Ca-H-Al exchanges and aluminium mobility in two Chinese acidic forest soils: a batch experiment

J. Guo · R. D. Vogt · X. Zhang · Y. Zhang · H. M. Seip · H. Tang

Abstract Exchange reactions between Ca^{2+} , H^+ and Al species and their effects on the aluminium mobility in two Chinese acidic forest soils were studied. The study was based on a batch experiment using extractant solutions with different base cation (calcium) concentrations and pH. The experimental data showed that increased Ca^{2+} concentrations increased the release of soil hydrogen—and aluminium ions, especially from the more acid soil. In agreement with a cation exchange process, the treatment with Ca^{2+} extracts gave significantly decreased soil aluminium saturation (AlS) and increased calcium saturation (CaS) on the ion exchanger. Geochemical calculation using ALCHEMI program showed that activities of Al^{3+} in the extracts were all strongly under-saturated with respect to any gibbsite mineral in the studied pH region (i.e. below 4.1). There were instead apparently two different mechanisms controlling the activities of Al^{3+} in extracts. At pH between about 4.1 and 3.7, the Al^{3+} activity did not change significantly with pH. This is especially the case in the more acid soil. Apparently there are no sizeable pools available to release Al in this pH region. At pH below 3.7 (induced by higher Ca^{2+} concentration) the activity of Al^{3+} increased with H^+ though not in a pattern that complies with a gibbsite solubility control. An increase of base cation deposition would therefore mainly enhance the release of hydrogen ions between pH 4.1 and 3.7 and aluminium ions below pH 3.7 from Chinese mature acidic soils. This

will cause an increased acidity of soil water in the short term and a decrease in the soil acidity in the long term. More attention should be paid to this fact in Chinese acid rain studies and control options.

Keywords Acidification · Exchange reactions · Aluminium mobility · Base cations · Chinese acidic forest soils

Introduction

Soil acidification is a major effect of acid deposition on Chinese terrestrial ecosystem (Pan 1992; Dai and others 1998). 8.4% of the country is receiving a precipitation with a pH below 4.5 and S deposition that is exceeding the critical load (i.e. Acid rain control zone), according to the Two control zone strategy laid out in the Chinese 10th 5-year plan. The mobilization of soil solid-phase aluminium (Al) is known as the dominating mechanism buffering acid input in acidified forest soils (Mulder and others 1989; Berggren and Mulder 1995). However, the exact processes controlling aluminium (Al) activities in Chinese mature acidic soil are still not fully understood. Elevated Al concentrations are toxic to terrestrial vegetation and aquatic organisms (Rosseland and others 1990). Improved insight into the processes controlling Al mobilization is therefore essential to better predict the potential for soil acidification and its effects on biota.

In addition to acid rain, a high base cation (i.e. K^+ , Na^+ , Ca^{2+} , Mg^{2+} and NH_4^+) loading is important feature of Chinese atmospheric deposition (Larssen and others 1999a; Larssen and Carmichael 2000). Several studies, as well as conventional soil/soil water interaction concepts, imply that enhanced deposition of base cations to acid soils will lead to the release of soil Al and H^+ (Reuss and Johnson 1986; Alva and Sumner 1990; Zhu and others 1994). Many short-term sea-salt episodic changes in stream water acidity have been reported to be related to exchange reactions between base cations, hydrogen ion and aluminium (Seip and others 1989; Mulder and others 1990; Vogt and Muniz 1997; Andersen and Seip 1999; Stutter and others 2001). The temporal variation in the

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J. Guo (✉) · R. D. Vogt · X. Zhang · Y. Zhang · H. Tang
Research Centre for Eco-Environmental Sciences,
The Chinese Academy of Sciences,
P.O. Box 2871, 100085 Beijing, China
E-mail: guojingheng@vip.sina.com
Tel.: +86-10-62849117
Fax: +86-10-62923563

R. D. Vogt · H. M. Seip
Department of Chemistry, University of Oslo, P.O. Box 1033,
Blindern, 0315 Oslo, Norway

concentration of especially Ca is large in the throughfall deposition in Chinese forested sites. This is mainly due to a high and variable loading of alkaline dust along with large seasonal variation in the amount of precipitation. Therefore, the exchange reactions between base cations with H^+ and Al in Chinese acidic soils are of special importance.

