Revista
Brasileira de
Ciência do Solo

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

Soil chemical properties and wheat production in three Andisol with applications of materials from the cellulose industry

Juan Hirzel[(1)]* iD, Sebastián Meier[(2)] iD, Arturo Morales[(2)] iD, Pablo Undurraga[(1)] iD and Francisco Salazar[(3)] iD

[(1)] Instituto de Investigaciones Agropecuarias, INIA Quilamapu, Chillán, Chile.
[(2)] Instituto de Investigaciones Agropecuarias, INIA Carillanca, Temuco, Chile.
[(3)] Instituto de Investigaciones Agropecuarias, INIA Remehue, Osorno, Chile.

ABSTRACT: Soil acidity is one of the main factors that reduces productivity in agricultural crops, and it is corrected with materials that neutralize acidification. Among soils affected by acidity are those of volcanic origin. At least 30 % of agricultural soils in Chile are of volcanic origin, and acidity correction practices must be constantly performed. This study aimed to evaluate the effectively of two materials that may neutralize soil acidification and increase yield production in Chilean volcanic soils. An experiment was conducted during the 2019-2020 season, in which different acidity correction materials derived from the cellulose industry [Ash + Dregs (AD) and Ash + Dregs + Sludge (ADS)] were applied in three volcanic soils cultivated with wheat in southern Chile (Chillán, Temuco, and Osorno). Treatments were a control, $CaCO_3$ at an adjusted rate to reach pH 6.0 and AD and ADS at the same rate as $CaCO_3$ and twice the rate. Results indicated that the application of AD and ADS produced similar effects on $pH(H_2O)$, Ca^{2+}, Mg^{2+}, and Al^{3+} contents, cationic exchange capacity (CEC), and Al saturation compared with $CaCO_3$ at the same rate; the effect was directly proportional to the rate used with each AD and ADS. Wheat production at each location was similar for AD, ADS, and $CaCO_3$ at the same rate; however, it fluctuated between 10.1 and 10.4, 5.4 and 6.5, and 10.0 and 10.3 Mg ha^{-1} at Chillán, Temuco, and Osorno, respectively. Results indicate that these materials are an alternative to be used as a soil acidity correction amendment.

Keywords: ash, dregs, lime, soil acidity, wheat grain yield.

INTRODUCTION

Intensive agriculture can degrade soil fertility, which affects physical, chemical, and biological properties and reduce the potential productivity of soils (Baldi et al., 2006; Stankowski et al., 2018; Hollang et al., 2019; Kalkhoran et al., 2019). The affected chemical properties include the loss of base content or increased acidity and other essential nutrients such as P, N, S, and micronutrients (Castro and Crusciol, 2013; Hirzel et al., 2019). Soil acidity is associated with the content of Al and H and availability of exchangeable bases such as Ca, Mg, K, and Na, which counteract this acidity (Castro and Crusciol, 2013; Fageria and Nascente, 2014; Moreira et al., 2015; Vargas et al., 2019). Therefore, acidity could be one of the main fertility problems of soils (Castro and Crusciol, 2013; Fageria and Nascente, 2014; Hollang et al., 2019).

The worldwide area affected by soil acidity is estimated at 30 % (4000 million ha) of the total cultivated area (Sumner and Noble, 2003; Kalkhoran et al., 2019). This acidification is caused by many factors, which include the leaching of bases (associated with rainfall and irrigation), acid deposition from the atmosphere (combustion of petroleum by-products pollutes rain with sulfuric and nitric acid), and use of ammonia fertilizers (production of H protons through the nitrification process) (Caires et al., 2015). Other factors are related to the generation of protons in the soil (derived from the C, N, and S cycle), physiological acidity (plant-induced process associated with base consumption and release of H^+ protons as soil load replacement), N fixation by leguminous plants (export of H protons in the rhizosphere during the symbiotic fixation process), organic matter (OM) mineralization (release of organic acids from the mineralization process), sulfate reduction and oxidation (sulfates present in marine waters are reduced to sulfides, H_2S, and volatile compound), and pyrite oxidation produces sulfuric acid as the final product (Fageria and Nascente, 2014).

The principal strategy to manage and control acidity is liming, which involves the application of calcium carbonate ($CaCO_3$) and dolomitic limestone ($MgCO_3 \cdot CaCO_3$) (Moore and Ouimet, 2014; Moreira et al., 2015; Vargas et al., 2019). This practice also directly benefits the soil, as it increases the available bases and nutrients such as N, P, S, and Mo, improves the symbiotic fixation of N_2, and reduces ion availability, which in high concentrations is toxic for plants (e.g., Al, Fe, and Mn) (Zambrosi et al., 2007; Fageria and Nascente, 2014; Moreira et al., 2015; Kalkhoran et al., 2019). However, the most important effects of liming for surface applications are observed in the top 0.20 m of soil (Li et al., 2019; Vargas et al., 2019), although incorporating liming materials such as $CaCO_3$ and $CaCO_3 \cdot MgCO_3$ with tasks related to stubble has shown greater in-depth movement, achieving an effect in the chemical properties up to 0.40 m (Vargas et al., 2019).

Biomass ash from the cellulose industry is also among the materials used to control soil acidity, which is characterized by a basic reaction and provides nutrients such as K, P, Mg, Ca, and micronutrients with a low content of heavy metals (Stankowski et al., 2018). To respect, Stankowski et al. (2018) conducted an experiment with spring wheat and pointed out that the application of ash from the wood industry at rates of 2, 4, and 6 Mg ha^{-1} produced a directly proportional increase in K, Ca, and Mn in the soil compared with the control that did not use liming materials, but it did not affect acidity control. In some countries, those materials are characterized by their high Na content and electrical conductivity, which may affect the plants production. However, studies made in Chile indicated that the use of by-products from the cellulose industry didn't affect the germination and growth of radish (*Raphanus sativus* L.) in Alfisol soil (Undurraga et al., 2018). In Chile, the mains cellulose industry wastes are located in the south-central and south, which is coincident with the main area planted with forest. Moreover, in those are of Chile occur the main problem of soil acidity.

Using cellulose industry wastes by correcting the soil acidity could increase crop production and decrease its production cost. Our hypothesis is that the use of cellulose industry

wastes allows correct the soil acidity and increase the wheat yield. The objective of the present study was to evaluate the effect of cellulose industry wastes on soil acidity correction and wheat grain yield in three Andisols in southern Chile.

