RESEARCH NOTE

Number of extractions in determination of effective cation-exchange capacity

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> **Abstract.** The number of successive extractions with 1 M KCl needed for adequate estimation of effective cation-exchange capacity was studied with four mineral soils. The effective CEC estimated as the sum of equivalents of exchangeable Ca, Mg, Na, H and Al extracted by four successive treatments ranged from 57 to 206 meq/kg soil. In three cultivated soils, 63—90 % of CEC was saturated by Ca and Mg, in the fourth soil (a deeper layer virgin soil), 60 % of CEC by exchangeable H and Al.

> By two successive treatments of ten minutes duration with 50 ml of 1 M KCl, the equivalent sum of exchangeable cations extracted amounted to 83-92 % of effective CEC in cultivated soils and 67 % of that in virgin soil; >90 % of exchangeable Ca and Mg, 78-97 % of Al, 48-62 % of H and 28-64 % of Na were extracted. By three successive treatments the equivalent sum amounted to 79-96 % of effective CEC, by the single treatment of 30 minutes duration with 100 ml of 1 M KCl to 57-79 %.

Two successive extractions with 1 M KCl may be enough for estimation of effective CEC in cultivated mineral soils with high degree of saturation by exchangeable Ca and Mg. Soils with high degree of saturation by exchangeable acidity require three successive extractions.

Index words: effective cation-exchange capacity, exchangeable Ca, Mg, Na, H and Al

Introduction

In determination of exchangeable cations and effective cation-exchange capacity of soil, exchangeable cations are commonly extracted by successive treatments with unbuffered neutral salt solutions like 1 M KCl (e.g. YUAN 1959, KAILA 1971 a, 1971 b, 1971 c, NISKANEN and JAAKKOLA 1986). The normal practice, four or five successive extractions, presupposes a laborious multiphase shaking and centrifugation procedure. A routine method with fewer successive extractions would facilitate analytical work. The purpose of this study was to investigate the possibility of reducing the number of successive extractions with 1 M KCl needed for adequate estimation of effective CEC in mineral soils.

Material and methods

The material consisted of four mineral soil samples from the Viikki Experimental Farm, University of Helsinki: three (No. 2—4) from plough layer (0—20 cm) of cultivated soils and one (No. 1) from deeper layer (20—40 cm) of virgin soil (Table 1). The samples were airdried and ground to pass a 2-mm sieve. The particle-size distribution of the inorganic matter of soil was determined by the pipette method (ELONEN 1971). Soil pH was measured in soil-0.01 M CaCl₂ suspension (1:2.5 v/v) (Ryti 1965). The organic carbon content was determined by a modified (GRAHAM 1948) ALTEN's wet combustion method.

Exchangeable cations were extracted from 10 g of soil by four successive treatments with 50 ml of 1 M KCl, shaking time 10 minutes. Uncombined extracts were analyzed for exchangeable Ca, Mg, Na, H and Al. Exchangeable cations were extracted from 10 g of soil

Table 1. Soil characteristics.

Soil sample	Sampling depth, cm	pH(CaCl ₂)	Org. C, %	Particle-size distribution, %					
				<2 µm	2—20 µm	20—60 µm	60—200 μm	>200 µm	
1	20-40	4.1	2.7	2	5	10	28	55	
2	0-20	5.3	4.4	10	7	15	61	7	
3	0-20	4.6	3.3	37	13	15	30	4	
4	0-20	4.5	3.4	43	33	18	3	2	

Table 2. Exchangeable cations, meq/kg soil (% of total), extracted with 1 M KCl by four successive treatments and by a single treatment.

