

Effect of Oxalic Acid, Na⁺, NH₄⁺, and Fe³⁺ on Release of Fixed Potassium and Basal Distance of Smectite in Smectitic Soils

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ABSTRACT

The effect of Oxalic Acid, Na⁺, NH₄⁺, and Fe³⁺ on Release of Fixed Potassium and Basal Distance of Smectite in Smectitic Soils (D. Nursyamsi): Laboratory experiment aimed to study effect of oxalic acid, Na⁺, NH₄⁺, and Fe³⁺ on the release of fixed potassium and basal distance of smectite in smectitic soils was conducted in Laboratory of Soil Sciences, Graduate School of Agriculture, Kyoto University, Japan. The experiment used four of topsoil samples of Typic Hapludalfs (B1), Chromic Endoaquerts (B2), Typic Endoaquerts (B3), and Typic Haplustalfs (B4) taken from Jonggol (Bogor), Sidareja (Cilacap), Padas (Ngawi), and Todanan (Blora) respectively. This experiment also used randomized completely block design, six treatments, i.e.: control (water), 1 N of oxalic acid, (oxalic acid+NaOH) pH=7, Na⁺, NH₄⁺, and Fe³⁺ respectively and they were replicated three times. After extracting the samples, potassium concentration in supernatant was measured by Atomic Absorption Spectrophotometer (AAS) method and basal distance of smectite in clay paste was measured by X-Ray Diffraction (XRD) method. The result showed that oxalic acid, (oxalic acid+NaOH) pH=7, Na⁺, NH₄⁺, and Fe³⁺ increased the release of fixed potassium by clay in all tested soils about 24.63-37.44%, 31.88-45.38%, 25.37-48.35%, 27.48-42.32%, and 28.17-35.49%, respectively. Among the treatments, (oxalic acid+NaOH) pH=7 was the most effective in releasing fixed K at Alfisols, while Na⁺ at Vertisols. Oxalic acid+NaOH pH=7, Na⁺, and Fe³⁺ increased the basal distance of smectite in the soils about 10.41%, 11.48%, and 15.30%, respectively. Among the treatments, Fe³⁺ was the highest in increasing the basal distance of smectite in the soils.

Keywords: Basal distance; Na⁺, NH₄⁺, and Fe³⁺, oxalic acid, release of potassium, smectitic soils

INTRODUCTION

Potassium (K) is an element essential for plant growth and its importance in agricultural production is well recognized. Total K reserves in soils are commonly large, although the distribution of K forms differs from soil to soil as a function of the dominant soil minerals (Sparks and Huang, 1985). Soil K is generally divided into four forms: water-soluble, exchangeable, and non-exchangeable, which includes fixed or mineral K forms. There are dynamic and equilibrium reactions between different forms of soil K. These reactions control an availability of soil K for plant growth and can describes whether K is taken by plants, leached into lower soil horizons, or converted into unavailable phases (Sparks, 1987).

Among the forms, the rate of non-exchangeable K⁺ release from soils can significantly influence K⁺ fertility of soils (Jalali and Zarabi, 2006).

Water-soluble K is taken up directly by plants but is usually found in low quantities in soils. Exchangeable K is held by negative charges of soil colloids (both soil organic matter and clay particles) at planar and edge position and is readily available to plants. Non-exchangeable K is fixed in interlayer space (inert position) of 2:1 type of clay mineral and is in structural of K bearing minerals. The last form of K is not readily but very slowly available to plants (Jalali and Zarabi, 2006). The quantity of K forms in the soil can be predicted by water, 1 N NH₄OAc pH 7, and HNO₃-HClO₄ extraction for water-soluble, exchangeable, and total K, respectively.

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The K forms are a function of the amount and type of dominant K mineral present in a soil and governs plant uptake of K at low exchangeable levels. Soils that are high in kaolinite (1:1 type of clay mineral), quartz, and other siliceous minerals contain little or no exchangeable and non-exchangeable K, whereas soil containing vermiculite, smectite (2:1 type of clay mineral), micas, and feldspars can have large amount of acid-extractable K (Martin and Sparks, 1983).