MATERIALS AND METHODS

The experiment was conducted during the 2019-2020 season at three locations: 1) Chillán, Santa Rosa Experimental Station, INIA Quilamapu (36° 31' S; 71° 54' W), Chile, with Melanoxerand soil (Andisol), whose chemical properties are: $pH(H_2O)$ 5.6 and exchangeable Ca, Mg, K, Na, and Al values of 4.55, 0.42, 0.50, 0.12, and 0.08 $cmol_c$ kg^{-1}, respectively. The climate is temperate Mediterranean characterized by a hot dry summer and cold wet winter. Precipitation was concentrated in winter and spring with 460 mm, mean temperature of 13.4 °C, and 980 mm evaporation; 2) Temuco, INIA Carillanca (38° 41' S, 72° 25' W), Chile, with Hapludands soil (Andisol), $pH(H_2O)$ 5.2 and exchangeable Ca, Mg, K, Na, and Al values of 4.70, 0.83, 0.81, 0.03, and 0.08 $cmol_c$ kg^{-1}, respectively. The climate at Temuco is typically temperate with monthly maximum and minimum mean temperatures of 11.1 and 2.5 °C, respectively, in the cooler months. In the warmer months, the maximum and minimum mean temperatures were 24.5 and 8.4 °C, respectively. Precipitation was concentrated in autumn and spring with 925 and 716 mm evaporation, respectively; and 3) Osorno, INIA Remehue (40° 31' S, 73° 03' W), Chile, with an Andisol soil from the Osorno soil series (Typic Hapludands) and the chemical properties $pH(H_2O)$ 5.4 and exchangeable Ca, Mg, K, Na, and Al values of 2.28, 0.81, 1.39, 0.15, and 0.56 $cmol_c$ kg^{-1}, respectively. The climate is temperate Mediterranean characterized by a hot dry summer and cold wet winter. The annual rainfall at the experimental site in 2019 was 910.4 mm, and the mean daily minimum and maximum temperatures were 4.3 and 13.1 °C in winter and 7.3 and 20.4 °C in summer, respectively, and 692.3 mm evaporation (Soil Survey Staff, 2014; Agrometeorología, 2020).

The wheat (*Triticum aestivum* L.) 'Rocky-INIA' was cultivated at the three locations. There were three acidity correction materials, boiler ash + dregs (AD), boiler ash + dregs + limed sludge (ADS), and commercial calcium carbonate ($CaCO_3$) as a reference (Soprocal). The AD and ADS materials were obtained from the CMPC plant located in Santa Fe, Biobío Region, Chile. The characterization of the acidity correction materials is shown in table 1. The $CaCO_3$ reference rate was determined under laboratory conditions (determination of soil buffering power) to obtain water pH 6.0 (Sadzawka et al., 2006), which was defined as rate 1. In addition, the acidity correction materials AD and ADS were used at the same rate as the $CaCO_3$ reference and at twice rate 1 (rate 2), although the neutralizing power of these two materials was lower than $CaCO_3$ (Table 1). The evaluated treatments at each location were the following: (1) control without any application of soil acidity correction materials, (2) AD rate 1 to reach water pH 6.0 in each soil, (3) AD rate 2, equivalent to twice rate 1 used in each soil, (4) ADS rate 1 to reach pH 6.0 in each soil, (5) ADS rate 2, equivalent to twice rate 1 used in each soil, and (6) commercial $CaCO_3$ at the rate necessary to reach water pH 6.0 in each soil (rate 1). The rates of the acidity correction materials for each soil and treatment are shown in table 2.

Each treatment had three replicates; therefore, there were 18 experimental units at each experimental site. All the treatments were applied between 5 and 30 May 2019, 30 days before sowing the wheat crop, and starting in the areas with earlier rainfall. Each experimental unit's dimension was 25 m^2 (5 × 5 m), without space between them and with 2 m of external borders, and the experimental area cropped at each study site was 450 m^2.

The crop was sown on 5, 15, and 30 June 2019 at Osorno, Temuco, and Chillán, respectively. The seed rate was 200 kg ha^{-1}; seeds were disinfected with thiophanate-methyl (16.66 %) + pyraclostrobin (0.83 %) + triticonazole (8.33 %) (Real Top) at rates of

Table 1. Chemical characterization of soil acidity neutralizing materials

Technical information	Boiler ash + dregs (AD)	Boiler ash + dregs + lime sludge (ADS)	CaCO₃ (Soprocal)
pH(H₂O)	12.01	12.10	12.40
Moisture (%)	5.53	5.83	<0.50
Neutralizing power (%)	60.0	61.0	90.0
CaO (%)	30.22	31.95	51.4
MgO (%)	3.43	3.11	0.95
Na (%)	3.8	3.19	Nd
K₂O (%)	1.35	1.32	Nd
P₂O₅ (%)	0.83	0.92	Nd
S (%)	0.72	0.71	Nd
Fe (%)	1.79	1.76	Nd
Mn (%)	0.53	0.48	Nd
B (mg kg⁻¹)	41.0	40.3	Nd
Cu (mg kg⁻¹)	76.2	68.1	Nd
Zn (mg kg⁻¹)	239.4	223.6	Nd

Nd: not determined.

Table 2. Rate of acidity neutralizing materials used in each soil and treatment

Acidity neutralizing material treatments	Rate of acidity neutralizing material		
	Santa Rosa	Carillanca	Remehue
	———— Mg ha⁻¹ ————		
AD Rate 1	3.11	8.51	6.82
AD Rate 2	6.22	17.02	13.64
ADS Rate 1	3.11	8.51	6.82
ADS Rate 2	6.22	17.02	13.64
CaCO₃	3.11	8.51	6.82

AD: boiler ash + dregs; ADS: boiler ash + dregs + lime sludge; Rate 1: CaCO₃ reference rate; Rate 2: 2 × rate 1.

200 cm^3 100 kg^{-1} seed. Fertilization at sowing was 523 kg ha^{-1} of the mixture consisting of N:P$_2$O$_5$:K$_2$O:MgO:S:Zn:B 7.3:29.4:11.4:3.4:4.2:0.76:0.38 using monoammonium phosphate, Sul-Po-Mag, potassium chloride, zinc sulfate, and boronatrocalcite fertilizers. Afterward, 100 kg ha^{-1} of N was applied at the beginning of the tillering stage using urea as a nitrification inhibitor (0.8 % 3.5 DMPP per 100 kg N, Gurretain) and 80 kg ha^{-1} of N at the beginning of the stem elongation stage in which urea was the nitrogen source. Foliar application with Zn (Metalosate Zinc, 8.5 % ZnO, Albion Balchem Minerals, USA) was also carried out at rates of 1.5 L ha^{-1} at the full tillering stage. Weed control consisted of applying pre-emergence herbicide using flufenacet (12 %) + flurtamone (12 %) + diflufenican (12 %) (Bacara Forte 360 Sc) at a rate of 1 L ha^{-1} dissolved in 200 L water. This was followed by the application of iodosulfuron-methyl-sodium (3.2 %) + metsulfuron-methyl (1.0 %) (Ovassion Extra) using 600 g ha^{-1} diluted in 200 L water to control grasses. Grain yield was determined at the end of the crop (based on 14.5 % grain moisture). In addition, soil sampling for fertility analysis was carried out 360 days after applying the treatments. The wheat crops in Osorno and Temuco locations were not irrigated because the precipitation in both areas was enough to maintain an appropriate soil water level. In Chillán location, it was necessary to perform two irrigation in October and November (between booting to milking stage of the wheat crop).