	Ca	Mg	Na	Н	Al	Total	
	Soil 1						
1st treatment	5.6 (68)	2.1 (62)	2.2 (20)	1.4 (18)	15.4 (61)	26.7 (48)	
2nd »	1.9 (23)	1.0 (30)	0.9 (8)	2.3 (30)	4.3 (17)	10.4 (19)	
3rd »	0.7 (8)	0.3 (8)	0.9 (8)	1.6 (21)	3.6 (14)	7.1 (13)	
4th »	0.1 (1)	0.0 (0)	7.0 (64)	2.3 (30)	2.1 (8)	11.5 (21)	
Total	8.3	3.4	11.0	7.6	25.4	55.7	
Single treatment	6.2 (75)	2.1 (62)	1.7 (16)	1.7 (22)	19.8 (78)	31.5 (57)	
	Soil 2						
1st treatment	95.8 (78)	27.1 (90)	4.8 (38)	1.8 (31)	0.0	129.5 (75)	
2nd »	25.2 (20)	1.2 (4)	0.9 (7)	1.8 (31)	0.0	29.1 (17)	
3rd »	1.1 (1)	1.8 (6)	1.3 (10)	1.2 (20)	0.0	5.4 (3)	
4th »	1.2 (1)	0.1 (0)	5.7 (45)	1.1 (19)	0.0	8.1 (5)	
Total	123.3	30.2	12.7	5.9	0.0	172.1	
Single treatment	102.3 (83)	26.7 (89)	5.2 (41)	2.3 (39)	0.0	136.5 (79)	
	Soil 3						
1st treatment	39.4 (83)	29.9 (83)	16.5 (47)	2.4 (24)	3.3 (97)	91.5 (69)	
2nd »	5.9 (12)	3.7 (10)	6.1 (17)	2.7 (27)	0.1 (3)	18.5 (14)	
3rd »	2.1 (4)	2.1 (6)	3.0 (9)	3.0 (30)	0.0 (0)	10.2 (8)	
4th »	0.1 (0)	0.3 (0)	9.6 (27)	2.0 (20)	0.0 (0)	12.0 (9)	
Total	47.5	36.0	35.2	10.1	3.4	132.2	
Single treatment	42.1 (89)	29.9 (83)	17.4 (49)	3.8 (38)	3.3 (97)	96.5 (73)	
	Soil 4						
1st treatment	127.2 (92)	26.2 (82)	5.7 (41)	0.8 (13)	8.8 (57)	168.7 (82)	
2nd »	8.8 (6)	4.4 (14)	1.7 (12)	2.4 (38)	3.3 (22)	20.6 (10)	
3rd »	2.4 (2)	0.9 (3)	0.9 (6)	1.7 (27)	2.1 (14)	8.0 (4)	
4th »	0.2 (0)	0.3 (1)	5.7 (41)	1.5 (23)	1.1 (7)	8.8 (4)	
Total	138.6	31.8	14.0	6.4	15.3	206.1	
Single treatment	113.2 (82)	25.4 (80)	6.5 (47)	1.8 (28)	12.6 (79)	159.5 (77)	

also by a single treatment with 100 ml of 1 M KCl, shaking time 30 minutes. Exchangeable Ca and Mg were determined by atomic absorption spectrophotometry, Na by flame photometry. Exchangeable H was determined by titrating an aliquot of extract with 0.01 M NaOH (phenolphtalein as indicator) until a red colour persisted; thereafter Al was determined by titrating with 0.01 M HCl (10 ml of 4 % NaF per 50 ml extract was added) until the solution was colourless (YUAN 1959). The experiment was carried out in duplicate.

Results and discussion

The effective cation-exchange capacity estimated as the sum of exchangeable cations extracted by four successive treatments of 1 M KCl was lowest in soil No. 1 with low pH and clay content and highest in soil No. 4 with the highest clay content (Table 2). The exchangeable cation composition of the virgin soil No. 1 included more H and Al than the other soils; 60 % of the estimated effective CEC of soil No. 1 was saturated with these acid cations (Table 3). The proportion of acid cations was lowest in soil No. 2 the pH of which was 5.3. No exchangeable Al was found in this soil. This was in agreement with the observation of KAI-LA (1971 a) that unbuffered 1 M KCl replaces Al only in samples with a pH(CaCl₂) below 5.3. In the other two soils with pH 4.5 and 4.6 the proportion of H and Al was about 10 % (Table 3).