Soil dominated by 2:1 (vermiculite and smectite) types of clay mineral have generally higher in K adsorption and release than other type of clay mineral, such as 2:1:1, 1:1, oxide/hydroxide, and amorphous types. Among 2:1 types, the beidellite has the highest fixation capacity on K. Research conducted at smectitic soils (Vertisols) in India showed that beidellite has higher fixation on K than montmorillonite, mica, illite, and vermiculite (Murthy *et al.*, 1987). Furthermore, the release of K from micaceous minerals is in order of phlogopite > biotite > muscovite (Singh and Pasricha, 1987).

Smectitic soils have high prospect to be developed to become agricultural land unless with proper soil and plant management. These soils have generally vertic characteristics and properties, such as deep cracks when soil are dry, intersecting slickenside, and wedge-shaped structural aggregates in the subsoil, surface mulching, and clay texture. The soils with those characteristics include Vertisols and some Inceptisols as well as Alfisols. In Indonesia, the three types of soils cover more than 2.12 million ha area (Vertisols 2.12 million ha plus a part of Inceptisols and Alfisols) distributed in Java (West, Central, and East Java provinces), West Nusa Tenggara province, and Sulawesi (South and Central Sulawesi as well as Gorontalo provinces) (Pusat Penelitian Tanah and Agroklimat, 2000).

Although total K content in the soils (potential K) is commonly high, the availability for plant growth (actual K), however, is frequently problem because it is fixed by 2:1 type of clay mineral, such as smectites (Borchardt, 1989) and vermiculites (Douglas, 1989) that are dominant in the soils. Ghousikar and Kendre (1987) reported that Vertisols in India have high K-fixing capacity and soil buffering capacity on K so that leaves of plant showed deficiency of K symptoms. Thus, several efforts are needed to solve this problem in the soils to increase the availability of soil K for plant growth.

Oxalic acid held an important role in releasing fixed K to become available K at soils that contain K bearing minerals (Song and Huang, 1988). The acid was the most dominant organic acid excreted by roots maize that was about 3.15-5.93 mg g⁻¹ dry weight (Nursyamsi, 2008). Besides oxalic acid, several cations are able also to liberate fixed K at soils dominated by 2:1 types of clay mineral. The cations were Na⁺ (Ismail, 1997), NH₄⁺ (Kilic *et al.*, 1999; Evangelou and Lumbanraja, 2002), and Fe³⁺ (Nursyamsi *et al.*, 2008). The last cation was able to liberate fixed K because according to lyotropic series, it has higher adsorption than K⁺ (Havlin *et al.*, 1999). Besides that, Fe³⁺ is also a micronutrient that frequently become a limiting factor in alkaline soils includes smectitic soils (Marschner, 1997). The rank of adsorption, buffering capacity, and maximum adsorption of cations in the soils was in order of Fe³⁺ > NH₄⁺ = Na⁺ (Nursyamsi, 2008). In kaolinitic soils such as in Inceptisols of rice field land in Bandar Lampung, soil tillage and K fertilizers were found out as the factors that effected on available soil K (Isnaini, 2004).

Considering the above mentions, this research aimed to study effect of oxalic acid, Na⁺, NH₄⁺, and Fe³⁺ on release of fixed K by clay mineral and basal distance of smectite in smectitic soils.

MATERIALS AND METHODS

Experiment Setup

Laboratory experiment was conducted in Laboratory of Soil Sciences, Graduate School of Agriculture, Kyoto University, Japan by using topsoil samples that represent the smectitic soils. The samples were taken from Jonggol (Bogor), Sidareja (Cilacap), Padas (Ngawi), as well as Todanan (Blora), and classified as Typic Hapludalfs (B1), Chromic Endoaquerts (B2), Typic Endoaquerts (B3), and Typic Hapludalfs (B4), respectively. The initial analysis result of all tested soils was showed at Table 1.

The experiment used randomized completely block design, six treatments, and three replications. The treatments were control (water), 1 N oxalic acid, (oxalic acid+NaOH) pH=7, Na⁺ from NaCl, NH₄⁺ from NH₄Cl, and Fe³⁺ from FeCl₃. Then, the experiment was conducted with four steps: (1) Separation of clay fraction, (2) Saturation of soil clay samples with 1 N K⁺ from KCl, (3) Extraction, and

(4) Measurement of concentration of K in supernatant and basal distance of smectite in clay paste.

