

Potassium analysis of soils before and after exhaustion of K

Kaliumanalyse af jord før og efter udpining for K

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Summary

Samples (19) from the top soil were exhausted of K by the cultivation of grass for 5 years. The soil analytical figures before and after exhaustion showed that the exchangeable K was used up within the first year. The following years only the content of acid soluble K, K_{HCl} and K_{HNO_3} , changed. In the course of the experiment the intensity of K in all soils decreased to the same level irrespective of type, but the capacity of K remained greatest in the clay soils. The solubility of both exchangeable and non-exchangeable K decreased after exhaustion, while the ability of the soil to fix K increased.

Key words: Potassium, soil.

Resumé

Jordprøver (19) fra pløjelaget blev udpint for K ved dyrkning af græs gennem 5 år. Jordanalysetallene før og efter udpiningen viste, at ombytteligt K var opbrugt i løbet af det første år. De følgende år ændredes kun indholdet af syreopløseligt K, K_{HCl} og K_{HNO_3} . I løbet af forsøgsperioden faldt intensiteten til det samme niveau i alle jorde uanset typen, medens kapaciteten af K forblev størst i lerjorde. Opløseligheden af såvel ombytteligt som ikke ombytteligt K blev mindre efter udpiningen, medens jordens fikseringsevne over for K øgedes.

Nøgleord: Kalium, jord.

Introduction

In the evaluation of the content of K in soil a distinction is normally made between exchangeable and non-exchangeable. The former is mainly adsorbed by the surface of clay and humic particles, while the latter is present partly as fixed and partly as elements in the structure of primary minerals e.g. feldspars and micas.

Only 1 to 5 per cent of the soil K is exchangeable but it is this part which is first used by the plants. Exchangeable K (Kt) is the index used in Denmark for evaluation of the K-state in soil, in spite of the fact that also Danish soils can release

non-exchangeable K. Field and pot experiments where the addition and removal of K is controlled, show that at least in the case of clay soils there is release and uptake of non-exchangeable K.

In some other countries non-exchangeable K is taken in consideration. This is the case in Norway and Sweden, where in addition to determination of exchangeable K, the soil samples are supplemented with an extraction of HNO_3 (Reitemeir, 1948) or HCl (Egnér, 1960), which causes a release of K 5 to 10 times greater than the exchangeable. Kolterman & Troug (1953) estimated the

content of non-exchangeable K in soil by ignition. The following methods are also stated to release a part of that K which is non-exchangeable: 1. Treatment with cation exchange resin (*Schmitz & Pratt, 1953*) 2. Extraction with kalignost (natriumtetraphenylborate), (*Conyers & McLean, 1969; Schulte & Corey, 1969*). The last 2 methods are rather complicated and not well suited to routine analysis.

Exhaustion of K in the soil changes the analytical data for K. Primarily the content of exchangeable K is changed but also the content of non-exchangeable K decrease due to exhaustion of the soil.

The purpose of the present investigation was to study the chemical changes in soil caused by its exhaustion of K. That included the amount and

solubility of both exchangeable and non-exchangeable K before and after the exhaustion of K in the soils. In addition the analytical error for the different soil analyses was evaluated.

Soils

In the investigation was used soil samples from the surface (0 to 20 cm) at 19 localities. Before the experiment the samples were analyzed for texture, hydrogen activity (Rt), P soluble with 0.2 n H₂SO₄ (Ft), electrical conductivity (Lt), exchangeable Na (Nat), Ca (Cat), Mg (Mgt) and exchange capacity. The results are shown in table 1 and the soils are arranged according to decreasing clay content. The soil analytical data for K is given in tables together with the results and the discussions.