Wheat yield

The plots were harvested manually at grain maturity on 10, 20, and 25 January 2020 at Chillán, Temuco, and Osorno, respectively. Plant samples were collected from a 2.1 m^2 plot area. Grain samples were oven-dried at 70 °C for 72 h.

Soil analysis and acidity correction materials

Composite samples were collected manually from the 0.00-0.20 m soil layer from each treatment on the same day the wheat was harvested. All samples were air-dried and sieved (2 mm mesh). Soil pH was determined in 1:2.5 soil:water extracts. According to the molybdate-ascorbic acid method, extractable soil P was performed using $NaHCO_3$ 0.5 mol L^{-1} (Olsen P). Exchangeable Ca, Mg, K, and Na were determined by NH_4OAc 1 mol L^{-1} extraction followed by flame spectroscopy: absorption (Ca and Mg) and emission (K and Na). Soil exchangeable Al was extracted by KCl 1 mol L^{-1}. Sulfur (SO_4^{2-}) was determined with calcium phosphate 0.01 mol L^{-1} and turbidimetry. Effective cationic exchange capacity (CEC) was determined as the sum of Ca, Mg, K, Na, and Al. The Al saturation (%) was determined as the ratio between exchangeable Al and CECe multiplied by 100. Micronutrients, Fe, Mn, Zn, and Cu were determined in a diethylenetriaminepentaacetic acid (DTPA) extract by atomic absorption spectroscopy. Boron was extracted with $CaCl_2$ 0.01 mol L^{-1} and determined by colorimetry with azometine. The total nutrient content analyses were performed according to Erich and Ohno (1992) and ASTM C25-19 (2019).

Experimental design and statistical analysis

An experimental block design was used at each location with three replicates. Each location was analyzed separately, and results were analyzed by ANOVA and Tukey's test (p = 0.05) using the SAS PROC MIXED Model procedure (SAS Institute, Cary, North Carolina, USA).

RESULTS

Wheat grain yield

Grain yield at the Chillán location was affected by the treatments to correct soil acidity (p<0.05) (Figure 1) and was within the normal range for this crop in the study area. There were no differences in wheat grain production among treatments with soil acidity correction materials at different rates (p>0.05). The use of AD, ADS, and $CaCO_3$ at the same application rate (rate 1) increased grain yield by 21.3, 25.6, and 22.7 % compared with the control, respectively (Figure 1).

The treatments also influenced grain yield at Temuco location to correct soil acidity (p<0.05) (Figure 2); however, yield values were lower than expected in this study area due to a frost event that occurred in November 2019 and affected the production of 10 % of the wheat seeds in this geographical area. The AD treatments at rate 2 and ADS treatments at both rates surpassed the control treatment yield (p<0.05). Meanwhile, the use of ADS at the same rate as $CaCO_3$ achieved a higher wheat grain yield than this Ca amendment used as a reference in the present study (p<0.05). The use of AD, ADS, and $CaCO_3$ at the same application rate increased grain yield by 12.9, 28.5, and 7.9 % compared with the control, respectively (Figure 2).

As in the other two locations, grain yield at the Osorno location was affected by the treatments to correct soil acidity (p<0.05) (Figure 3) and was within the normal range for this crop in the study area. There were no differences in wheat grain production among treatments applying soil acidity correction materials at different rates (p>0.05). The use of AD, ADS, and $CaCO_3$ at the same application rate increased grain yield by 31.6, 35.6,
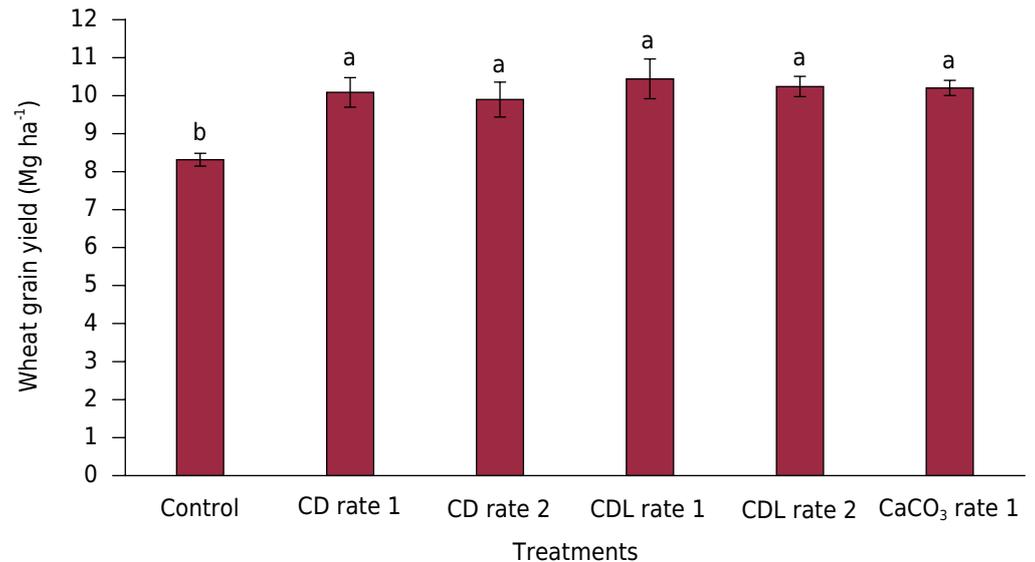
Figure 1. Wheat grain yield at Chillán in response to six treatments of soil acidity control. Different letters over the bars indicate differences between treatments according to Tukey's test (p<0.05). Lines over the bars indicate standard error. AD: boiler ash + dregs; ADS: boiler ash + dregs + lime sludge; Rate 1: CaCO$_3$ reference rate; Rate 2: 2 × rate 1.
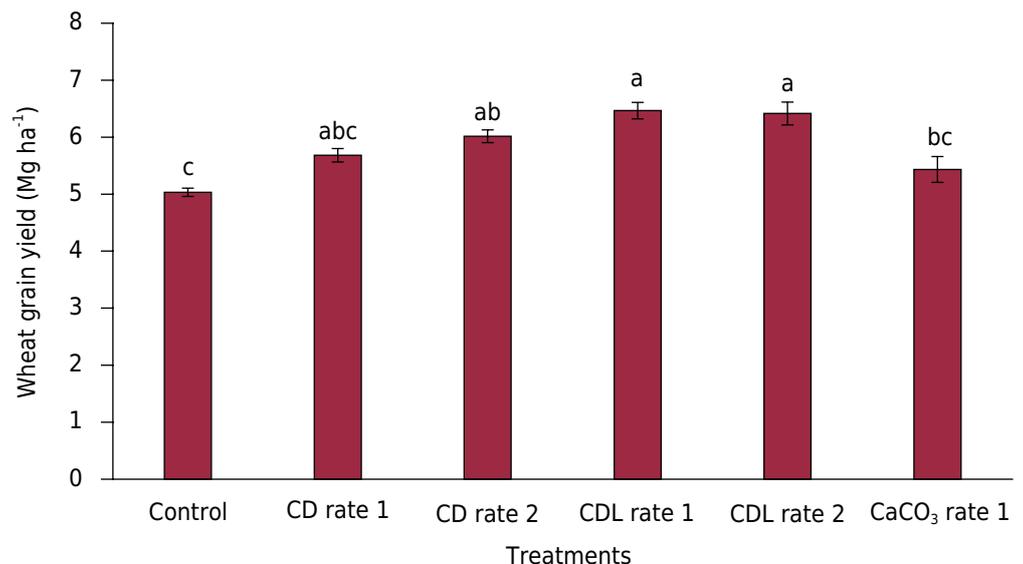
Figure 2. Wheat grain yield at Temuco in response to six treatments of soil acidity control. Different letters over the bars indicate differences between treatments according to Tukey's test (p<0.05). Lines over the bars indicate standard error. AD: boiler ash + dregs; ADS: boiler ash + dregs + lime sludge; Rate 1: CaCO$_3$ reference rate; Rate 2: 2 × rate 1.