The degree of saturation by exchangeable Ca and Mg in soils No. 2 and 4 was of the same order as in the material of KAILA (1972): 60—80 % and 10—30 % for Ca and Mg, respectively (Table 3). The degree of saturation by exchangeable Ca in soils No. 1

Table 3. Exchangeable cations as % of effective CEC.

Soil	Ca	Mg	Na	Н	Al
1	15	6	20	14	46
2	72	18	7	3	0
3	36	27	27	8	3
4	67	15	7	3	7

and 3 was much lower than the mean value of corresponding soil groups in the material of KAILA (1972). The content of exchangeable Na in experimental soils (Table 2) of old sea bottom was much above average values reported by KAILA (1972). In soils 1 and 3 the proportion of Na was considerable, Na being the dominant basic cation in soil 1 (Table 3).

The first 10-minute treatment with 1 M KCl extracted most of the total exchangeable Ca and Mg (Table 2), about 80 % or more was extracted in soils 2—4. The percentage was lower, <70 %, in soil 1 with low content of exchangeable Ca and Mg. As compared with the first 10-minute treatment, the single 30-minute treatment with double quantity of extractant extracted a little more Ca from soils 1—3 and less Ca from clay soil 4, while about equal amounts of exchangeable Mg were extracted by both treatments.

The second 10-minute treatment extracted 6-23 % of exchangeable Ca and 4-30 % of exchangeable Mg (Table 2). Thus, the first two successive 10-minute treatments extracted totally >90 % of exchangeable Ca and Mg of the experimental soils. Compared to the single treatment with 100 ml of 1 M KCl the first two successive treatments with two 50 ml portions of 1 M KCl extracted 6-16 % and 5-30 % more of total exchangeable Ca and Mg, respectively, than the single treatment.

The extraction pattern of trivalent Al was rather similar to that of divalent Ca and Mg. The first treatment extracted nearly all the exchangeable Al from soil 3 with low content of exchangeable Al and about 60 % from soils 1 and 4 (Table 2). The first two successive treatments with 50 ml of 1 M KCl extracted about 80 % of the exchangeable Al of these soils; the same amount was extracted by single treatment with 100 ml of 1 M KCl (Table 2).

The extraction pattern of monovalent cations deviated from that of di- and trivalent cations. Most of Ca, Mg and Al was extracted by the first two treatments, while considerable quantities of exchangeable Na and H were extracted by further treatments. The first treatment extracted only 20 % of the exchangeable Na in soil 1 and 38—47 % of that in the other soils (Table 2). The single treatment with 100 ml of KCl extracted slightly more Na from soils 2—4. An appreciable proportion (27— 64 %) of exchangeable Na was still extracted by the fourth treatment. Extraction of exchangeable H continued evenly throughout the four successive treatments (Table 2). The single treatment with 100 ml of 1 M KCl extracted more H than the first treatment with 50 ml of 1 M KCl.

In soils 2 and 4 with high degree of saturation by exchangeable Ca and Mg, the equivalent sum of cations extracted by the first two successive treatments amounted to 92 % of effective CEC (Table 2), in soils 1 and 3 with high proportion of Na to 67 and 83 % and by the single treatment to >70 % in soils 2–4 (Table 2). By three successive treatments the equivalent sum amounted to >90 % in soil 3 and to about 80 % in soil 1.

In Finnish cultivated mineral soils the effective CEC is overwhelmingly saturated with exchangeable Ca and Mg (KAILA 1972, NISKANEN and JAAKKOLA 1986). Thus two treatments with 1 M KCl may be enough for estimation of effective CEC. Virgin soils with high degree of saturation by exchangeable acidity require more treatments. An adequate result can be achieved with three successive treatments as recommended by BOWER et al. (1952) for extraction with neutral ammonium acetate.