Table 1. Analysis of soil before the experiment

Locality	texture					m equiv./100 g soil						
	clay	silt	fine sand	coarse sand	humic	Rt	Lt	Ft	Cat	Mgt	Nat	exch. capc.
Ribe	22.9	22.9	44.3	5.0	4.9	7.7	2.23	9.6	585	37.5	18.9	29.9
Silstrup	19.3	12.0	41.2	24.0	3.5	7.0	0.76	6.9	335	9.7	4.8	19.2
Abed	17.3	19.4	41.9	19.1	2.3	7.8	0.83	10.8	475	5.5	3.5	21.4
Højer	16.9	18.2	60.3	1.3	3.3	7.7	0.52	13.1	400	25.0	3.5	25.4
Blangstedg.	13.1	14.7	39.2	31.0	2.0	5.5	0.20	4.2	186	13.1	1.5	13.3
Askov ler	11.9	14.7	33.5	35.9	4.0	6.7	0.42	5.9	272	3.4	2.3	14.7
Ødum	11.8	19.0	47.0	19.7	2.5	6.8	0.46	9.2	255	5.9	2.3	14.9
Årslev	11.4	15.3	43.7	26.7	2.9	6.5	0.37	5.2	191	2.9	1.3	15.8
Roskilde	11.2	18.0	44.7	23.6	2.5	6.6	0.38	7.8	180	1.5	2.2	14.2
Tystofte	11.0	13.8	45.2	27.5	2.5	6.3	0.24	5.3	198	2.9	1.3	10.8
Virumgård	9.4	15.5	47.2	25.8	2.1	5.5	0.33	11.7	111	2.8	1.2	11.6
Spangsbjerg	6.8	11.4	46.6	32.7	2.5	5.5	0.31	15.4	100	4.3	1.6	11.7
Borris	5.5	10.5	53.0	28.1	2.9	5.9	0.30	6.0	120	3.2	1.3	9.9
Studsgård	5.3	5.8	41.1	43.8	4.0	5.7	0.18	2.4	139	1.9	1.5	13.3
Askov sand	4.9	5.4	33.3	54.4	2.0	6.7	0.23	6.5	139	1.9	1.8	5.9
Hornum	4.9	8.2	48.1	35.9	2.9	6.5	0.27	12.5	168	6.5	1.0	8.7
Lundgård	3.8	4.0	24.9	64.9	2.4	6.6	0.43	5.4	147	3.6	4.8	5.0
St. Jyndevad	3.7	3.5	17.0	73.0	2.8	6.6	0.47	2.3	168	6.5	1.3	7.4
Tylstrup	3.4	2.1	75.3	16.4	2.8	5.5	0.20	8.8	84	3.1	0.6	6.5

The execution of the experiment

For all the soils there were 2 treatments:

- Addition each year of 4 mg K per 100 g soil. K was added as K₂SO₄.
- No K-addition.

During the experiment all essential plant nutrients other than K were supplied in optimum. The soils were cultivated with perennial ryegrass in 2 l pots for 5 years. In the experiment the soils were mixed with sand 1:1 and the pots contained 3

kg of the soil/sand mixture. In the autumn the pots were emptied and samples of the soils were taken for analysis. After being refilled the pots were placed in the voliére and water was added up to about 60 per cent of the capacity. In that way the soils were alternately frozen and thawed during the winter.

The following spring grass was again sown in the soils and exhaustion of K continued in this way for 5 years.

Soil analyses

The following analyses have been made: Exchangeable K (Kt), Lt, Ft, Nat, Cat and Mgt (Fælles arbejdsmetoder 1972); K-release by extraction with 2 n HCl, K-fixation wet (*Dissing Nielsen, 1970*); K-release by extraction with 1 n HNO₃ (*Lind, 1971*); quantity/intensity relations for soil K (*Dissing Nielsen, 1971*); texture analysis (*Hansen, 1961*); exchange capacity (*Jensen, 1975*).

Moreover the solubility of soil K is estimated by the following methods: 5 g soil (10 g soil/sand) is extracted in a centrifugal glass for 10 min. with 50 ml 0.4 m CaCl₂. The suspension is centrifugated for 5 min. after which the K content in the decanted solution is determined. This process is repeated 5 times.

Results

In table 2 the analysis of variance for the soil samples is shown. It shows that the difference between year and soil types is significant for the 3 stated methods of determination of K in soil and there is an interaction between year and soil types. Addition of K has not affected the analytical indexes of the soil and in the following no distinction is made between the soil to which 4 mg K per 100 g was added yearly, and the soil not receiving K. All the soil analyses were repeated twice and in table 2 the relative mean deviation is shown. It is greatest for K_{HCl} despite the fact that more K is extracted with HCl compared with HNO₃ and particularly ammoniumacetate.

In table 3 the Kt value before the experiment and for the following years is shown. After one years cultivation Kt has decreased from 1 to 4

Table 2. Analysis of variance for Kt, K_{HCl}, K_{HNO₃}.