and 31.7 %, respectively, compared with the control (Figure 3). The wheat crop yield at the three study locations indicated that the soil acidity correction materials AD and ADS are alternative materials to be used as soil acidity correction amendments.

Soil properties at the Chillán location

The statistical analysis indicated differences for pH, Ca, Al, and Cu contents, CEC and Al saturation values (p<0.05) (Table 3). The highest pH occurred with AD and ADS at rate 2 and using CaCO$_3$ (p<0.05); however, this value was similar to the value obtained with AD and ADS at the same rate as CaCO$_3$ (p>0.05) (Table 3). In quantitative terms, AD and ADS at the same rate as CaCO$_3$ did not achieve the same effect on pH as that obtained with this reference amendment in the present study (p<0.05).
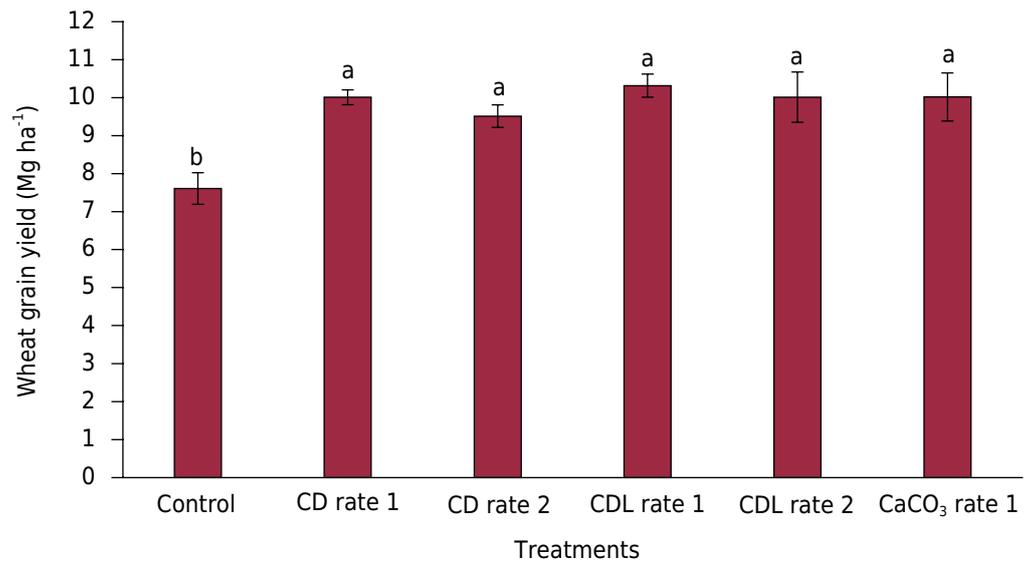
Figure 3. Wheat grain yield at Osorno in response to six treatments of soil acidity control. Different letters over the bars indicate differences between treatments according to Tukey's test (p<0.05). Lines over the bars indicate standard error. AD: Boiler ash + dregs; ADS: Boiler ash + dregs + lime sludge; Rate 1: CaCO$_3$ reference rate; Rate 2:2 × rate 1.

Table 3. Soil chemical properties at the Santa Rosa location 360 days after the application of six soil acidity control treatments

Treatment	Control	AD Rate 1	AD Rate 2	ADS Rate 1	ADS Rate 2	CaCO$_3$ Rate 1	P-value
pH(H$_2$O)	5.8 b	5.9 ab	6.2 a	6.1 ab	6.2 a	6.2 a	0.14
Available P (mg kg^{-1})	18.4 a	17.1 a	17.5 a	18.9 a	17.2 a	16.7 a	0.78
Ca^{2+} (cmol$_c$ kg^{-1})	3.79 b	4.49 ab	5.89 ab	5.09 ab	4.87 ab	6.47 a	0.03
Mg^{2+} (cmol$_c$ kg^{-1})	0.30 a	0.35 a	0.42 a	0.35 a	0.37 a	0.36 a	0.18
K$^+$ (cmol$_c$ kg^{-1})	0.39 a	0.34 a	0.31 a	0.26 a	0.27 a	0.32 a	0.25
Na$^+$ (cmol$_c$ kg^{-1})	0.05 a	0.11 a	0.24 a	0.26 a	0.19 a	0.12 a	0.09
Al^{3+} (cmol$_c$ kg^{-1})	0.10 a	0.06 ab	0.03 b	0.05 ab	0.05 ab	0.03 b	0.02
CECe (cmol$_c$ kg^{-1})	4.6 b	5.4 ab	6.9 ab	6.0 ab	5.7 ab	7.0 a	0.03
Al saturation (%)	2.2 a	1.2 ab	0.4 b	0.9 ab	0.9 ab	0.5 b	0.008
Available Zn (mg kg^{-1})	0.37 a	0.28 a	0.55 a	0.36 a	0.34 a	0.56 a	0.36
Available Fe (mg kg^{-1})	23.4 a	22.6 a	22.6 a	24.0 a	21.9 a	22.6 a	0.35
Available Cu (mg kg^{-1})	0.81 b	0.80 b	0.88 ab	0.86 ab	0.86 ab	0.92 a	0.02
Available Mn (mg kg^{-1})	2.8 a	3.2 a	3.5 a	3.5 a	3.5 a	3.5 a	0.21
Available B (mg kg^{-1})	0.36 a	0.30 a	0.24 a	0.31 a	0.32 a	0.30 a	0.10
Available S (mg kg^{-1})	31.7 a	23.7 a	31.2 a	31.8 a	33.1 a	31.7 a	0.38

AD: boiler ash + dregs; ADS: boiler ash + dregs + lime sludge; Rate 1: CaCO$_3$ reference rate; Rate 2: 2 × Rate 1; CECe: effective cationic exchange capacity. Different letters in the same row indicate differences between treatments according to Tukey's test (p<0.05).