In this paper, acidic forest soils, collected from within the Chinese *Acid rain Control zone*, were extracted using acid solutions with different base cation (calcium) concentrations and pH to study the response reactions and their effects on Al mobility.

Materials and methods

Site descriptions

The soils used in this experiment were collected from the Luchongguan (LCG) and Caijiatang (CJT) catchments. These two catchments are monitoring sites in the *Integrated Monitoring Program on Acidification of Chinese Terrestrial Systems* (IMPACTS; Tang and others 2001). The LCG catchment (106°43'E, 26°38'N) is about 10 km northeast of Guiyang city, the capital of Guizhou province, at an elevation of about 1,320–1,400 m. The climate is mild (annual mean temp.: 15.3 °C) and humid with the annual precipitation about 1,118 mm. The LCG catchment was selected for studies of the effects of acid deposition in the early 1990s. This catchment is representative for a suburban site located in the proximity of numerous large atmospheric emission sources resulting in high deposition of sulphur as well as alkaline dust. Only for wet deposition the flux of S was 4.4 g S m⁻² year⁻¹ in 2002. The dominant ions in the precipitation are Ca^{2+} and SO_4^{2-} ; the volume-weighted average pH of precipitation was about 4.5 in 2002. The percentile concentrations of Ca^{2+} in ground vegetation throughfall ranged from 168 to 2,442 $\mu\text{eq L}^{-1}$ with median value of 494 $\mu\text{eq L}^{-1}$ during 2002 (IMPACTS, unpublished data).

The CJT catchment is a few km west of Shaoshan City (112°26'E, 27°55'N), southeast of Changsha city, capital of Hunan province, at an elevation of about 450–500 m. Annual mean temperature is about 17.5 °C and the annual precipitation is 1,524 mm. Precipitation in CJT catchment is acidic with volume-weighted pH of 4.47 (in 2002). The CJT site represents a site with high S and N (5.3 and 2.0 g m⁻² yr⁻¹, respectively, from wet only) loading and with somewhat less sensitive soils. The percentile concentration of Ca^{2+} in ground vegetation throughfall ranged from 141 to 735 $\mu\text{eq L}^{-1}$ with median value of 378 $\mu\text{eq L}^{-1}$ during 2002 (IMPACTS, unpublished data).

The LCG and CJT soils are classified as *Haplic Alisol* and *Orthic Acrisol*, respectively, in FAO systems. In the Chinese soil classification systems both soils are considered to be acidic mountain yellow soils. The LCG soil was collected from the AB-horizon, while the CJT soil was collected from the A-horizon. More information about the LCG and CJT catchments can be found in Lydersen and others (1997).

Experiment

Thirty extraction solutions were made using HCl, $CaCl_2$ and ion-exchanged water. First three acidic stock solutions were prepared by diluting HCl to pH 3.46, 4.24 and 4.84. Concentrated $CaCl_2$ (0.1 N) was then diluted to 10 different Ca^{2+} concentrations (0, 0.025, 0.05, 0.1, 0.2, 0.5, 1.5, 2.5, 5.0 and 12.5 mmol L⁻¹) using the three acidic stock solutions. The 30 acidic extractants were classified into three series according to their pH value (i.e. series 1 (S1): pH 3.46; series 2 (S2): pH 4.24 and series 3 (S3): pH 4.84). This pH range was selected to cover the pH values commonly found in precipitation in the Chinese acid rain control zone. Calcium is usually the dominating base cation in precipitation and especially in throughfall. A simple matrix was selected in the extraction solution instead of ambient precipitation quality (with also high NH_4^+) to simplify and highlight the effects of alkaline dust on soil solution. The range in Ca^{2+} concentration was set higher than in real precipitation to enhance the response and to include the effect of extreme episodic events, such as dust storms.

All soils were air-dried and passed through a 2.0 mm sieve, thoroughly mixed and evenly divided into 25 g subsets and transferred to 30 bottles. The 30 extraction solutions were introduced into these 30 bottles. The ratio of solution to soil was 10:1. The 0.5-L bottles were then shaken for 12 h every 24 h for two days. This shaking time was assumed sufficient in order to reach equilibrium as it is considerably longer than that used in other soil experiments found in the literature (e.g. Liao and others 1998). The suspension was then decanted, centrifuged and passed through a 0.45- μm filter membrane (Millex-HV Millipore). The soil sediments were rinsed using 500 mL ion-exchanged water, dried at 40 °C and stored prior to determination of soil parameters.

