

Effect of Al compounds on soil pH and bioavailability of Al in two acid soils

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Abstract: Two aluminum compounds, AlCl_3 and $\text{Al}(\text{OH})_3$, were used to determine the effect of Al compounds on pH and bioavailability of Al in 2 acid soils. Both soils were incubated for periods of 3, 10, and 30 days. Al-tolerant (ET8) and Al-sensitive (ES8) wheat seedlings were used as a testing plant to confirm bioavailability of Al^{3+} in soil solution. Both soils were acidified through the addition of Al compounds to detect the maximum Al^{3+} toxicity level of these compounds. The results showed that the AlCl_3 compound increased the bioavailability of Al^{3+} in soil solutions. In contrast, AlCl_3 decreased bulk soil pH. This combination of high levels of extractable Al and low pH decreased the root proliferation of both ES8 and ET8. However, $\text{Al}(\text{OH})_3$ did not change soil pH, the bioavailability of Al, or the root length of either ES8 or ET8. These findings indicated that $\text{Al}(\text{OH})_3$ did not increase Al^{3+} activity in soil solutions. It can be concluded from this study that AlCl_3 and a short incubation period can be used to manipulate soil pH and the bioavailability of Al in soil for further study. However, $\text{Al}(\text{OH})_3$ cannot be used to manipulate soil pH or the bioavailability of Al in soil.

Key words: Aluminum, bioavailability, ES8, ET8, incubation time, soil type

Introduction

Soil aluminum chemistry is quite complex and different forms of Al available in soil are involved in the retention of anions, cations, and phytotoxicity of Al ions in acid soils (Soon 1993). The type of Al compound is also responsible for producing different forms of bioavailable Al in soil (Skylberg 1999). This availability of different forms of Al ions in soil solutions can be manipulated by the application of different types and rates of Al compounds and lime (Bache 1974). Thus, the amount of Al present in a soil solution depends on the amount and kind of Al compounds present in the soil and its reaction capacity within the soil solution (Magstad 1925).

The speciation of Al compounds is controlled by the hydrolysis of the Al^{3+} ion (Perdue 1985). During the hydrolysis process, a proton is released. This is because of the extent to which acid cations like Al^{3+} induce hydrolysis and decrease soil pH (Milne et al. 1995).

The bioavailability of Al and nutrients depends on their concentration in the soil solution (Curtin and Smillie, 1995). Incubation time can affect different Al compounds in terms of the activity of the Al^{3+} ion in soil solutions (Menzies and Bell 1988). Menzies et al. (1991) concluded that Al^{3+} , toxic to plants, reached a stable concentration in soil solutions after incubation for 1 day. Thus, understanding the

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effect of incubation time on the solubilization of Al compounds is necessary to study Al toxicity in plants.

In this paper, the effects of Al compounds and incubation time on soil pH and extractable Al were examined in order to select an appropriate Al compound for varying the pH and Al toxicity levels in soil and to define a preincubation time for soil amendments. Different soil pH and Al levels were achieved by incubating the soil with various amounts of CaCO_3 , AlCl_3 , and $\text{Al}(\text{OH})_3$, respectively. Lime (CaCO_3) was used to increase soil pH and decrease soil extractable Al. Wheat genotype growth responses were also examined under conditions of high pH and low extractable Al in soil. The hypothesis of this study is that AlCl_3 will increase the bioavailability of Al^{3+} in soil and that a longer incubation period (more than 3 days) will have no further effects on soil pH and extractable Al.

Materials and methods

Soils and basal nutrients

This study used 2 acid soils, a Podosol and a Chromosol (Isbell 2002). The Podosol had an initial pH of 3.76 (0.01 M CaCl_2), a pH buffer capacity (pHBC) of $0.81 \text{ cmol kg}^{-1} \text{ pH}^{-1}$, and an extractable Al value of 3.90 mg kg^{-1} . The Chromosol had an initial pH of 4.10 (0.01 M CaCl_2), a pHBC of $1.46 \text{ cmol kg}^{-1} \text{ pH}^{-1}$, and an extractable Al value of 3.9 mg kg^{-1} . The other basic soil properties are described in Table 1. The Chromosol was mixed with sand at a ratio of 3:1 to reduce its pHBC. Podosol already has a low pHBC, so no sand was added. Both the Podosol and the Chromosol were mechanically sieved to a diameter of <2 mm. Likewise, the sand was sieved in a 600- μm sieve before being mixed with the Chromosol. The following types of basal nutrients were added in

this experiment: $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$, $\text{NaH}_2\text{PO}_4 \cdot 2\text{H}_2\text{O}$, K_2SO_4 , $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$, $\text{MnSO}_4 \cdot 7\text{H}_2\text{O}$, $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$, $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$, H_3BO_3 , $\text{Na}_2\text{MoO}_4 \cdot 2\text{H}_2\text{O}$.

