H and pH\text{water} saturate factor 1 in the same direction as CEC and production. On the other hand, CEC and OM saturated factor 1 in the opposite direction to production, pH and $\Delta$H the first year. Similarly, saturation of the cation exchange capacity (BS) on the first PC increased during the third year as a result of the increase in pH-dependent charge (Al-KCl/CEC) derived mainly from the organic component.

Finally, the presence of pH-dependent charge in the Ap1 and Ap2 horizons was ascribed to organic matter in this umbric horizon, which was sandy loam in texture and consisted mainly of kaolinite and illite. Based on the CEC, clay and OM values for this epipedon, the fraction of CEC due to OM was 92 cmol(+) kg$^{-1}$ [versus only 18 cmol(+) kg$^{-1}$ for clay]. This was largely the result of the fulvic component (Vidal et al., 2006), as revealed by the mean results for the control plots during the three years. The results of Fig. 4 clearly expose a decreased Al-KCl/CEC ratio in the Ap1 and Ap2 subhorizons in relation to AB.

As final conclusions, ordinary least-squares (OLS) multiple linear regression models were used to predict total biomass production of rye on an extremely acid raña soil in the province of León (Spain). The soil was supplied with four different types of calcareous amendments: limestone, dolomite, gypsum and sugar foam waste. Based on the results of a three-year experiment, the accuracy of the models, and hence the explained variance, increased with decreasing number of horizons included in the model and was highest during the third year. Thus, the highest explained variance ($R^2 = 0.865$) was that obtained by considering the Ap1 subhorizon (0-12 cm) alone and the third cropping year only. The soil variables most strongly correlated with biomass production were BS, Al-KCl, Al-CaCl$_2$ and exchangeable Ca. The effect of the amendments only reached the topmost (12 cm) of soil during the three years of the field study. Finally, OM in the Ap (umbric) horizon was found to contain pH-dependent charge and the charge to have a substantial effect on the concomitant increase in CEC and decrease in positive charge in the soil.

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