Analytical methods

The pH values in solutions were measured directly using a combination pH electrode (Rex E-201-C-9) (ISO 1994c). Major anions (SO_4^{2-} , NO_3^- , Cl^- and F^-) and cations (Ca^{2+} , Mg^{2+} , K^+ , Na^+ and NH_4^+) in the $CaCl_2$ extracts were determined by Ion Chromatography (IC) (Dionex DX 120 using IonPac AS14A and IonPac CS12 analytical columns) (ISO 1992, 1998). Silica in the extracts was analysed using the molybdosilicate colorimetric method (Goltermann and others 1978). Their content of dissolved organic matter (DOM) was approximated using ultraviolet absorbance at 254 nm (McMurry 1990). The monomeric (i.e. mainly labile) aluminium (Ala) fraction was determined by measuring the absorbency of the 8-hydroxy-quinoline complex with the aluminium that was formed within 20 s (Barnes 1975). The organic fraction (Alo) of the Ala was determined by first passing the sample through a pH adjusted Amberlight IR-120 cation exchange column. The inorganic monomeric Al fraction (Ali) was calculated as the difference between Ala and Alo (Driscoll 1984). The concentrations of different Al-species (e.g. Al^{3+}) in the Ali fraction were calculated by using the computer program ALCHEMI (Schecher and Driscoll 1987). Ion balance (i.e. $IB = (\Sigma \text{cations} - \Sigma \text{anions}) / (\Sigma \text{cations} + \Sigma \text{anions})$) was also

calculated to ensure the data quality. Discrepancy in IB values for all samples were less than 10%.

Soil pH values were determined in suspensions of 5 g air-dried and sieved soil with 25 mL ion-exchanged water that had been shaken for 0.5 h (ISO 1994b). The effective cation exchange capacity (CEC_E) along with the aluminium saturation (ALS), base saturation (BS) and calcium saturation (CaS) was measured using the BaCl_2 extraction method (Hendershot and Duquette 1986). CEC_E was calculated as the sum of equivalent concentration of major cations (i.e. H^+ , Ca^{2+} , Mg^{2+} , K^+ , Na^+ , Al^{3+} and Fe^{3+}) in BaCl_2 extracts. ALS, CaS and BS were expressed as their percentage of CEC_E . This method, used on acid soils, is simpler yet compatible and produces similar results as the ISO (1994a) method (Austnes 2003). All the major metal cations in the BaCl_2 extracts (i.e. Ca^{2+} , Mg^{2+} , K^+ , Na^+ , Al^{3+} and Fe^{3+}) were determined using ICP-AES (ISO 1996), and H^+ was determined using a combination pH electrode (Rex E-201-C-9). The content of soil organic matter was determined using the K_2CrO_7 oxidation method (Anonymous 1998).

Results

Soil properties

Some basic soil parameters are listed in Table 1. The LCG soil is more acidic compared to the CJT soil, with a lower pH and a base saturation (BS) less than 20%. The A horizon soil from CJT had a soil organic matter content of 5.1%, while the AB horizon soil from LCG had a slightly lower organic content. Despite this the effective cation exchange capacity (CEC_E) of LCG soil was 106 meq kg^{-1} , which is considerably higher than that of CJT soil. This is likely due to higher clay content.

Release of hydrogen and aluminium ions

With an increase in Ca^{2+} concentration in the extraction solution the pH values of the extracts decreased. At lower Ca^{2+} concentrations ($<0.5 \text{ mmol L}^{-1}$) the pH values of extracts were also significantly influenced by the H^+ in the original extraction solution. At higher Ca^{2+} concentrations the pH of all three extract series tended to level off at a pH of about 3.42. None of the extracts had pH significantly higher than the soil pH (see Table 1), despite higher pH in two of the extraction solution series (S2 and S3). The shifts in H^+ activities between extracts and the original

extraction solution ($\text{Hrel.} = \text{H}^+ \text{ in extracts} - \text{H}^+ \text{ in extractants}$) are shown in Fig. 1. Positive Hrel.-values indicate H^+ release. Extraction series (S2 and S3), with initial pH higher than soil pH, gave as expected higher H^+ release (Hrel.) than extraction series S1 with a pH lower than soil pH. Moreover, H^+ release was less from the CJT soil than from the more acid LCG soil.