Al compound

This study used the 2 aluminum compounds AlCl_3 and $\text{Al}(\text{OH})_3$. Aluminum sulfate ($\text{Al}_2(\text{SO}_4)_3$) was not used in this experiment because the Al-SO_4 complex makes this compound less toxic (Alva et al. 1991).

Plants and their genotypic characteristics

Both Al-tolerant (ET8) and Al-sensitive (ES8) wheat genotypes were used as test plants. The ES8 and ET8 genotypes were near-isogenic (over 95%) lines differing in Al tolerance at the *ALMT1* locus (Ahn and Matsumoto 2006). The ET8 and ES8 lines were derived from a cross between the Al-tolerant cultivar Carazinho and the Al-sensitive cultivar Egret, with the resulting progeny backcrossed 8 times to Egret or derivatives of Egret (Fisher and Scott 1987).

Experimental design and treatments

The experiment was arranged factorially with the following treatments:

- 1) $4 \text{ AlCl}_3 \times 3 \text{ CaCO}_3 \times 2 \text{ soils} \times 2 \text{ genotypes}$,
- 2) $4 \text{ Al}(\text{OH})_3 \times 1 \text{ CaCO}_3 \times 1 \text{ Podosol} \times 2 \text{ genotypes at } 0.5 \text{ g CaCO}_3$.

Each treatment had 3 replications. The CaCO_3 was used to manipulate soil pH and Al levels in both soils. Both AlCl_3 and $\text{Al}(\text{OH})_3$ levels were 0, 0.5, 1, and 2 g kg^{-1} . High levels of AlCl_3 were added to determine the maximum Al^{3+} activity within the soil solutions.

Soil incubation

Into each pot, 200 g of soil was weighed and kept to 100% field capacity by weighing the pots during incubation. The water in the soil was maintained to field capacity for both the Podosol (19% water per

Table 1. Properties of soils used in this study.

Soil type	Collection site	GPS location	EC (mS cm^{-1})	Field capacity (% water per water)	Sand %	Silt %	Clay %	Total N%	Total C%	$\text{NH}_4\text{-N}$ (mg kg^{-1})	$\text{NO}_3\text{-N}$ (mg kg^{-1})
Podosol	Cranbourne, Victoria	38°06'S 145°16'E	0.113	19	92	4	4.0	0.15	3.40	14.7	10.7
Chromosol	Wagga Wagga, New South Wales	35°05'S 147°35'E	0.069	13	55	25	20	0.05	0.58	2.7	14.1

water) and the Chromosol (13% water per water). To prevent evaporation loss during the incubation period, all the pots were placed in plastic boxes covered with lids in a temperature-controlled room at 25 °C. A standing-water level of 2.5 cm was also maintained at the bottom of each box during incubation. The soils were sampled at 3, 10, and 30 days of incubation using a small core sampler that collected about 6 g per core. All holes were closed after soil collection by shaking or oscillating each pot. All collected soils were air-dried prior to taking pH and Al measurements.

Plant growth

Following the last soil sampling (at 30 days), the soil of each pot was divided into 2 equal parts (90 g each) and transferred into 2 separate 50-mL vials. The soils were properly compacted by tapping each vial while it was being filled with soil. Five uniform pregerminated seeds were placed in each vial and covered with 0.5 cm of soil from the top of the vial. Each vial contained 2 Al-tolerant (ET8) and Al-sensitive (ES8) wheat (*Triticum aestivum* L.) genotypes. Water was added daily by spraying each vial during the plant growth period. The plants were grown in a growth cabinet under controlled environment conditions in which the day and night temperatures were a constant 20 °C, there was 10 h of dark and 14 h of light, and the average light intensity was 210 $\mu\text{M photons m}^{-2} \text{ s}^{-1}$. The plants were harvested 11 days after sowing. The roots were collected and washed with deionized water before measurements were taken.

Measurements

Soil pH was determined with a 1:5 extraction of 0.01 M CaCl_2 after 17 h of shaking. Extractable Al was determined in 0.01 M CaCl_2 using the PCV method (Kerven et al. 1989). Root length was measured and analyzed using the WinRHIZO image analysis system (Regent Instruments Inc., Quebec, Canada) (Arsenault et al. 1995).

Statistical analysis

Results were analyzed using either 2-way or 3-way analysis of variance (ANOVA) using GenStat, 5th edition (Lawes Agricultural Trust, UK).