The CaCO$_3$ treatment showed the highest exchangeable Ca content and soil CECe (p<0.05) (Table 3). The highest exchangeable Al concentration and Al saturation value were obtained in the control with no application of soil acidity correction materials (p<0.05) (Table 3). The same exchangeable Al and Al saturation values occurred with AD and ADS at the same rate as CaCO$_3$ (p>0.05) (Table 3).

The highest available Cu contents was obtained in the CaCO$_3$ treatment (p<0.05) (Table 3); however, higher available S, Fe, Mn, Cu, Zn, and B contents were expected in the treatments with AD and ADS, associated with the contribution of these nutrients when using these soil acidity correction materials (Table 1). The alkaline reaction of the

applied amendments probably decreased the availability of metallic microelements (mainly Mn) associated with oxidation-reduction processes.

Soil properties at the Temuco location

The statistical analysis indicated differences for pH, Ca, Mg, Na, Al, Cu, Mn, and S concentrations, and CECe and Al saturation values ($p < 0.05$) (Table 4). The higher pH occurred with ADS at rate 2 ($p < 0.05$); however, this value was similar to $CaCO_3$ ($p > 0.05$) (Table 4). In general, all the treatments with acidity correction materials increased pH compared with the control. The use of AD and ADS at the same rate as $CaCO_3$ did not achieve the same effect on pH as with the reference amendment in the present study ($p < 0.05$).

The highest exchangeable Ca content and soil CECe value were found in the AD and ADS treatments at rate 2, the same as with $CaCO_3$ ($p < 0.05$) (Table 4). Statistically, only AD at rates similar to $CaCO_3$ achieved the same values of exchangeable Ca and CEC obtained with the reference amendment in the present study ($p > 0.05$) (Table 4).

The treatments with AD and ADS at rate 2 showed the highest exchangeable Mg concentration ($p < 0.05$). This was associated with the contribution of Mg of these amendments, 3.43 and 3.11 %, respectively (Table 1). Statistically, AD and ADS at the same rate as $CaCO_3$ had the same exchangeable Mg values obtained with the reference amendment in the present study ($p > 0.05$) (Table 4).

Associated with the Na supply of this amendment, the highest exchangeable Na content was obtained in the treatment with ADS at rate 2 ($p < 0.05$) (Table 4). The highest exchangeable Al concentration and Al saturation value were obtained in the control with no application of soil acidity correction materials ($p < 0.05$) (Table 4). The same exchangeable Al and Al saturation values occurred with AD and ADS at the same rate as $CaCO_3$ ($p > 0.05$) (Table 4).

For Zn, its highest available content was obtained in the treatments with AD at rate 1 and ADS at rate 2 ($p < 0.05$) (Table 4), which was related to the contributions of Zn from

Table 4. Soil chemical properties at the Carillanca location 360 days after the application of six soil acidity control treatments

Treatment	Control	AD Rate 1	AD Rate 2	ADS Rate 1	ADS Rate 2	$CaCO_3$ Rate 1	P-value
$pH(H_2O)$	5.1 d	5.9 c	6.2 b	5.8 c	6.6 a	6.4 ab	0.002
Available P ($mg\ kg^{-1}$)	10.5 a	17.8 a	17.2 a	13.7 a	15.8 a	13.2 a	0.24
Ca^{2+} ($cmol_c\ kg^{-1}$)	2.89 c	9.76 abc	11.38 ab	7.73 bc	17.21 a	16.73 a	0.0012
Mg^{2+} ($cmol_c\ kg^{-1}$)	0.47 c	0.96 ab	1.17 a	0.72 bc	1.32 a	0.69 bc	0.0004
K^+ ($cmol_c\ kg^{-1}$)	0.50 a	0.56 a	0.50 a	0.48 a	0.61 a	0.38 a	0.8
Na^+ ($cmol_c\ kg^{-1}$)	0.04 b	0.17 ab	0.26 ab	0.12 ab	0.42 a	0.07 b	0.03
Al^{3+} ($cmol_c\ kg^{-1}$)	0.08 a	0.02 b	0.02 b	0.02 b	0.01 b	0.01 b	0.0009
CECe ($cmol_c\ kg^{-1}$)	4.0 c	11.5 abc	13.3 ab	9.1 bc	19.6 a	17.9 ab	0.0015
Al saturation (%)	2.1 a	0.2 b	0.2 b	0.2 b	0.1 b	0.1 b	0.0001
Available Zn ($mg\ kg^{-1}$)	0.72 b	1.95 a	1.82 ab	1.18 ab	2.08 a	1.19 ab	0.01
Available Fe ($mg\ kg^{-1}$)	70.0 a	83.7 a	70.8 a	70.9 a	68.0 a	77.7 a	0.08
Available Cu ($mg\ kg^{-1}$)	2.0 b	2.7 a	2.6 a	2.3 ab	2.8 a	2.3 ab	0.004
Available Mn ($mg\ kg^{-1}$)	14.5 a	10.7 ab	10.3 ab	10.3 ab	9.9 ab	7.9 b	0.05
Available B ($mg\ kg^{-1}$)	0.32 a	0.29 a	0.32 a	0.25 a	0.20 a	0.16 a	0.15
Available S ($mg\ kg^{-1}$)	40 b	9.4 ab	8.0 ab	9.8 ab	13.3 a	16.6 a	0.008

AD: Boiler ash + dregs; ADS: boiler ash + dregs + lime sludge; Rate 1: $CaCO_3$ reference rate; Rate 2: 2 × Rate 1; CECe: effective cationic exchange capacity. Different letters in the same row indicate differences between treatments according to Tukey's test ($p < 0.05$).

these amendments, 239 and 224 mg kg^{-1}, respectively (Table 1). However, the same effect of increased available Zn compared with the control in all the AD and ADS treatments was expected. A similar effect was observed in the available Cu content in which higher values were expected in the AD and ADS treatments and associated with the contribution of Cu in these amendments, 76 and 68 mg kg^{-1}, respectively (Table 1). For available Mn, although the AD and ADS amendments provide this element, the greater acidification effect achieved in control with no application of soil acidity correction materials increased the concentration of this nutrient ($p<0.05$) (Table 4).

The treatments ADS at rate 2 and CaCO$_3$ showed the highest available S concentration ($p<0.05$) (Table 4), associated with the contribution of this amendment. However, a significant increase in available S was expected with AD and ADS compared with CaCO$_3$ because of the contribution of S in these two amendments, 0.72 and 0.71 %, respectively (Table 1). As pointed out for the soil at the Chillán location, the alkaline reaction of the applied amendments could have decreased the availability of metallic microelements (mainly Mn) associated with oxidation-reduction processes.