	Kt	K _{HCl}	K _{HNO₃}
Year	***	***	***
K-additions	none	*	none
Soil types	***	***	***
Year & K-additions	none	none	none
Year & soil types	***	**	***
K-additions & soil types	none	*	none
Year & soil types & K-additions	none	none	none
Deviation in per cent from average	3.2	7.3	5.5

***P < 0.001

**P < 0.01

*P < 0.05

units in all the soils except Abed, Højer, Silstrup and Ribe. At the start of the experiment there was no clear difference between Kt in clay and sand samples but after the exhaustion of K, Kt in sand soil was less than that in clay soil.

Prior to the experiment and after 5 years K-exhaustion the K-intensity was estimated. Comparison of the intensity before and after the experiment shows a decrease to the same level (table 4), irrespective of the K-intensity in the soil prior to the experiment. Table 4 also shows that the intensity in soils not exhausted of K is in most cases greater for sand than for clay soils.

The buffer base effect PBC_K which acts against changes of the K-intensity in the soil increased its capacity in several of the soils during the experiment. The reason for this is that the exhaustion emphasizes the initial part of the Q/I relation. At this section the slope of the curve is larger than in its latter part (*Dissing Nielsen, 1971*). Contrary to the intensity PBC_K is greatest in clay soils which is shown in table 4, where the soils are placed in order according to decreasing clay content.

The rate of release for exchangeable and non-exchangeable K is estimated by successive extraction with 0.4 m CaCl₂. Release of K in the first 3 extractions corresponds approximately to the amount of exchangeable K and for this part of the soil K, the release rate k₁ is calculated. It is

Table 3. Release of K from soils extracted with 0.5 m ammoniumacetate (Kt).

Locality	year	mg K per 100 g soil					
		1972	1973	1974	1975	1976	1977
Ribe		19.7	11.2	10.3	9.3	8.4	8.6
Silstrup		16.6	4.5	4.7	3.9	3.4	3.7
Abed		12.2	4.0	4.2	4.0	4.0	3.6
Højer		28.8	8.4	7.0	6.6	6.1	5.3
Blangstedgård		10.0	3.1	2.6	2.7	2.8	2.0
Askov ler		12.6	2.4	3.3	2.8	2.1	2.7
Ødum		23.4	3.4	3.0	3.0	3.8	2.8
Årslev		15.8	2.7	2.1	2.4	2.0	2.0
Roskilde		16.8	2.4	2.2	2.2	2.0	2.0
Tystofte		9.0	2.7	3.1	2.6	2.1	2.1
Virumgård		19.9	1.9	2.0	2.0	1.7	1.7
Spangsbjerg		16.6	1.4	2.4	1.8	1.5	1.4
Borris		10.8	1.4	1.2	1.6	1.4	1.3
Studsgård		1.9	0.6	1.8	1.4	1.4	1.3
Askov sand		4.0	2.0	2.0	1.4	1.2	1.1
Hornum		10.4	1.6	1.2	1.5	1.4	1.2
Lundgård		7.2	2.0	2.3	1.1	1.2	1.0
St. Jynde vad		14.8	0.9	0.8	1.0	0.9	0.7
Tylstrup		6.4	2.0	2.0	1.0	1.0	1.0

Table 4. Intensity (AREK) and capacity (PBCk) of K before and after exhaustion.

Locality	year	AREK		PBCk		$\frac{\log c_0 \div \log c_t}{\text{no of extraction}}$
		1972	1977	1972	1977	
Ribe		0.0030	0.0008	65	180	
Silstrup		0.0028	0.0004	55	68	
Abed		0.0021	0.0004	74	148	
Højer		0.0080	0.0008	50	86	
Blangstedgård		0.0035	0.0007	43	38	
Askov ler		0.0065	0.0007	24	37	
Ødum		0.0085	0.0005	33	38	
Årslev		0.0084	0.0006	21	36	
Roskilde		0.0162	0.0006	14	23	
Tystofte		0.0023	0.0007	32	32	
Virumgård		0.0218	0.0005	19	21	
Spangsbjerg		0.0220	0.0006	14	16	
Borris		0.0200	0.0006	14	18	
Studsgård		0.0019	0.0006	11	13	
Askov sand		0.0044	0.0006	10	13	
Hornum		0.0214	0.0007	9	14	
Lundgård		0.0200	0.0007	10	11	
St. Jynde vad		0.0251	0.0006	4	6	
Tylstrup		0.0215	0.0006	10	11	

seen from the curve (fig. 1) that this process is a first order reaction and consequently k_1 can be calculated as $k_1 = (\log c_1/c_2) / t_1$ or $k_1 =$

$c_0 = 1\text{st} + 2\text{nd} + 3\text{rd}$ extraction of mg K (exchangeable K) $c_t = c_0 \div$ K extracted (exchangeable K at the time t).