Results

Bulk soil pH, extractable Al, and root length of wheat seedlings in Podosol

The addition of 500 mg $\text{CaCO}_3 \text{ kg}^{-1}$ soil increased the bulk soil pH by 0.33 units. In the soil amended with 500 mg of CaCO_3 , the addition of AlCl_3 at 2000 mg kg^{-1} resulted in a pH reduction of 0.57 units. In comparison, the addition of $\text{Al}(\text{OH})_3$ at the same rate resulted in a pH increase of around 0.05 units. Bulk soil pH is similar in soils with 500 and 1000 mg kg^{-1} $\text{Al}(\text{OH})_3$ with 500 mg kg^{-1} CaCO_3 availability. However, bulk soil pH dropped significantly ($P < 0.05$) in soils with 500 and 1000 mg kg^{-1} AlCl_3 with 500 mg kg^{-1} CaCO_3 availability (Figure 1a, Table 2).

The concentration of extractable Al in bulk soil remained unchanged following the addition of $\text{Al}(\text{OH})_3$ in a no-lime control treatment. However, it decreased significantly ($P < 0.05$) in soil with 500 mg kg^{-1} CaCO_3 but was not affected by the increasing addition of $\text{Al}(\text{OH})_3$ (Table 2). In contrast, the concentration of extractable Al in bulk soil increased remarkably due to an increasing addition of AlCl_3 in soil with 500 mg kg^{-1} CaCO_3 (Figure 1b).

Root length of Al-tolerant and Al-sensitive wheat seedlings under soil pH and extractable Al amended by AlCl_3

The root lengths of both ES8 and ET8 seedlings increased significantly ($P < 0.05$) with the increase in bulk soil pH (Table 2). In contrast, the root lengths of both ES8 and ET8 seedlings dramatically decreased with the increase in extractable Al in bulk soil. There was no significant difference in root length between the genotypes for both bulk soil pH and extractable Al (Figures 2a and 2b).

Effect of incubation time on soil pH in Podosol and Chromosol

The incubation time did not differ for the 0 and 2000 mg $\text{CaCO}_3 \text{ kg}^{-1}$ treatments under various levels of AlCl_3 availability in Podosol. The incubation time differed for the 500 and 1000 mg $\text{AlCl}_3 \text{ kg}^{-1}$ availabilities for the 0 and 2000 mg CaCO_3 treatments, respectively, in Chromosol. Both soils with 0 AlCl_3 and 0 CaCO_3 become more acidic after 3 days of incubation and generally reached similar values of pH after 30 days of incubation. At 2000 mg kg^{-1} of AlCl_3 , both soils exhibited similar changes in soil pH, which suggests