Al^{3+} concentrations in the extracts increased with increased Ca^{2+} concentrations (Fig. 2) in the extraction solution. For both LCG and CJT extract, the increasing rate of Al^{3+} concentration decreased slightly with increasing Ca^{2+} concentration. Minor differences in the releases of Al between the extraction series followed the original H^+ concentration in the extraction solutions (i.e. S1>S2>S3), implying that original H^+ in extraction solutions also was a contributor to the release of soil aluminium, albeit secondary. The more acid LCG soil released considerably more Al than CJT soil.

Increasing substitution of Ca for Al on the soil exchanger with increasing Ca^{2+} in the extract led to a general trend of decreased aluminium saturation (ALS) and increased calcium saturation (CaS) (Fig. 3). Changes in CaS were therefore negatively correlated with changes in ALS ($r^2=0.98$ and 0.95 for LCG and CJT soil, respectively). The changes were large at low BS and tended to level off at BS greater than 20%, especially in CJT. Changes in ALS and

Fig. 1

The release of H (Hrel.) for varying Ca^{2+} concentration in the extracts

Table 1

Basic parameters of the soil samples

Soils	LCG	CJT
Soil horizon	AB	A
Soil pH _{water (5:1)}	4.02	4.13
Soil pH _{BaCl2}	3.24	3.62
Soil organic carbon (%)	4.8	5.1
CEC (meq/kg soil)	106	68.2
ALS (%)	81	72
BS (%)	12	23
CaS (%)	7.3	14
HS (%)	5.5	4.4

Fig. 2

The changes in Al^{3+} concentration with increased Ca^{2+} concentration in extracts

CaS were also somewhat influenced by pH in the extraction solution. The CaS increased and the AIS decreased more in soils equilibrated with extraction solutions with high pH, i.e. S1:S2 < S3. The increases in CaS were slightly greater than the decrease in AIS due to exchange between Ca^{2+} and cations other than Al (e.g. H^+).

Aluminium mobility

In most soil acidification models the Al^{3+} activity in soil solution is assumed to be controlled by dissolution equilibria of gibbsite minerals ($\text{Al}(\text{OH})_3$) (Tiktak and Van Grinsven 1995).

$$\text{pAl} = 3\text{pH} + \text{pKsp} \quad (2)$$

where pKsp denotes the solubility constant of the gibbsite phase that ranges from -8.11 for synthetic gibbsite to -10.8 for amorphous $\text{Al}(\text{OH})_3$. An ion exchange control of the Al^{3+} activity also dictates the same pH vs. pAl relationship, though a lower pK value is possible.

pAl increased with increasing pH in both LCG and CJT soil extracts (Fig. 4). However, geochemical calculation using the ALCHEMI program suggested that the extracted solutions were undersaturated with respect to even synthetic

Fig. 3

The changes in calcium saturation (CaS) and aluminium saturation (AIS) with Ca^{2+} concentration in extracts

gibbsite. Moreover, pH-pAl linear regression gave slopes of 0.97 and 1.11 for LCG and CJT extracts, respectively, which strongly deviate from the gibbsite model dictating a slope of 3. A closer inspection shows that in the extracts with pH higher than 3.7, the Al^{3+} activity did not actually change significantly with pH, especially in LCG extracts. Though in the extracts with pH less than 3.7, the Al^{3+} activity did in fact increase with the decrease of extract pH. These pAl vs. pH diagrams differ from those presented in several previous field studies and soil acidification experiments (see Larssen and others 1999b; Berggren and Mulder 1995). The reason for this is the lower pH range (i.e. below pH 3.7) that is achieved in these soils. In the pH range down to pH 3.7 the pAl data coincide with previous reported data, while at pH less than 3.7 the Al^{3+} activity appears to be governed by other mechanisms.

Discussions

From Figs. 1 and 3 it can be seen that at low Ca^{2+} concentrations ($<0.5 \text{ mmol L}^{-1}$) the main response of soils was to release H^+ (Fig. 1) by exchange for Ca^{2+} on the ion exchanger (Fig. 3), although some exchangeable Al was

Fig. 4

The relationships between extract pAl and pH in the extracts

also mobilized. This resulted in a generally greater increase in the CaS than a decrease in AIS (Fig. 3). This situation prevailed until the pH in the extracts dropped below 3.7 (at Ca^{2+} concentrations in the extraction solution $>0.5 \text{ mmol L}^{-1}$). At pH values less than 3.7 a significant amount of Al^{3+} became mobilized (Fig. 2) from exchanger, causing a decline in the AIS that was more comparable to the increase in CaS (Fig. 3). Because H^+ was also partly consumed through aluminium dissolution, the H^+ release rate (Fig. 1) decreased more significantly than the Al^{3+} release rate (Fig. 2) at higher Ca^{2+} concentration (lower pH).