Separation of Clay Fraction

Separation of soil primer particles (sand, silt, and clay) was conducted by eliminating the soil cementing agent. Elimination of carbonate materials was done by adding HCl pH=5, whereas organic matter with 25% H₂O₂. Liberated soil sample from cementing agent was dispersed, then hard particles were separated by using 50 µm sieve. Separation of clay from silt fraction was done according to Stokes method. Soil suspension was allowed to stand overnight and top part of the suspension was then taken out and put into centrifuge tube. The samples were centrifuged with centrifuge at 3000 rpm during 30 minutes, then, water was discarded to separate clay fraction.

Saturation with K

The clays were saturated with K and to ensure that part of the added K was fixed (Tan, 1978). The clay suspensions were shaken with excess KCl

solutions for 6 h and the mixtures were subjected to two cycles of drying and wetting treatments. Drying was conducted at 45°C in a forced draft oven for 24 h each time. Wetting was done by adding sufficient amounts of deionized water to make a paste of the dried clay. The samples treated above were then washed by shaking with 1 N Na-acetate solutions to remove excess and exchangeable K, after which they were centrifuged with the Sorvall Superspeed centrifuge at 15,000 rpm, washed thoroughly 4 times with 98% ethyl-alcohol, dried at 45°C, ground, and labeled as K-smectite. The treatment above was known as illitization.

Extraction

One hundred mg saturated K-clay was weighed each time and added 20 mL of the treatment solutions. A blank was included using 100 mg K-clay and 20 mL deionized water. The mixtures were shaken for 6 h and allowed to stand overnight, after which they were centrifuged at 15,000 rpm. The supernatant were collected by filtration into 50 mL volumetric flasks. The clay residues were washed three times with

Table 1. Initial analyses of tested topsoil samples (0-20 cm).

Soil properties	Method	Typic Hapludalfs	Chromic Endoaquerts	Typic Endoaquerts	Typic Haplustalfs
Texture	Pipet				
Sand (%)		32	5	6	48
Silt (%)		26	33	22	20
Clay (%)		52	62	72	32
pH	H ₂ O (1:2.5)	5.52	6.33	6.06	7.09
	KCl 1 N (1:2.5)	3.89	4.67	4.48	5.89
Organic matter					
Organic-C (g kg ⁻¹)	Kurmies	8.9	10.6	7.4	8.0
Total-N (g kg ⁻¹)	Kjedahl	0.9	0.9	0.9	0.7
C/N		10	11	8	12
Potential P and K	HCl 25%				
P ₂ O ₅ (mg kg ⁻¹)		720	3810	570	1140
K ₂ O (mg kg ⁻¹)		300	1720	260	700
Available P (mg kg ⁻¹)	Bray 1	0.33	5.28	4.06	2.19
Cation exch.	NH ₄ OAc 1 N pH 7				
Ca _{exch} (cmol (+) kg ⁻¹)		11.53	33.72	53.72	11.03
Mg _{exch} (cmol (+) kg ⁻¹)		1.44	10.68	10.85	0.62
K _{exch} (cmol (+) kg ⁻¹)		0.08	0.38	0.11	0.11
Na _{exch} (cmol (+) kg ⁻¹)		0.08	0.69	0.08	0.14
CEC (cmol (+) kg ⁻¹)	NH ₄ OAc 1 N pH 7	30.03	47.03	43.92	10.13
BS (%)		49	95	100	100
Acidity	KCl 1 N				
Al _{exch} (cmol(+) kg ⁻¹)		6.88	0.00	0.00	0.00
H _{exch} (cmol(+) kg ⁻¹)		0.93	0.40	0.50	0.17

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deionized water and the washings were added to the extracts in the 50 mL volumetric flasks. The volumes were made up to 50 mL with deionized water, while clay residues were then sampled, mounted on glass slide, and airdried at room temperature.

Measurement

One mL supernatant was pipetted, put in 20 mL glass tube, diluted 10 times, and then homogenized with mixer. After standard solutions were prepared, amount of K in supernatant was measured by Atomic Absorption Spectrophotometer (AAS) method. Basal distance of smectite was measured by X-Ray Diffraction (XRD) method using Cu lamp at 4-30° angle after air drying clay pastes.