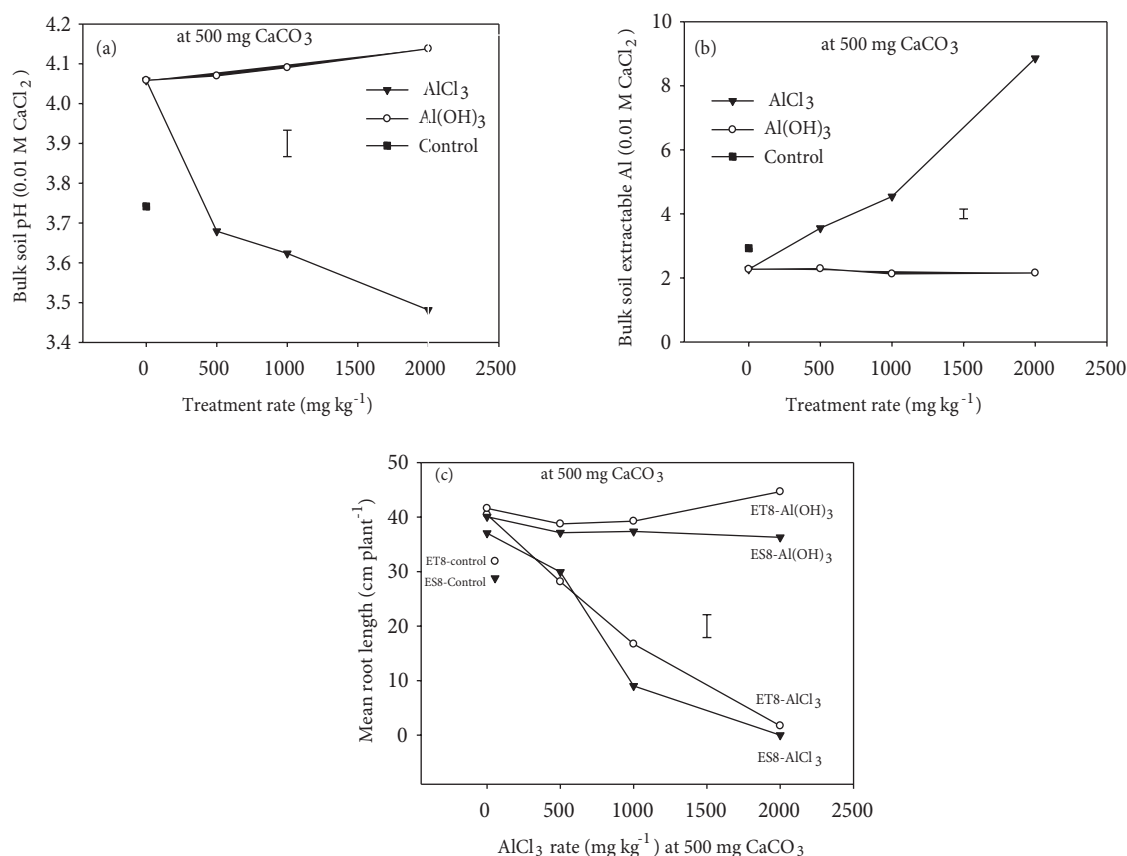


Figure 1. The effect of aluminum compounds ($\text{Al}(\text{OH})_3$ and AlCl_3) on a) bulk soil pH, b) extractable Al in bulk soil, and c) root length of Al-tolerant (ET8) and Al-sensitive (ES8) wheat seedlings grown in Podosol amended with 500 mg kg^{-1} of CaCO_3 . Vertical bars represent the least significant difference ($P = 0.05$) for genotype and Al compound interaction.

that the pH level did not change in 3, 10, or 30 days of incubation. The soil pH dramatically dropped due to different levels of AlCl_3 with $2000 \text{ mg CaCO}_3 \text{ kg}^{-1}$ in Podosol and Chromosol. Soil pH was similar among the 3, 10, and 30 days of incubation at different AlCl_3 levels with $2000 \text{ mg CaCO}_3 \text{ kg}^{-1}$ in Podosol (Figures 3a-3d).

Effect of incubation time on soil extractable Al in Podosol and Chromosol

In general, the concentration of extractable Al was not affected by incubation time in Podosol. The soil extractable Al increased significantly ($P < 0.05$) with an increasing AlCl_3 supply along with 0 CaCO_3 in both Podosol and Chromosol (Table 2). The extractable Al did differ for 3, 10, and 30 days of incubation with 0 and 2000 mg CaCO_3 treatments in Podosol at various levels of AlCl_3 availability. The extractable Al concentration in Chromosol

with 0 CaCO_3 did not differ for 3, 10, or 30 days of incubation at various levels of AlCl_3 availability. The extractable Al concentration in Chromosol with $2000 \text{ mg CaCO}_3 \text{ kg}^{-1}$ treatments differed for 3, 10, and 30 days of incubation under 1000 and $2000 \text{ mg AlCl}_3 \text{ kg}^{-1}$ treatments. The available Al in the soil was 2-fold higher in Podosol than in Chromosol at 2000 mg CaCO_3 under different AlCl_3 treatments. This was because the concentration of extractable Al was originally higher in Podosol than in Chromosol. Similar to that for 0 CaCO_3 , the effect of incubation was not significant at 2000 mg CaCO_3 under different AlCl_3 treatments for either soil (Figures 4a-4d).

Effect of AlCl_3 addition on mean root length in Podosol and Chromosol

The mean root length of ES8 seedlings was initially 29% greater in Chromosol than in Podosol at 0 CaCO_3 availability. It decreased significantly ($P <$

Table 2. Significance levels for the main and interactive effects of soil, compound, and wheat genotype on bulk soil pH, bulk soil extractable Al, incubation time, and root length.

Source of variation	Bulk soil pH	Bulk soil extractable Al	Root length
Compound	***	***	NA
Treatment	***	***	NA
Compound × treatment	***	***	NA
AlCl ₃	NA	NA	***
Al(OH) ₃	NA	NA	**
AlCl ₃ × Al(OH) ₃	NA	NA	n.s.
Genotype	NA	NA	n.s.
Genotype × compound	NA	NA	n.s.
Bulk soil pH	NA	NA	***
Bulk soil extractable Al	NA	NA	***
Genotype	NA	NA	n.s.
Treatment	NA	NA	***
Soil	NA	NA	***
Treatment × soil	NA	NA	***

The symbols n.s. and *** represent $P > 0.05$ and $P \leq 0.001$, respectively. 'NA' indicates that no data are available. Values were means of 6 replicates for bulk soil pH as well as extractable Al in bulk soil (as no plant effect in bulk soil) and 3 replicates for root length. Sampling was done after 3, 10, and 30 days of incubation and plants were grown after 30 days of incubation.