There appears, therefore, to be two different mechanisms controlling the relationship between Al^{3+} and H^+ in the pH range of 4.1 to 3.3. At pH above 3.7, H^+ concentrations were mainly controlled by its exchange reaction with Ca^{2+} . Al^{3+} activities did not change significantly with pH, especially in LCG soil (Fig. 4, Table 2), although likely a set of minor

aluminium pools were mobilized and depleted. This caused the decreasing pKsp value or a pAl/pH slope of 0.08 and 0.84 at LCG and CJT, respectively (Table 2). These small Al pools were least important at the more acid LCG site probably because these rather easily available Al pools already have been depleted in such acidic soils. At pH lower than 3.7, there appears to be new dominating process controlling Al^{3+} activity. Although Al^{3+} activities increased significantly with pH decrease (Fig. 2), it still seemed to be different from an ion exchange model. Saturation index (SI) with respect to jurbanite, calculated by ALCHEMI program, showed a rather constant level (-1.34 ± 0.06 and -1.52 ± 0.08 for LCG and CJT soil extracts, respectively) at $\text{pH} < 3.7$. This may indicate that some jurbanite-like co-desorption possibly control the Al^{3+} activity.

Alkaline dust is an important constituent of atmospheric deposition in China (Larssen and others 1999a; Lydersen and others 1997). Many studies have emphasized the important role of alkaline dust (e.g. CaCO_3 , CaO) in neutralizing atmospheric acid deposition (Wang and Wang 1996). In the long term the atmospheric deposition of Ca^{2+} also has an antagonistic effect on the soil acidification. This study shows that in the short term large temporal fluctuations in the Ca^{2+} concentration in deposition clearly plays an important role in short-term changes in water chemistry in acid soils. Comparable studies, regarding sea salts, have shown similar results in northern Europe (Rosenqvist 1978; Mulder and others 1990; Vogt and Muniz 1997; Andersen and Seip 1999). Although the applied Ca^{2+} concentrations comprise higher values than normally found in precipitation, the results corroborate earlier claims that more emphasis should be given to the base cation content in atmospheric deposition in China. Such studies are scarce in China. Careful evaluation of the role of atmospheric base components is therefore necessary in acidification research and acid rain controls measures.

Summary

Increased Ca^{2+} input concentrations enhanced extract acidity and aluminium concentrations in soil with high acidity. The increase of Ca^{2+} concentration in extracts led also to a significant decrease in soil aluminium saturation (AIS) and increase in calcium saturation (CaS), due to cation exchange processes. Al^{3+} activities in the extracts were strongly undersaturated with respect to gibbsite minerals in the pH range (4.1–3.3) studied. At pH between 4.1 and 3.7, the pAl did not vary significantly. While at pH in the

Table 2

The regression results of pAl-pH relationship in extracts

Soil	Data	Slope	Intercepts	R	N	SD
LCG	Total data	0.97 ± 0.10	1.16 ± 0.36	0.88	30	0.12
	pH < 3.7	1.82 ± 0.13	-1.97 ± 0.45	0.97	14	0.06
	pH > 3.7	0.08 ± 0.12	4.45 ± 0.46	0.17	16	0.06
CJT	Total data	1.11 ± 0.07	0.34 ± 0.28	0.95	30	0.07
	pH < 3.7	1.34 ± 0.18	-0.46 ± 0.63	0.91	14	0.06
	pH > 3.7	0.84 ± 0.15	1.39 ± 0.56	0.84	16	0.06

extracts below 3.7 the Al^{3+} activity increased significantly with H^+ . The observed data pattern must therefore instead be explained by two different processes controlling Al^{3+} activities. Short term increases in deposition concentration of base cations may enhance the release of aluminium and hydrogen ions from mature acidic soils and thereby cause episodic increases in the acidity of soil water and possibly surface water. This should be paid more attention to in Chinese acid rain studies and control options.

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