References

- BOWER, C.A., REITEMEIER, R.F. & FIREMAN, M. 1952. Exchangeable cation analysis of saline and alkali soils. Soil Sci. 73: 251-261.
- ELONEN, P. 1971. Particle-size analysis of soil. Acta Agr. Fenn. 122: 1–122.
- GRAHAM, E. 1948. Determination of soil organic matter by means of a photoelectric colorimeter. Soil Sci. 65: 181–183.
- KAILA, A. 1971 a. Aluminium and acidity in Finnish soils. J. Scient. Agric. Soc. Finl. 43: 11–19.
- 1971 b. Effective cation-exchange capacity in Finnish mineral soils. J. Scient. Agric. Soc. Finl. 43: 178—186.
- 1971 c. Über den Anteil organischer Substanz an der Austauschkapazität von Mineralböden in Finnland.

Spomenica uz 70. god. prof. Gračanina, Zagreb, p. 53—56.

- 1972. Basic exchangeable cations in Finnish mineral soils. J. Scient. Agric. Soc. Finl. 44: 164–170.
- NISKANEN, R. & JAAKKOLA, A. 1986. Estimation of cation-exchange capacity in routine soil testing. J. Agric. Sci. Finl. 58: 1—7.
- RYTI, R. 1965. On the determination of soil pH. J. Scient. Agric. Soc. Finl. 37: 51-60.
- YUAN, T.L. 1959. Determination of exchangeable hydrogen in soils by a titration method. Soil Sci. 88: 164–167.

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SELOSTUS

Uuttokertojen lukumäärä efektiivisen kationinvaihtokapasiteetin määrityksessä

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Efektiivisen kationinvaihtokapasiteetin tyydyttävään määrittämiseen tarvittavien peräkkäisten 1 M KCluuttojen lukumäärää tutkittiin neljällä kivennäismaalla. Neljässä peräkkäisessä käsittelyssä uuttuneiden vaihtuvien kationien (Ca, Mg, Na, H, Al) ekvivalenttisummana määritetty efektiivinen kationinvaihtokapasiteetti oli 57—206 meq/kg maata. Kolmessa viljelysmaata edustavassa näytteessä 63—90 % kationinvaihtokapasiteetista oli vaihtuvan Ca:n ja Mg:n kyllästämää ja luonnontilaista pohjamaata edustavassa näytteessä 60 % kationinvaihtokapasiteetista oli vaihtuvan H:n ja Al:n kyllästämää.

Kahdella peräkkäisellä käsittelyllä (50 ml 1 M KCl, 10 min.) uuttuneiden vaihtuvien kationien ekvivalenttisumma oli viljelysmaissa 83—92 % ja luonnontilaisessa maassa 67 % efektiivisestä kationinvaihtokapasiteetista; >90 % vaihtuvasta kalsiumista ja magnesiumista, 78— 97 % aluminiumista, 48—62 % vedystä ja 28—64 % natriumista uuttui. Kolmella peräkkäisellä käsittelyllä uuttuneiden vaihtuvien kationien ekvivalenttisumma oli 79– 96 % efektiivisestä kationinvaihtokapasiteetista. Yhdellä puolen tunnin käsittelyllä (100 ml 1 M KCl) uuttuneiden vaihtuvien kationien ekvivalenttisumma oli 57–79 % efektiivisestä kationinvaihtokapasiteetista.

Kaksi peräkkäistä uuttoa 1 M KCl:lla saattaa olla riittävä efektiivisen kationinvaihtokapasiteetin määrittämiseen viljellyillä kivennäismailla, joissa vaihtuvan Ca:n ja Mg:n osuus on suuri. Jos vaihtuvan happamuuden osuus on suuri, tarvitaan ainakin kolme peräkkäistä uuttoa.