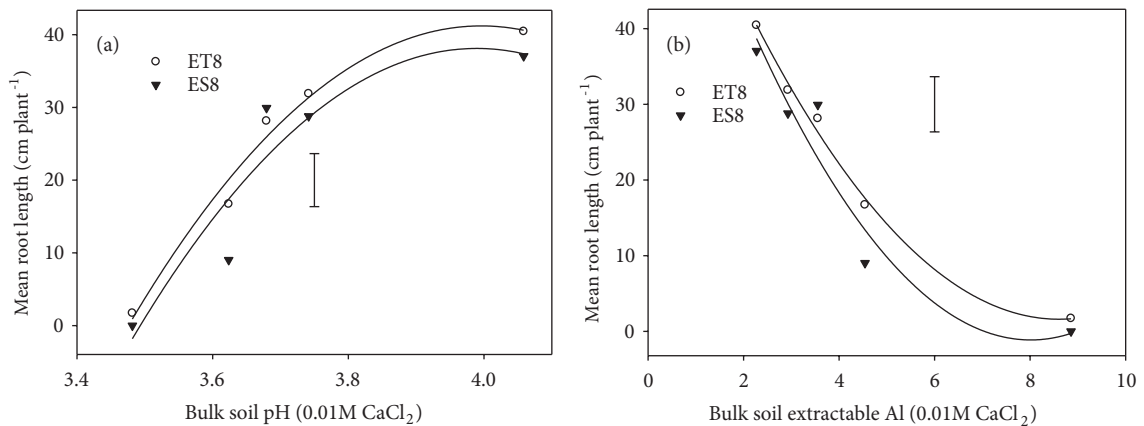


Figure 2. Changes in mean root length due to an increase in a) bulk soil pH and b) extractable Al in bulk soil for the Al-tolerant (ET8) and Al-sensitive (ES8) wheat seedlings. Data were mean values of 3 replicates. Vertical bars represent the least significant difference ($P = 0.05$) for genotype and extractable Al in both bulk soil and bulk soil pH interaction.