RESULT AND DISCUSSION

Release of fixed K

Effect of oxalic acid, Na⁺, NH₄⁺, and Fe³⁺ on release of fixed K at Alfisols and Vertisols were showed in Table 2 and Table 3, respectively. All treatments significantly increased the release of fixed K in both soils. Rank of the release of fixed K was in order of (oxalic acid+NaOH) pH = 7 > Fe³⁺ > NH₄⁺ > oxalic acid > Na⁺ > water at Typic Hapludalfs, while at Typic Haplustalfs was in order of (oxalic acid+NaOH) pH = 7 > Na⁺ > Fe³⁺ > NH₄⁺ > oxalic acid > water (Table 2). The rank at Chromic Endoaquerts was in order of Na⁺ > oxalic acid > (oxalic acid+NaOH) pH = 7 > NH₄⁺ > Fe³⁺ > water, whereas at Typic Endoaquerts was in order of Na⁺ > NH₄⁺ > oxalic acid > Fe³⁺ > (oxalic acid+NaOH) pH = 7 > water (Table 3). Among the tested treatments,

it seems that (oxalic acid+NaOH) pH=7 was the most effective in releasing fixed K at Alfisols, while Na⁺ at Vertisols.

It was assumed that total K content in the soil was equal with K extracted with strong acid of HNO₃+HClO₄. Thus, the total K content in each soil was 13.2, 6.2, 24.4, and 26.4 mg K g⁻¹ for Typic Hapludalfs, Typic Haplustalfs, Chromic Endoaquerts, and Typic Endoaquerts, respectively. Percentage of K release in Alfisols as a result of the treatments was 1.37-36.93% and 1.86-45.38% at Typic Hapludalfs and Typic Haplustalfs, respectively (Table 2), while in Vertisols, it was 2.37-34.08% and 3.90-48.35% at Chromic Endoaquerts and Typic Endoaquerts, respectively (Table 3). Among the treatments at all tested soils, the percentage of K release was 24.63-3744%, 31.88-45.38%, 25.37-48.35%, 27.48-42.32%, and 28.17-35.49% for use of oxalic acid, oxalic acid+NaOH pH=7, Na⁺, NH₄⁺, and Fe³⁺, respectively. The rank of the percentage of K release indicated amount of K that can be released by the treatments in both Alfisols and Vertisols.

Oxalic acid and other organic compounds excreted by roots were able to increase available K for plant growth in smectitic soils. Organic acid exudates concentrations in rhizosphere were very high. It was indicated with soil pH in rhizosphere (4.8) that was lower than the pH in bulk soil (7.5) (Marschner, 1997). Organic compound exudates were energy source for microorganisms life so that it was possible for microbes to increase their activities in rhizosphere. Consequently, biochemistry process in this area was also very fast. Besides that, the activity of microbes was able to produce excretion of many

Table 2. Effect of oxalic acid, Na⁺, NH₄⁺, and Fe³⁺ on release of fixed K at Alfisols.

Treatment	Typic Hapludalfs (B1)		Typic Haplustalfs (B4)	
	mg K g ⁻¹	Percentage (%)	mg K g ⁻¹	Percentage (%)
Control (water)	0.18 d	1.37	0.11 e	1.86
Oxalic acid	3.58 b	27.13	1.53 cd	24.63
(Oxalic acid+NaOH) pH=7	4.87 a	36.93	2.81 a	45.38
Na ⁺	3.35 b	25.37	2.42 ab	39.06
NH ₄ ⁺	3.63 b	27.48	1.89 bc	30.55
Fe ³⁺	3.72 b	28.17	1.93 bc	31.20
CV (%)	8.9		10.4	

Means with different letters indicated significantly different at 5% level according to Duncan's Multiple Range Test.

Table 3. Effect of oxalic acid, Na⁺, NH₄⁺, and Fe³⁺ on release of fixed K at Vertisols.