Soil properties at the Osorno location

The statistical analysis indicated differences for pH, Ca, Mg, Na, Al, and S concentrations, and for CECe and Al saturation values ($p<0.05$) (Table 5). The highest pH was obtained with AD and ADS at rate 2, AD at rate 1, and CaCO$_3$ ($p<0.05$). However, only the values of AD and ADS at rate 2 and CaCO$_3$ were higher than the control ($p<0.05$).

The highest exchangeable Ca content and soil CECe value showed the same behavior described for soil pH, with AD and ADS at the same CaCO$_3$ rate they were similar to those with this Ca amendment used as reference in the present study ($p>0.05$) (Table 5). Associated with Mg contribution of these amendments, the highest exchangeable Mg concentration was reached in the treatments with AD and ADS, 3.43 % and 3.11% MgO, respectively (Tables 1 and 5). However, only the treatments with AD at both rates and ADS at rate 2 achieved a higher exchangeable Mg concentration than CaCO$_3$ ($p<0.05$).

The contribution of Na of this amendment was associated with the highest exchangeable Na content obtained in the treatment with ADS at rate 2 ($p<0.05$) (Table 5). The highest exchangeable Al concentration and Al saturation value were obtained in the control treatment ($p<0.05$) (Table 5). The values for these parameters with AD and ADS at the same rate as CaCO$_3$ were similar to those obtained in this Ca amendment used as a reference in the present study ($p>0.05$) (Table 5).

The highest available S concentration was obtained in the treatment with AD at rate 2 ($p<0.05$) (Table 5) associated with the contribution of S of this amendment. However, a significant increase in available S was expected with AD compared with CaCO$_3$ because of the contribution of S in these two amendments, 0.72 and 0.71 %, respectively (Table 1).

DISCUSSION

The grain yield of the wheat crop at the Chillán location was normal for the study area (Hirzel et al., 2019). The response to the application of acidity correction materials for this soil with an initial pH 5.6 was expected in the wheat crop, as reported by Stankowski et al. (2018) and Iljkić et al. (2019). No response between treatments with different soil acidity correction materials and their rates on wheat yield was expected when using rates that increased pH to 6.0 with the reference material used in the present study (CaCO$_3$), given that this crop exhibits its highest yield potential in pH ranging from 5.5 to 7.0 (Havlin et al., 1999). There are as yet no specific response studies of 'Rocky-INIA' to different levels of soil acidity. Lollato et al. (2018) indicated that there are critical soil acidity levels for

Table 5. Soil chemical properties at the Remehue location 360 days after the application of six soil acidity control treatments

Treatment	Control	AD Rate 1	AD Rate 2	ADS Rate 1	ADS Rate 2	CaCO₃ Rate 1	P-value
pH(H$_2$O)	5.5 c	5.7 abc	6.0 a	5.6 bc	5.9 ab	5.7 abc	0.0057
Available P (mg kg^{-1})	20.7 a	19.5 a	18.6 a	19.4 a	20.8 a	20.6 a	0.32
Ca^{2+} (cmol$_c$ kg^{-1})[1]	2.33 c	5.31 ab	6.77 ab	4.50 bc	7.40 a	6.71 ab	0.001
Mg^{2+} (cmol$_c$ kg^{-1})	0.75 bc	1.01 a	1.00 a	0.80 abc	0.92 ab	0.57 c	0.0004
K$^+$ (cmol$_c$ kg^{-1})	0.98 a	0.96 a	0.96 a	1.04 a	0.98 a	0.65 a	0.08
Na$^+$ (cmol$_c$ kg^{-1})	0.07 b	0.24 ab	0.53 a	0.21 b	0.32 ab	0.06 b	0.0038
Al^{3+} (cmol$_c$ kg^{-1})	0.54 a	0.16 b	0.11 b	0.26 b	0.09 b	0.15 b	0.0018
CECe (cmol$_c$ kg^{-1})	4.7 c	7.7 ab	9.4 ab	6.8 bc	9.7 a	8.1 ab	0.0009
Al saturation (%)	11.6 a	2.0 b	1.2 b	3.8 b	1.0 b	1.9 b	0.0003
Available Zn (mg kg^{-1})	1.04 a	1.61 a	1.23 a	1.13 a	1.28 a	0.92 a	0.29
Available Fe (mg kg^{-1})	73.0 a	56.6 a	61.7 a	63.3 a	61.2 a	68.9 a	0.24
Available Cu (mg kg^{-1})	2.4 a	2.4 a	2.5 a	2.5 a	2.5 a	2.5 a	0.84
Available Mn (mg kg^{-1})	16.4 a	14.7 a	15.0 a	14.7 a	14.1 a	15.2 a	0.68
Available B (mg kg^{-1})	0.58 a	0.47 a	0.34 a	0.40 a	0.42 a	0.35 a	0.16
Available S (mg kg^{-1})	14.1 c	36.5 ab	39.4 a	28.4 ab	31.2 ab	25.3 bc	0.0006

AD: Boiler ash + dregs; ADS: boiler ash + dregs + lime sludge; Rate 1: CaCO₃ reference rate; Rate 2: 2 × Rate 1; CECe: effective cationic exchange capacity. Different letters in the same row indicate differences between treatments according to Tukey's test ($p < 0.05$).

wheat cultivars sensitive or tolerant to acidity; for a 3-years study (2013-2015) at two Oklahoma locations, the critical water pH to maximize yield in both types of cultivars was 4.8 and 5.8, respectively.

Similar effects in yield potential and response to soil acidity correction treatments were obtained at the Osorno location, where the initial pH was 5.4. Iljkić et al. (2019) mentioned that the response of increased grain yield in the wheat crop during three consecutive seasons for the application of CaCO₃·MgCO₃ at rates of 3.56 and 7.14 Mg ha^{-1} in a soil with water pH 5.4 had no effect when the highest rate of this acidity correction material was used (14.25 Mg ha^{-1}).

In contrast with grain yields obtained at Chillán and Osorno, grain yield at the Temuco location did not surpass 6.47 Mg ha^{-1}; the cause was a frost event recorded in November (wheat flowering stage) that reduced crop yield by 25 % on average in the study area (INIA Breeding Program, 2020). The Temuco location also had a response with erratic effects to soil acidity control treatments, given that the use of CaCO₃ achieved the same yield as the control. A great response was expected for the application of this amendment, given the initial soil pH (5.2), whose effect could be explained if the genetic material used in this wheat crop was tolerant to acidity (Lollato et al., 2018). Nevertheless, there was a response at the other two locations to the application of CaCO₃ to increase pH to 6.0. The use of ADS at this location produced a grain yield on the average 10 % higher than the AD treatments, but the nutritional characteristics of both types of acidity correction materials are very similar and therefore cannot explain the difference obtained in the field experiment at the Temuco location (Table 1). As well as the AD and ADS use provided essential nutrients to obtain high-yielding wheat, the fertilization use in all the experiments enables to reach the yield potential for the wheat crop in the south and south-central of Chile (higher to 8 Mg ha^{-1}; INIA Breeding Program, 2020).