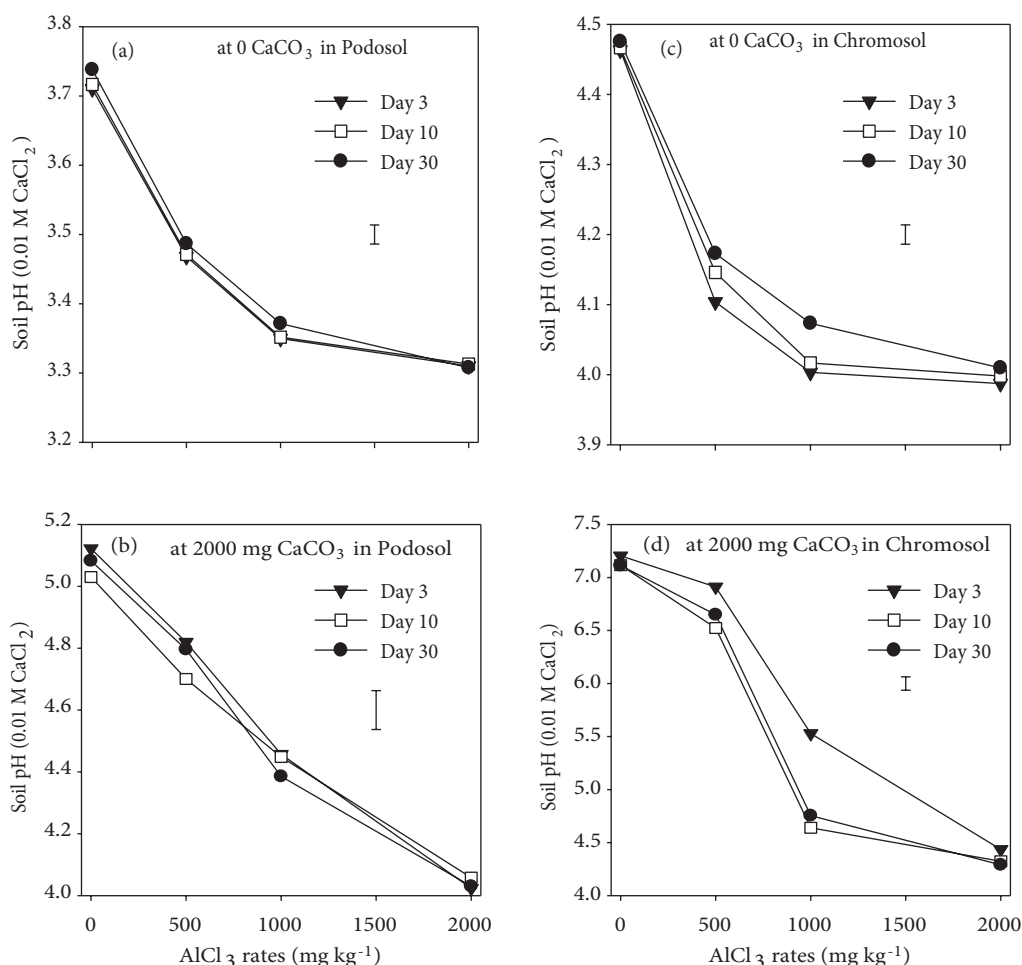


Figure 3. The effect of incubation time on soil pH for a) Podosol at 0 mg of CaCO₃ availability, b) Podosol at 2000 mg of CaCO₃ availability, c) Chromosol at 0 mg of CaCO₃ availability, and d) Chromosol at 2000 mg of CaCO₃ availability under different rates of AlCl₃ addition. Vertical bars represent the least significant difference (P = 0.05) for day, soil, and treatment interaction. Data were the average values of 3 replicates.

0.05) with the addition of AlCl₃ along with 0 CaCO₃ (Table 2). The mean root length of ES8 seedlings in Podosol differed significantly (P < 0.05) between the 0 and 2000 mg CaCO₃ treatments under different AlCl₃ availabilities (Table 2). However, the mean root length of ES8 seedlings in Chromosol did not significantly differ between the lime treatments at the 0 and 2000 mg AlCl₃ kg⁻¹ availabilities, respectively. Similarly, mean root length did not differ between 0 and 2000 mg of CaCO₃ along with 2000 mg AlCl₃ kg⁻¹ treatment in Chromosol. Moreover, the mean root length of ES8 seedlings was greater for the 0 than the 500 mg CaCO₃ kg⁻¹ treatment with the 500 and 1000 mg AlCl₃ kg⁻¹ treatments, respectively, in Chromosol (Figures 5a and 5b).

Discussion

Effect of Al compounds on soil acidity

The 2 Al compounds, aluminum chloride (AlCl₃) and aluminum hydroxide (Al(OH)₃), differed markedly in their ability to increase the bioavailability of Al in soil. The bulk soil pH declined and the concentration of extractable Al increased with the addition of AlCl₃ to the soil. In contrast, these measurements did not change with the addition of Al(OH)₃ to the soil. As a result, root growth of both the Al-tolerant (ET8) and Al-sensitive (ES8) wheat seedlings declined only with increasing amounts of AlCl₃ (Figures 1a-1c, Table 2). The differences between these 2 Al compounds can be attributed to their different solubility product