Treatment	Chromic Endoaquerts (B2)		Typic Endoaquerts (B3)	
	mg K g ⁻¹	Percentage (%)	mg K g ⁻¹	Percentage (%)
Control (water)	0.58 c	2.37	1.03 d	3.90
Oxalic acid	7.88 a	32.28	9.89 bc	37.44
(Oxalic acid+NaOH) pH=7	7.78 a	31.88	8.96 c	33.94
Na ⁺	8.32 a	34.08	12.77 a	48.35
NH ₄ ⁺	7.57 a	31.02	11.17 b	42.32
Fe ³⁺	7.54 a	30.91	9.37 c	35.49
CV (%)	11.7		9.4	9.4

Means with different letters indicated significantly different at 5% level according to Duncan's Multiple Range Test.

kind of organic compounds including oxalic acid (Bolton *et al.*, 1993).

This experiment indicated that Na⁺ was able to increase the availability of soil K in the soils. In Vertisols particularly, it was the most effective in releasing fixed K (Table 3). Besides that, Na⁺ was a beneficial mineral element needed by some plant species, such as rice, sugarcane, etc. In addition, it can substitute a part of plant K requirement because Na can substitute a part of K function as a catalyst in plant metabolic system (Marschner, 1997).

In term of fixation in interlayer space of 2:1 type of clay mineral, characteristic of NH₄⁺ was similar with K⁺ so that the cation was able to replace fixed K and vice versa. Research conducted by Kilic *et al.* (1999) at smectitic soils and Evangelou and Lumbanraja (2002) at soils dominated by vermiculite and hydroxyl interlayer vermiculite showed that fixed NH₄⁺ at interlayer space was able to be replaced by K⁺ and vice versa. Besides that, NH₄⁺ was also one of macronutrients (N) needed by plants for their growth and development (Havlin *et al.*, 1999). The nutrient form commonly appears in the soils of lowland rice because of anaerobic condition.

In soil system, Fe³⁺ is always covered by water molecules so that it has a function to increase soil water retention. Its adsorption in soil colloid is higher than K because its valence is III while K valence is I. The cation was not only able to increase available K in the soils but also as micronutrient needed by high plants. The nutrient plays an important rule in biosynthesis of heme coenzymes and chlorophyll. Furthermore, Fe has also a rule in chloroplast development and photosynthesis (Marschner, 1997). Iron is required for protein synthesis, thus in iron-

deficient maize leaves, for examples, total protein content decreases by about 25% and that of the chloroplast by about 82%, most probably because of a particularly high iron requirement of chloroplastic mRNA and rRNA (Spiller *et al.*, 1987).

Basal Distance of Smectite

Basal distance of smectite at Alfisols and Vertisols as a result of the treatments was showed in Table 4, while diffractogram of clay fraction of the soils was showed in Figure 1 and Figure 2 for Alfisols and Vertisols, respectively. The Table 4 indicated that basal distance of smectite didn't change with oxalic acid, decreased slightly with NH₄⁺, and increased significantly with (oxalic acid+NaOH) pH=7, Na⁺, and Fe³⁺ in all tested soils. In average of the soils, the basal distance increased about 10.41%, 11.48%, and 15.30% with (oxalic acid+NaOH) pH=7, Na⁺, and Fe³⁺ treatments, respectively. The increase of basal distance of smectite was the highest as a result of Fe³⁺ treatment which increased from 13.00 to 15.95 Å (22.69%), 13.81 to 14.88 Å (7.75%), 12.71 to 16.07 Å (26.44%), and 12.74 to 16.07 Å (26.13%) at Typic Hapludalfs, Typic Haplustalfs, Chromic Endoaquerts, and Typic Endoaquerts, respectively (Figure 1 and Figure 2).

Increase of basal distance of smectite by using (oxalic acid+NaOH) pH=7 showed that the ion was able to penetrate into interlayer space of smectite and then liberate same fixed K (Table 2 and Table 3). Tan (1978) reported that organic acids (humic and fulvic acids) were able to liberate about 25% fixed K and increase basal distance of 2:1 type of clay mineral at montmorillonitic and illitic soils. The basal distance

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Table 4. Effect of oxalic acid, Na⁺, NH₄⁺, and Fe³⁺ on basal distance of smectite at both Alfisols and Vertisols.