The release of K during the 4th, 5th and 6th extraction is non-exchangeable and this part of the reaction is of zero order, which means that the release rate k_2 is constant. For biotite *Mortland* (1958) states that K released by elution with NaCl is a zero order process and the chemically disintegrated product is vermiculite.

Table 5 shows k_1 and k_2 at the start of the experiment and after 5 years K exhaustion. In nearly all soils exhaustion of K has decreased k_1 and it shows that the final part of the exchangeable K is less soluble than its first part. On the other hand there is no difference between clay

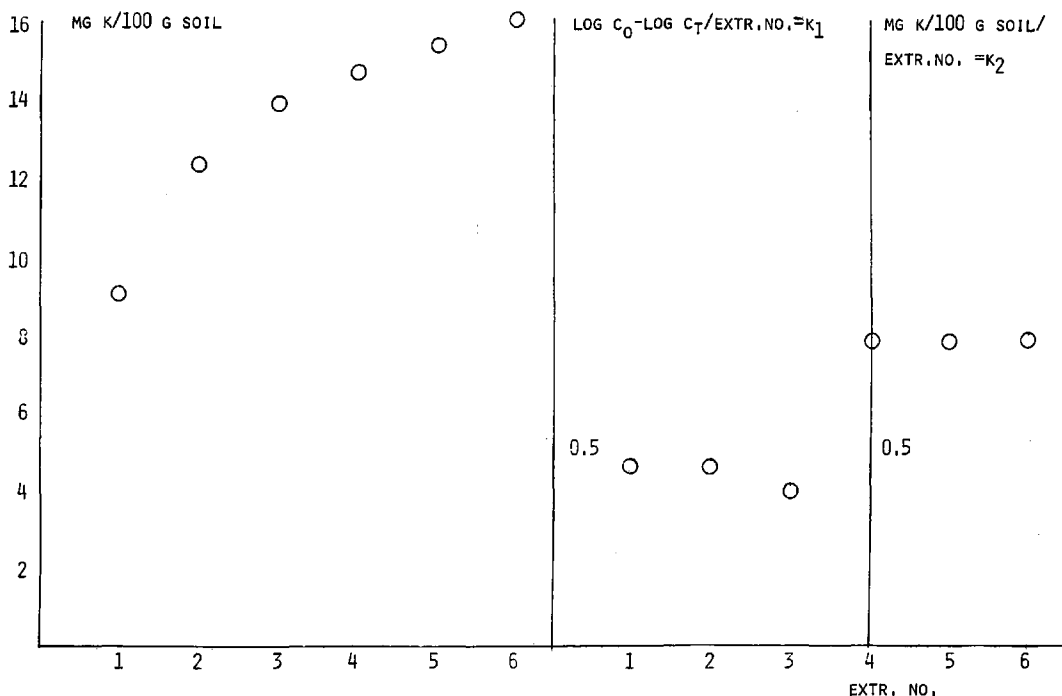


Fig. 1. Extraction with 0.4 m CaCl₂ of soil sample from Abed. Abscissa number of extractions; ordinate from

left 1. mgK/100 g soil, 2. solubility rate for exch. K, 3. solubility rate for non-exch. K.

Table 5. Solubility rate k_1 and k_2 for exchangeable and non exchangeable K extracted with 0.4 m CaCl₂ before and after exhaustion.

Locality	year	k_1		k_2	
		1972	1977	1972	1977
Ribe		0.50	0.45	1.23	0.55
Silstrup		0.46	0.42	0.87	0.20
Abed		0.47	0.46	0.73	0.20
Højer		0.59	0.39	0.80	0.10
Blangstedgård ...		0.49	0.29	0.53	0.10
Askov ler		0.50	0.55	0.83	0.10
Ødum		0.53	0.35	0.97	0.25
Årslev		0.59	0.50	0.37	0.15
Roskilde		0.53	0.38	1.00	0.20
Tystofte		0.46	0.41	0.60	0.20
Virumgård		0.57	0.44	0.97	0.20
Spangsbjerg		0.53	0.44	0.80	0.15
Borris		0.63	0.25	0.10	0.10
Studsgård		0.37	0.44	0.50	0.10
Askov sand		0.46	0.54	0.50	0.10
Hornum		0.63	0.39	0.23	0.10
Lundgård		0.52	0.39	0.50	0.10
St. Jynde vad		0.70	0.46	0.10	0.10
Tylstrup		0.49	0.39	0.53	0.20

and sand soils neither before nor after the experiment.