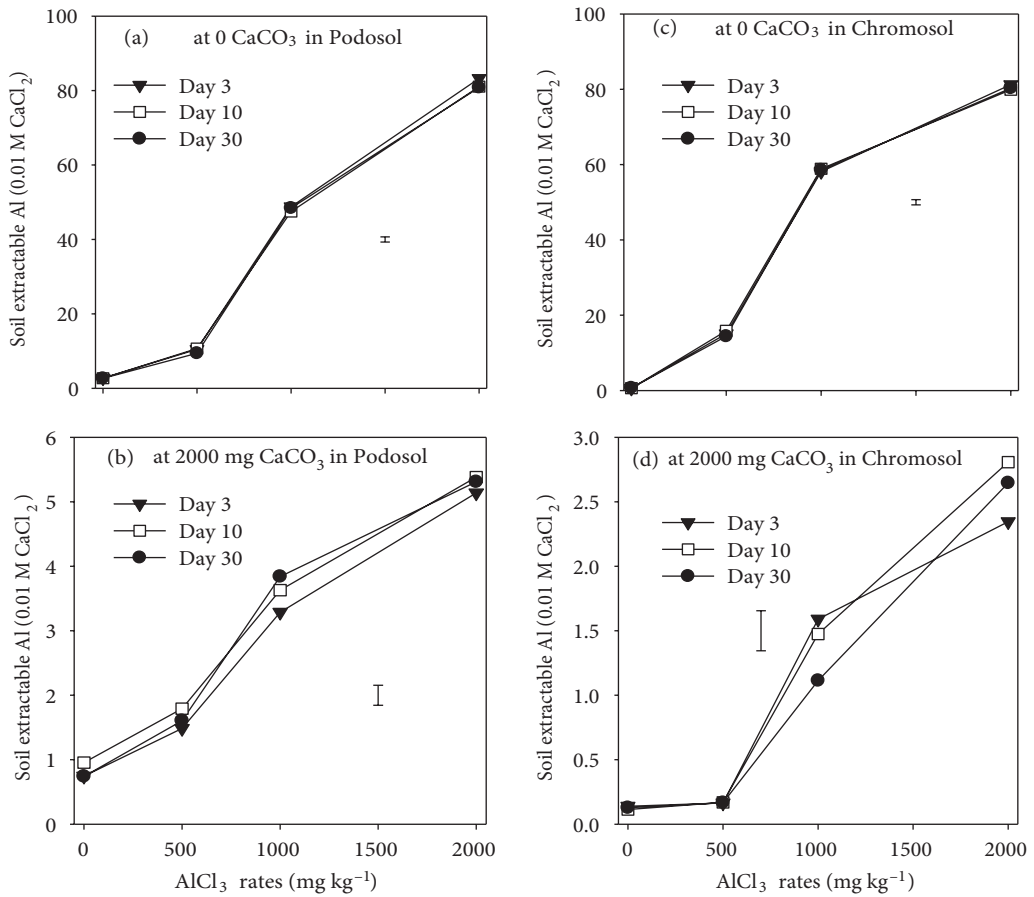


Figure 4. The effect of incubation time on soil extractable Al for a) Podosol at 0 mg of CaCO₃ availability, b) Podosol at 2000 mg of CaCO₃ availability, c) Chromosol at 0 mg of CaCO₃ availability, and d) Chromosol at 2000 mg of CaCO₃ availability under different rates of AlCl₃ addition. Vertical bars represent the least significant difference (P = 0.05) for day, soil, and treatment interaction. Data were the average values of 3 replicates.

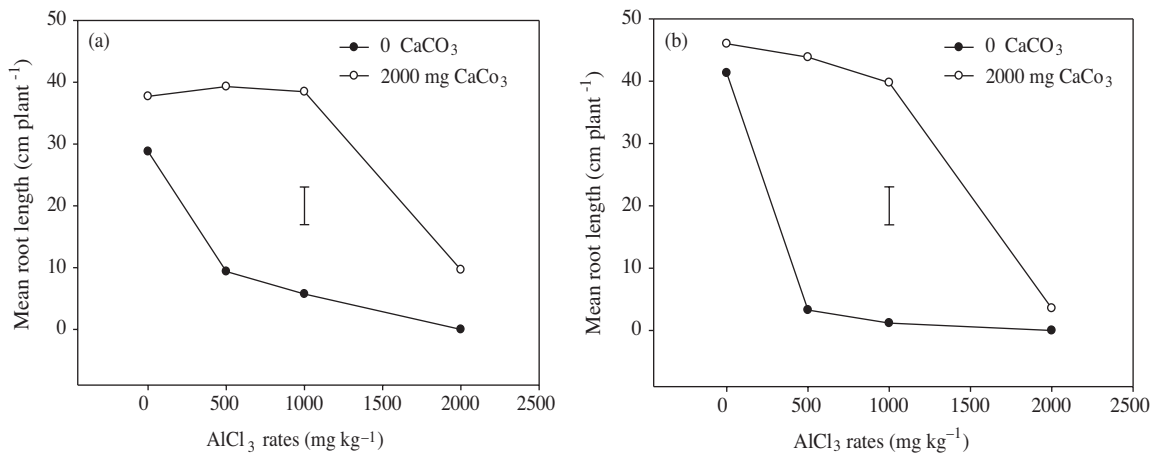


Figure 5. The effect of AlCl₃ addition on the mean root length of Al-sensitive (ES8) wheat seedlings grown for 11 days in a) Podosol and b) Chromosol. The soils were incubated for 30 days prior to commencement of the plant growth experiment. Vertical bars represent the least significant difference (P = 0.05) for soil × treatment interaction. Data were the average values of 3 replicates.

constants (K_{sp}). The K_{sp} values for $AlCl_3$ and $Al(OH)_3$ at 25 °C are 2.04×10^4 and 1.8×10^{-33} , respectively (Chang 2010). Thus, the mass of $AlCl_3$ that will dissolve in 100 mL of water at 25 °C is 111 g, whereas $Al(OH)_3$ practically does not dissolve in water (Budavari 1989).

The chemical properties of the $AlCl_3$ and $Al(OH)_3$ compounds and their solubility in the soil solution are responsible for the differences in their effects on soil pH and extractable Al. The dissociation of $AlCl_3$ ($AlCl_3 \rightarrow Al^{3+} + 3Cl^-$) initiates acidity due to the reaction of Al^{3+} with water in the soil solution ($Al^{3+} + 2H_2O \rightarrow Al^{3+}(OH)^{2+} + 2H^+$). Thus, $AlCl_3$ dissociates to Al^{3+} in the soil solution and increases both the bioavailability of Al in the soil and of Al^{3+} in the soil solution. This Al^{3+} also reacts with water to form mononuclear hydroxy-Al species ($Al(OH)^{2+}$), releasing H^+ ions and lowering the soil pH (Serrano 2003). In complete contrast, $Al(OH)_3$ does not dissolve in the soil solution at pH 4.0-6.0, and so it does not increase the bioavailability of Al in soil solution. This experiment demonstrates that $AlCl_3$ is the preferred compound for manipulating different levels of bioavailability of Al^{3+} in soil. It was therefore used in subsequent experiments in this study.