Regarding the effect of applying acidity correction materials on soil chemical properties, an inversely proportional relationship between soil pH and exchangeable Al concentration was observed, as expected, at the three locations; the same occurred between soil pH and Al saturation (Castro and Crusciol, 2013; Caires et al., 2015; Vargas et al., 2019). Unlike other studies by Vargas et al. (2019) and Fageria and Nascente (2014), the effect

of the acidity correction materials and their increasing rates was partially proportional to the concentration of the basic reaction cations Ca, Mg, and Na and on CECe.

The Na concentration obtained in the soil was largely affected by AD and ADS due to the Na content in both materials, 3.80 and 3.19 %, respectively (Table 1), although the effect was significant only at the Temuco and Osorno locations. The highest concentration of Ca in $CaCO_3$, used as reference in the present study for AD and ADS (Table 1), was confirmed in the soil exchangeable Ca concentration for the treatments at the same rate of acidity correction material at the three locations; this occurred to a greater extent at the Temuco location and with significant differences only in the Temuco and Osorno locations for the use of ADS. A similar effect was expected for soil exchangeable Ca with AD and ADS, given that the Ca concentration in both products is similar; this can be explained by a higher speed of Ca delivery from AD, which was not evaluated in the present study.

Although the application of AD and ADS generated a substantial contribution of Mg compared with $CaCO_3$, the increase in soil exchangeable Mg with ADS at the Temuco and Osorno locations was achieved only with rate 2 of this acidity correction material, while an effect for AD was observed at the Osorno location with rates similar to $CaCO_3$. The absence of this effect on soil Mg with similar rates of AD and ADS compared with $CaCO_3$ can be explained by the soil buffering capacity of each location (Havlin et al., 1999; Fageria and Nascente, 2014), especially when considering that volcanic soils are characterized by a high soil buffering capacity, which increases proportionally with increasing pH due to the variable charge characteristics of these soils (Havlin et al., 1999; Neall, 2006). Vargas et al. (2019) and Castro and Crusciol (2013) showed that the Mg cation moves within the soil profile at a higher speed than the Ca cation, so that part of the Mg contributed by these soil acidity correction materials could move deeper than the top 0.20 m of soil evaluated in the present study.

The P and K contents were not affected by the evaluated treatments in any of the volcanic soils of this experiment; this concurs with findings by Castro and Crusciol (2013) in the top 0.20 m of soil 12 months after the application of dolomitic lime, although these authors used an Oxisol soil. These same authors indicated an increase in the available P content only in the top 0.10 m of soil associated with the release of P from Fe and Al phosphates, which generated precipitation of Fe and Al with the hydroxides released in the alkaline reaction of the application of dolomitic lime. Soils evaluated in our experiment only increased quantitatively in the available P content at the Temuco location, but it was nonsignificant.

The absence of the effect on the K content in the evaluated soils is associated with the soil buffering capacity (Havlin et al., 1999; Neall, 2006; Ebeling et al., 2011). Although the AD and ADS materials provided K, which could be quantitatively important at Temuco and Osorno locations (Table 2), the volcanic soils showed a significant K desorption (Havlin et al., 1999; Neall, 2006) that, in addition to the fertilization used in the crop, could mask significant effects of the contribution of this element with AD and ADS.

Some cationic micronutrients showed a quantitative increase in concentration associated with the contribution of the acidity correction materials (Table 1), as found for Cu at the Chillán and Temuco locations and for Zn at the Temuco location. However, only the Fe and Mn contributions with the use of AD and ADS were quantitatively important (1.79 and 1.76 % of Fe and 0.53 and 0.48 % of Mn in AD and ADS, respectively). Although increased Fe and Mn contents were expected in soils with the application of AD and ADS at different rates, this was not the case. In addition, the soil Mn at Temuco exhibited an inversely proportional relationship with soil pH. These effects are explained by oxidation reactions on cationic microelements in their reduced states (Fe^{2+}, Fe^{3+}, Mn^{2+}, Cu^{2+}, and Zn^{2+}) when increased soil pH occurs (Havlin et al., 1999; Fageria and Nascente, 2014).

For available B, Fageria and Nascente (2014) indicated a dramatic drop when the pH level was higher than 6.0, which was observed with the ADS treatments at rate 2 and $CaCO_3$ only at the Temuco location (Table 4). For available S, there was an increased effect in content at the Temuco and Osorno locations associated with the contribution of this element by the acidity correction materials AD and ADS derived from the cellulose industry (Table 1). Finally, the values obtained for the soil chemical properties at the Chillan location were usually similar to those reported by Undurraga et al. (2009) and Hirzel et al. (2020) for the same study area, although the available Mn and S contents were lower and higher, respectively, for findings indicated by Undurraga et al. (2009).

CONCLUSIONS

For most of the soils in the present study, the application of soil cellulose industry wastes Ash + Dregs (AD) and Ash + Dregs + Sludge (ADS) produced similar effects on the soil acidity correction related to the use of $CaCO_3$ at the same rate.

The application of cellulose industry wastes AD and ADS at increasing rates produced an increasing effect on the soil chemical properties indicating acidity, pH, exchangeable Ca, Mg, and Al contents, CECe, and decreased Al saturation, which was directly proportional to the rate used in most of the soils in the present study.

The wheat grain yield obtained in the three study locations indicate that the celulose industry wastes AD and ADS constitute an alternative as soil acidity correction amendments.

AUTHOR CONTRIBUTIONS

Conceptualization: (iD) Juan Hirzel (lead) and (iD) Pablo Undurraga (supporting).

Methodology: (iD) Juan Hirzel (lead) and (iD) Pablo Undurraga (supporting).

Software: (iD) Juan Hirzel (lead), (iD) Sebastian Meier (supporting), and (iD) Francisco Salazar (supporting).

Validation: (iD) Juan Hirzel (lead), (iD) Sebastian Meier (supporting), (iD) Arturo Morales (supporting), and (iD) Francisco Salazar (supporting).

Formal analysis: (iD) Juan Hirzel (lead), (iD) Sebastian Meier (supporting), (iD) Arturo Morales (supporting), and (iD) Francisco Salazar (supporting).

Investigation: (iD) Juan Hirzel (lead), (iD) Sebastian Meier (supporting), (iD) Arturo Morales (supporting), and (iD) Francisco Salazar (supporting).

Resources: (iD) Juan Hirzel (lead), (iD) Sebastian Meier (supporting), (iD) Arturo Morales (supporting), and (iD) Francisco Salazar (supporting).

Data curation: (iD) Juan Hirzel (lead), (iD) Sebastian Meier (supporting), (iD) Arturo Morales (supporting), and (iD) Francisco Salazar (supporting).

Writing – original draft: (iD) Juan Hirzel (lead) and (iD) Pablo Undurraga (supporting).

Writing – review and editing: (iD) Juan Hirzel (lead) and (iD) Pablo Undurraga (supporting).