Treatment	Alfisols	Vertisols	Alfisols+Vertisols
 Å		
Control (water)	13.41 cd	12.73 b	13.07 c
Oxalic acid	13.41 cd	12.73 b	13.07 c
(Oxalic acid+NaOH) pH=7	14.05 b	14.81 a	14.43 b
Na ⁺	14.34 b	14.81 a	14.57 ab
NH ₄ ⁺	12.84 d	12.46 b	12.65 c
Fe ³⁺	15.42 a	14.73 a	15.07 a
CV (%)	2.6	1.4	3.3

Means with different letters indicated significantly different at 5% level according to Duncan's Multiple Range Test.

increased from 11 to 11.9 Å and 11 to 12.3 Å by using humic and fulvic acids, respectively.

Use of NH₄⁺ decreased basal distance of smectite at all tested soils. As mentioned earlier that the cation had characteristics that was similar to K⁺. Saturation of smectite with K⁺ caused the basal distance of smectite decreased significantly from about 15 to 12 Å at the soils (Nursyamsi, 2008). The probability explanation was because hydrated ionic radius of the cation was low (5.3 Å). Hydrated ionic radius of NH₄⁺ was nearly equal to that of K⁺ (5.6 Å) so that it caused its characteristic was similar to K⁺ (Havlin *et al.*, 1999). Although NH₄⁺ decreased basal distance of smectite, it increased release of fixed K in the soils (Table 2 and Table 3). It was related to ability of NH₄⁺ to exchange K⁺ in interlayer space of 2:1 type of clay mineral (Kilic *et al.*, 1999; Evangelou and Lumbanraja, 2002).

Different with NH₄⁺, other cations (Na⁺ and Fe³⁺) increased basal distance of smectite in the soils. Hydrated ionic radius of Na⁺ (7.9 Å) was higher than that of K⁺ (Havlin *et al.*, 1999), so that it was able to increase the basal distance of smectite. Because of covered with water molecules in soil system, the hydrated ionic radius of Fe³⁺ was quite high about 9.0 Å (Tan, 1998). Consequently, the basal distance of smectite increased drastically with Fe³⁺ treatment. Penetration of Fe³⁺ into interlayer space of smectite caused basal distance increased, then the cation exchanged fixed K to become release and available for plant growth (Table 2 and Table 3).

Among the treatments tested, Fe³⁺ was the highest in increasing basal distance of smectite in all tested soils. It was because hydrated ionic radius of Fe³⁺ was higher than that of Na⁺, NH₄⁺, and K⁺. In

contrary, amount of K liberated with Fe³⁺ was lower than with other cations. Ability of cation to exchange fixed K depends on amount in solution (molarity) and valence of the cation (Tan, 1998). This experiment used the treatments which were same normality (1 N). Since valence of FeCl₃ (aq), NaCl (aq), and NH₄Cl (aq) was 3, 1, and 1 respectively, thus the molarity of Fe³⁺ (0.33 M) was lower than that of Na⁺ (1 M), and NH₄⁺ (1 M). Consequently, although Fe³⁺ increased basal distance of smectite higher than both Na⁺ and NH₄⁺ but its ability to exchange fixed K in interlayer space of smectite was the lowest.

CONCLUSION

Oxalic acid, (oxalic acid+NaOH) pH=7, Na⁺, NH₄⁺, and Fe³⁺ increased the release of fixed potassium by clay in all tested soils about 24.63-37.44%, 31.88-45.38%, 25.37-48.35%, 27.48-42.32%, and 28.17-35.49%, respectively. Among the treatments, (oxalic acid+NaOH) pH=7 was the most effective in releasing fixed K at Alfisols, while Na⁺ at Vertisols.

Oxalic acid + NaOH pH = 7, Na⁺, and Fe³⁺ increased the basal distance of smectite in the soils about 10.41%, 11.48%, and 15.30%, respectively. Among the treatments, Fe³⁺ was the highest in increasing the basal distance of smectite in the soils.