The effect of incubation time

Increasing incubation time from 3 to 30 days did not markedly affect the level of soil pH (Figures 3a-3d) or extractable Al (Figures 4a-4d), with the exception of 2000 mg of $CaCO_3$ in Chromosol, following the addition of $AlCl_3$ to the soils in this study. There was a rapid decrease in the pH and an increase in extractable Al in both soils after the application of $AlCl_3$; the effects stabilized during the measurement on day 3 and remained constant over the 30-day incubation period. The high solubility of $AlCl_3$ means that it quickly dissociates in water followed by the hydration of Al^{3+} forming mononuclear hydroxyl-Al species like $Al(OH)^+$ (Richens 1997). Likewise, Tanaka et al. (1987) reported that the chemical species of Al changed as soon as an $AlCl_3$ solution was added to the soil.

Other studies have reported similar effects of incubation time for inorganic and organically complexed forms of Al. Thus, neither organic nor inorganic Al significantly changed with incubation time and both remained stable for periods of time. For

example, Menzies et al. (1991) incubated soil samples at 28 °C from 1 to 64 days. They found that soil pH and extractable Al did not change significantly with the extended (up to 64 days) incubation times. They also reported that the plant-toxic monomeric Al (Al^{3+}) reached a stable concentration in soil solution after 1 day of incubation. It should be pointed out that they did not manipulate soil Al. They just collected 5 acid soils of widely divergent chemical and physical characteristics and incubated them at 28 °C from 1 to 64 days. Similarly, Menzies and Bell (1988) also found that incubating a Krasnozem surface soil at 28 °C for 1 day was sufficient to attain an equilibrium condition for most ions, similar to that present under field conditions. Moreover, they did not manipulate soil Al but incubated soils for a period of up to 16 days. These findings indirectly suggest that short incubation periods are required for experiments in which the activity of Al^{3+} in soil is manipulated. Thus, a short incubation would allow Al^{3+} concentrations to stabilize in the soil solutions.

Effect of soil type

The Podosol was an inherently more acidic soil than the Chromosol in this experiment. The Podosol had a lower pH (3.8 in 0.01 M $CaCl_2$) and a higher concentration of extractable Al (3.9 mg kg^{-1}) than Chromosol. Even when 2000 mg of $CaCO_3$ was applied to both soils, the Podosol still had a lower pH and a higher extractable Al level than the Chromosol (Figures 3 and 4, Table 2). Thus, there was less root proliferation of the Al-sensitive wheat (ES8) seedlings in the Podosol than in the Chromosol both with and without $CaCO_3$ addition, due to the lower pH and higher extractable Al (Figures 5a and 5b, Table 2).

This particular acidic Podosol soil was collected from the surface layer of soil at Cranbourne, Victoria, Australia. Its acidity, with a low pH and high extractable Al, markedly reduced the growth of wheat roots. In 2 preliminary experiments (data not presented), it was not possible to demonstrate differences in root growth between the Al-sensitive ES8 and more Al-tolerant ET8 wheat genotypes as the root growth of both genotypes was markedly suppressed by the acidic properties of this surface soil. A soil with less acidic properties will be needed for subsequent experiments, in which the differential effects of acidic soil properties on these 2 genotypes

will be studied. Therefore, another Podsol collected from the subsoil at Frankston, Victoria, with an initial pH of 4.5 and extractable Al of 5.0 mg kg⁻¹, will be used in subsequent experiments involving the manipulation of Al concentrations using the addition of AlCl₃.

One type of Al compound affected soil pH and Al³⁺ activity in soil solution. The AlCl₃ compound increased the concentration of extractable Al and decreased pH in soil. The root length of ES8 and ET8 decreased during AlCl₃ treatments due to increases in the bioavailability of Al³⁺ in the soil. In contrast, Al(OH)₃ had no effect on soil pH or the activity of Al³⁺ in soil solution. As a result, ES8 and ET8 had similar root proliferation with or without the addition of Al(OH)₃. Moreover, no remarkable

incubation effect was found for any of the treatments regarding soil pH and extractable Al. Therefore, for successive experiments, a short incubation period with an increased temperature (7 days at 30 °C) was selected for soil incubation and AlCl₃ was chosen to manipulate soil available Al (Iqbal et al. 2010).

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