Visualization: (iD) Juan Hirzel (lead) and (iD) Pablo Undurraga (supporting).

Supervision: (iD) Juan Hirzel (lead), (iD) Sebastian Meier (supporting), (iD) Arturo Morales (supporting), and (iD) Francisco Salazar (supporting).

Project administration: (iD) Juan Hirzel (lead).

REFERENCES

Agrometeorología. Red agrometeorológica de Chile. Chile: INIA; 2020 [cited 2020 Sep 28]. Available from: https://agrometeorologia.cl/.

ASTM C25-19. Standard test methods for chemical analysis of limestone, quicklime, and hydrated lime. West Conshohocken, PA: ASTM International; 2019. Available from: www.astm.org

Baldi E, Toselli M, Marcolini G, Marangoni B. Effect of mineral and organic fertilization on soil chemical, biological and physical fertility in a commercial Peach orchard. Acta Hortic. 2006;721:55-62. https://doi.org/10.17660/ActaHortic.2006.721.6

Caires EF, Haliski A, Bini AR, Scharr DA. Surface liming and nitrogen fertilization for crop grain production under no-till management in Brazil. Eur J Agron. 2015;66:41-53. https://doi.org/10.1016/j.eja.2015.02.008

Castro GSA, Crusciol CAC. Effects of superficial liming and silicate application on soil fertility and crop yield under rotation. Geoderma. 2013;195-196:234-42. https://doi.org/10.1016/j.geoderma.2012.12.006

Ebeling AG, Anjos LH, Pérez DV, Pereira MG, Valladares GS. Relationship between acidity and other chemical attributes of soils with high organic matter content. Bragantia. 2011;70:157-65. https://doi.org/10.1590/S0006-87052008000200019

Erich MS, Ohno T. Titrimetric determination of calcium carbonate equivalence of wood ash. Analyst. 1992;117:993-5. https://doi.org/10.1039/an9921700993

Fageria N, Nascente A. Management of soil acidity of South American soils for sustainable crop production. Adv Agron. 2014;128:221-75. https://doi.org/10.1016/B978-0-12-802139-2.00006-8

Havlin JL, Tisdale SL, Nelson W, Havlin J, Beaton J. Soil fertility and fertilizers: An introduction to nutrient management. 6th ed. Upper Saddle River, New Jersey: Prentice-Hall; 1999.

Hirzel J, Undurraga P, León L, Panichini M, González J, Carrasco J, Matus I. Maize grain production, plant nutrient concentration and soil chemical properties in response to different residue levels from two previous crops. Acta Agr Scand B-S P. 2020;70:285-93. https://doi.org/10.1080/09064710.2020.1725619

Hirzel J, Undurraga P, León L, Panichini M, González J, Carrasco J, Matus I. Different residues affect wheat nutritional composition. J Soil Sci Plant Nutr. 2019;20:75-82. https://doi.org/10.1007/s42729-019-00102-2

Iljkić D, Kovačević V, Rastija M, Antunović M, Horvat D, Josipović M, Varga I. Long term effect of Fertdolomite on soil, maize and wheat status on acid soil of eastern Croatia. J Central Eur Agric. 2019;20:461-74. https://doi.org/10.5513/JCEA01/20.1.2140

Kalkhoran SS, Pannell DJ, Thamo T, White B, Polyakov M. Soil acidity, lime application, nitrogen fertility, and greenhouse gas emissions: Optimizing their joint economic management. Agr Syst. 2019;176:102684. https://doi.org/10.1016/j.agsy.2019.102684

Li GD, Conyers MK, Helyar KR, Lisle CJ, Poile GJ, Cullis BR. Long-term surface application of lime ameliorates subsurface soil acidity in the mixed farming zone of south-eastern Australia. Geoderma. 2019;338:236-46. https://doi.org/10.1016/j.geoderma.2018.12.003

Lollato RP, Ochsner TE, Arnall DB, Griffin TW, Edwards JT. From field experiments to regional forecasts: Upscaling wheat grain and forage yield response to acidic soils. Agron J. 2018;111:287-302. https://doi.org/10.2134/agronj2018.03.0206

Moore J, Ouimet R. Effects of two types of Ca fertilizer on sugar maple nutrition, vigor and growth after 7 years. For Ecol Manag. 2014;320:1-5. https://doi.org/10.1016/j.foreco.2014.02.017

Moreira A, Sfredo GJ, Moraes LAC, Fageria NK. Lime and cattle manure in soil fertility and soybean grain yield cultivated in tropical soil. Commun Soil Sci Plant Anal. 2015;46:1157-69. https://doi.org/10.1080/00103624.2015.1033542

Neall VE. Volcanic soils. In: Verheye WH, editor. Encyclopedia of land use, land cover and soil sciences – Soils and soil sciences. Part II. Oxford: Eolss Publisher, Unesco; 2006. v. VII. p. 23-45.

Sadzawka A, Carrasco MA, Grez R, Mora MDLL, Flores H, Neaman A. Métodos de análisis recomendados para los suelos de Chile. Santiago, Chile: Instituto de Investigaciones Agropecuarias (INIA); 2006. (Serie Actas-INIA, 34).

Soil Survey Staff. Keys to soil taxonomy. 12th ed. Washington, DC: United States Department of Agriculture, Natural Resources Conservation Service; 2014.

Stankowski S, Sobolewska M, Jaroszewska A, Gibczyñska M. Influence of biomass ash, lime and gypsum fertilization on macro- and microelement contents in the soil and grains of spring wheat. Soil Sci Annu. 2018;3:177-83. https://doi.org/10.2478/ssa-2018-0018

Sumner ME, Noble AD. Soil acidification: the world story. In: Rengel Z, editor. Handbook of soil acidity. New York: Marcel Dekker; 2003. p. 1-28.

Undurraga P, Hirzel J, Celis J, Perez C, Sandoval MA. Toxicity of paper mill pelletized waste using germination and biomass production as bioindicators. Cienc Inv Agr. 2018;45:147-57. https://doi.org/10.7764/rcia.v45i2.1865

Undurraga P, Zagal E, Sepúlveda G, Valderrama N. Dissolved organic carbon and nitrogen in Andisol for six crop rotations with different soil management intensity. Chil J Agr Res. 2009;69:445-54. https://doi.org/10.4067/S0718-58392009000300018

Vargas JPR, Santos DR, Bastos MC, Schaefer G, Parisi PB. Application forms and types of soil acidity corrective: Changes in depth chemical attributes in long term period experiment. Soil Till Res. 2019;185:47-60. https://doi.org/10.1016/j.still.2018.08.014

Zambrosi FCB, Alleoni LRF, Caires EF. Nutrient concentration in soil water extracts and soybean nutrition in response to lime and gypsum applications to an acid Oxisol under no-till system. Nutr Cycl Agroecosys. 2007;79:169-79. https://doi.org/10.1007/s10705-007-9105-7