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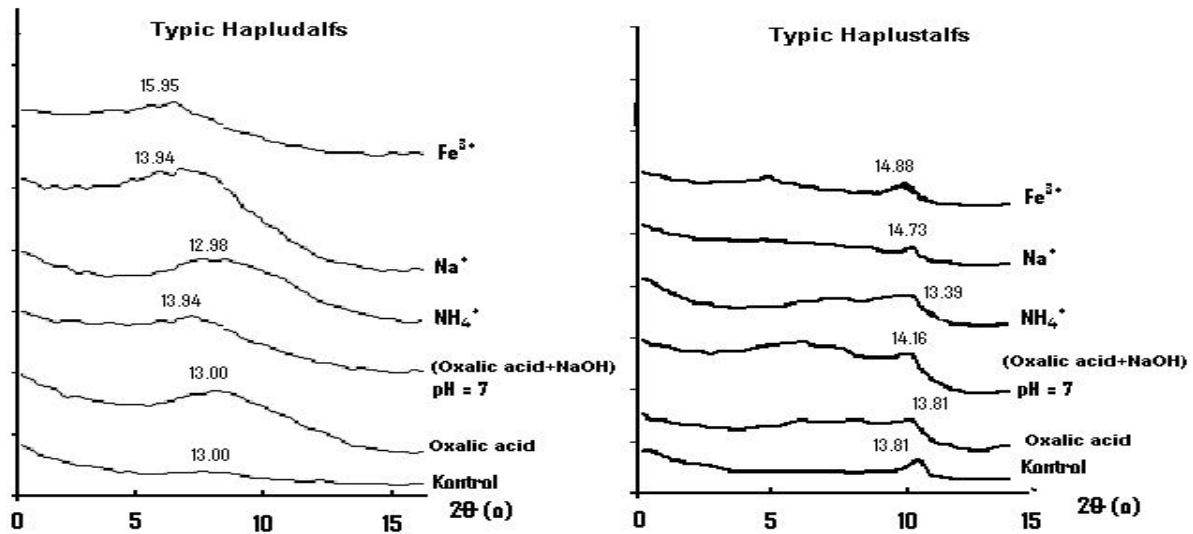


Figure 1. Effect of oxalic acid, Na^+ , NH_4^+ , and Fe^{3+} on basal distance of smectite at Alfisols.

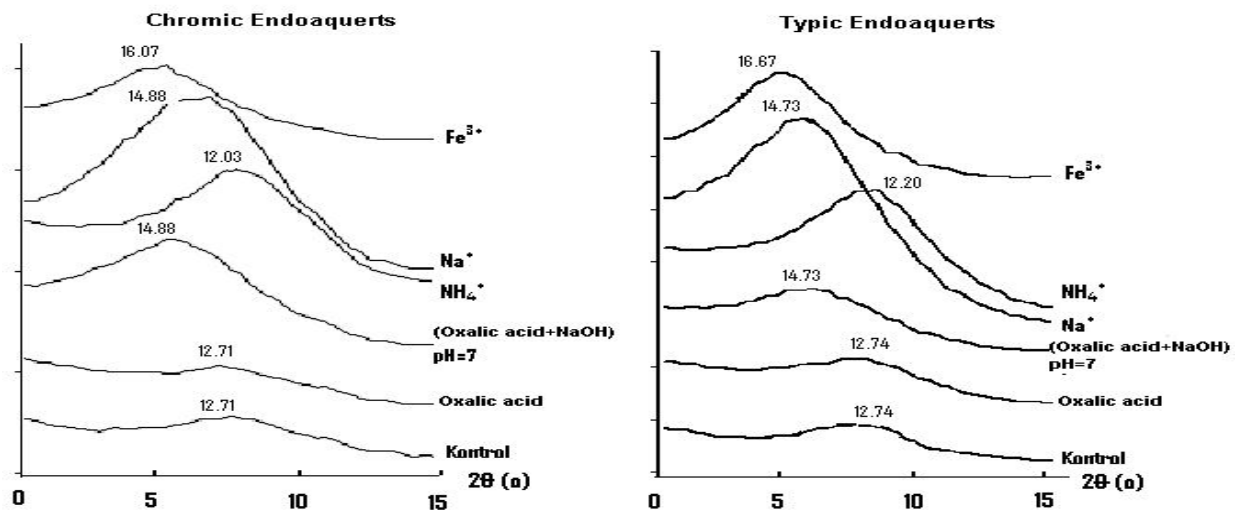


Figure 2. Effect of oxalic acid, Na^+ , NH_4^+ , and Fe^{3+} on basal distance of smectite at Vertisols.

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