k_2 has clearly decreased during the experiment but in the case of k_2 there is also a difference between the soil types. Clay soils differentiate from sand soils in that the release rate k_2 for non-exchangeable K is greater both before and after the exhaustion. k_2 is large in soils from Ribe and Højer corresponding to the high content of clay and silt in these soils.

The soil indexes for K_{HCl} and K_{HNO_3} are given in table 6 and 7 and the figures show a decrease during the experiment of K_{HCl} and K_{HNO_3} . The decrease is most pronounced the first year and it continues the following years contrary to the data for K_t . Generally K_{HCl} and K_{HNO_3} are greatest for the clay soils, but as can be seen in the tables, the order is not always the same for K_{HCl} , K_{HNO_3} and clay content. Irrespective of the duration of the exhaustion, it is the same soils which have a high content of K_{HCl} and K_{HNO_3} and the opposite is also true.

Table 6. Release of K from soils extracted with 2 n HCl (K_{HCl}).

Locality	year	mg K per 100 g soil					
		1972	1973	1974	1975	1976	1977
Ribe		207	182	167	166	159	160
Silstrup		162	90	63	68	61	62
Abed		101	90	78	74	69	72
Højer		154	139	130	127	120	113
Blangstedgård		79	68	43	44	46	51
Askov ler		67	44	45	44	41	39
Ødum		94	58	54	46	45	46
Årslev		74	56	43	36	33	36
Roskilde		84	70	49	47	51	49
Tystofte		128	60	47	45	41	41
Virumgård		85	58	41	45	39	41
Spangsbjerg		50	49	35	29	30	30
Borris		48	36	21	20	22	24
Studsgård		29	28	15	10	13	13
Askov sand		24	22	18	20	17	9
Hornum		61	34	28	25	21	19
Lundgård		24	18	10	6	6	9
St. Jynde vad		46	19	4	3	6	3
Tylstrup		44	22	10	13	12	14

Table 7. Release of K from soils extracted with 1 n HNO₃ (K_{HNO_3}).

Locality	year	mg K per 100 g soil					
		1972	1973	1974	1975	1976	1977
Ribe		79	54	45	50	45	46
Silstrup		48	37	28	27	24	25
Abed		50	27	26	22	25	26
Højer		73	49	44	42	39	40
Blangstedgård		40	25	20	17	17	18
Askov ler		31	15	9	10	12	13
Ødum		50	31	20	25	21	22
Årslev		31	17	12	10	13	13
Roskilde		46	27	23	22	21	23
Tystofte		38	25	22	23	19	23
Virumgård		46	27	17	19	16	18
Spangsbjerg		29	10	13	11	9	10
Borris		20	10	7	5	5	5
Studsgård		8	5	2	5	3	5
Askov sand		15	7	4	5	4	5
Hornum		25	7	7	6	5	6
Lundgård		13	4	7	1	0	0
St. Jynde vad		18	4	0	1	0	1
Tylstrup		15	7	7	7	5	6

Table 8. Fixation of K in soils not exhausted and exhausted for K. Added 2.5 m eqv. K per 100 g soil.

Locality	mg K per 100 g soil		
	year	1972	1977
Ribe	13	38	
Silstrup	18	21	
Abed	18	28	
Højer	22	29	
Blangstedgård	16	13	
Askov ler	7	14	
Ødum	9	10	
Årslev	7	6	
Roskilde	9	6	
Tystofte	7	9	
Virumgård	4	9	
Spangsbjerg	6	5	
Borris	1	5	
Studsgård	3	5	
Askov sand	2	4	
Hornum	1	2	
Lundgård	1	3	
St. Jynde vad	4	4	
Tylstrup	2	4	

The fixation of K prior to and after the experiment is given in table 8. Particularly in the case of clay soils the fixation ability is increased by the exhaustion of K. Both before and after exhaustion of K the ability to fixate K is small in sand soils but it shows an increasing trend following the exhaustion of K in the soil.

Discussion of results

Kt, K_{HCl} and K_{HNO_3} are clearly different from year to year but it is only the first year that the variation of Kt is significant (table 3). The varying clay content of the soils is one of the reasons for the difference of their K_{HCl} and K_{HNO_3} (Dissing Nielsen, 1972). Even through the decrease in K_{HCl} and K_{HNO_3} differs for soil types, there is no clear distinction between clay and sand soils (table 6 and 7) and it must be assumed that exhaustion has changed the K compounds of primary as well as secondary minerals. The quantity of K added was far less than the plant requirement and it did not effect the analytical figures.

The exchangeable K in the soil is practically all consumed in the first growth season and the last 1

or 2 units do not disappear even in the course of several years exhaustion. The results in table 5 confirm that the solubility of exchangeable K decreases with exhaustion.

The relatively high Kt in soils from Ribe and Højer after exhaustion of K is explained by the high content of acid soluble K (tables 6 and 7) in these soils and by the relatively high solubility rate of this K-fraction prior to the experiment in soils from these 2 places.

Before the experiment the intensity of K is generally higher and there is therefore a higher K concentration in soil water in the sand soils as compared with the clay soils and this is explained by a higher sorption and capacity for K in the latter. During the experiment both sand and clay soils are exhausted of exchangeable K and the release rate decreases to nearly the same level. The intensity is also nearly the same for all soils after the exhaustion. In spite of a rather high final Kt in soils from Ribe and Højer, the final K intensity is also small for these soils and close to the threshold value for K uptake by plants. It is stated by Woodruff (1955) to be $0.0006 \sqrt{M/l}$.

Clay minerals are sorbing cations and among these are K. In this way the soil operates as a buffer against changes of K concentration in the soil water and reaches greatest effectivity in clay soils. Normally the capacity is constant irrespective of the method of cultivation and fertilization (Dissing Nielsen, 1971). Following the thorough exhaustion of K in the soils, it is the Q/I relationship for the non-exchangeable K which is determined and it has a PBCk level which is higher than that of exchangeable K.

The decrease of release rates k_1 and k_2 shows that the final soil K is less available than the first part. This is in accordance with results from Scheffer & Schachtschabel (1966) showing that exchangeable K is present partly on the surface and partly in the interior structures of the soil particles. It may be assumed that the latter part is less soluble than the former.

Dyal & Hendricks (1952) demonstrate that the distance between the layers in clay minerals is reduced by the uptake of K, but release of K facilitates access for other cations and also for

exchange of lattice K. The present results, however show that k_2 is reduced by the exhaustion in the majority of soils. *Newman* (1969) explains the decreasing exchange rate for mica K by the increase of diffusion distance.

Release of non-exchangeable K is influenced by other factors than exchange with cations, and weathering of soil minerals will also release K. Moreover k_2 before and after the experiment is estimated when the exchangeable K has been removed and *Mortland et al.* (1956) states that this K-fraction causes contraction of clay minerals.

Comparison of K_{HCl} , K_{HNO_3} and Kt shows that the decrease of the latter is also least the first year. *Semb* (1959) states that cultivation trials change the K-compounds in soil and the solubility decreases. The decrease of K_{HNO_3} was much greater than the corresponding plant uptake of K.

The results have demonstrated that K is also released from sand soil by acid extraction. It presumably originates from primary minerals as feldspars and micas which are decomposed by chemical treatment of the soil. However, *Reitemeir* (1951) demonstrated that K in primary minerals is practically inaccessible for the plants.

For soils capable of fixing K the fixation is increased by K-exhaustion. Similar results were previously shown in field and pot experiments (*Dissing Nielsen*, 1970). It may be assumed that a part of the utilized K was fixed and the vacant areas of fixation are refilled when K is supplied.

Conclusion

K-exhaustion of the soil drastically changes the soil analytical figures for K. The amount, intensity and solubility of exchangeable K all decrease to a level near the threshold value for uptake of K by plants.

The analytical figures for non-exchangeable soil K decrease more than the corresponding plant uptake of K and this is the case both in sand and clay soils.

The investigation showed that the exchangeable K can soon disappear from the soil. It can for example be consumed by the plants and the release of non-exchangeable K caused by

weathering and chemical changes is not sufficient to maintain an optimal K level in the soil.

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