Effect of exhaustive cropping on potassium depletion and clay mineral transformations

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Abstract
Identification of various clay minerals and their changes in the clay fraction of soil samples of 3 types of Indian soils before and after depletion of K by exhaustive cropping by growing Sudan grass (*Sorghum vulgare)* was done on the basis of strongest XRD peaks from basal planes. Sudan grass was grown in clay pots containing 5 kg of three types of soils from India and potassium was applied at the rate of 0, 50, 100, and 200 mg /kg before starting the experiment and after each of the first three cuttings. Seven cutting of Sudan grass were taken over a period totalling 280 days, each time the forage was grown for 4-6 weeks. It has been observed that, permanent removal of K from Illite-dominating soils leads to the degradation of Illite with its transformation to expandable or non-expandable minerals and decrease in Illite content was high in Vertisol (46%), medium in Alfisol (33%) and low in Inceptisol (14%)**.** There was a decrease in intensity of peak at 10 Å resulting in degradation of illite due to permanent removal of potassium ions by plants from inter-layers of clay lattice. This proved the formation of vermiculite, chlorite and smectite.

Key Words
K exhaustion, K depletion, clay mineralogy, and exhaustive cropping.

Introduction
The clay fraction was analyzed for semi-quantitative composition by X-ray diffraction following the method of Gjems (1967). Intensive cropping of soils without application of K-fertilizers may lead to a degradation of Illite as has been shown by Boguslawski and Lieres (1981) in a soil derived from loess. Ross *et al.* (1985) conducted a six-year field manure application experiment and found the transformation of vermiculite to pedogenic mica by fixation of potassium and ammonium. Potassium fixation caused a marked increase in the 1.0 nm peak at the expense of the 1.4 nm peak due to collapse of vermiculite layers to form pedogenic mica.

Mutscher and Tran Vu (1988) reported that biological depletion of potassium decreases the K concentration in the soil solution and induces the release of inter-layer potassium (K_i). Due to the massive decrease of the K concentration directly near active roots, easily releasable K_i takes part in the K supply even if the potassium concentration of the bulk soil solution and the average contents of exchangeable K are still high. Permanent removal of K from Illite-containing soils leads to the degradation of Illite and transformation of smectite and interstratified minerals. On natural habitats such K removal from the surface soil layer may be brought about by leaching. In cultivated soils large amounts of K are removed from the soil clays by plants. If the resulting K deficit in the soil is not balanced by K fertilizer application a degradation of clay minerals, especially Illite, will proceed. Thus, the growing plant seemed to be very effective in promoting the alteration clay minerals.

Tributh *et al*. (1987) studied the effect of potassium removal by crops on transformation of illitic clay minerals in soils from long-term field experiments and exhaustive cropping pot experiments. X-ray diffraction analysis of soil samples revealed that cropping without K fertilizer application had led to a substantial decrease in illitic content and to an increase of smectite and interstratified Illite / smectite minerals.

Materials and methods
Effect of continuous and exhaustive cropping on clay mineral changes due to K depletion was studied by X-ray diffraction analysis. Soils were dispersed and fractionated to separate clay fraction according to Jackson (1956). The mineralogy of the clay fraction is conveniently assessed separately, following pretreatment and separation of the fractions using the methods of particle-size analysis. This study was carried out on 3 types of Indian soils, namely Inceptisol (Delhi), Alfisol (Bangalore), and Vertisol (Bhopal), before and after high and moderate K exhaustion by Sudan grass to examine the effect of continuous exhaustive cropping on clay mineral changes due to K depletion. Sudan grass crop was grown in properly sealed 8-kg capacity clay pots

containing 5 kg of three types of soils, namely Alfisol, Vertisol, and Inceptisol from India and placed randomly in greenhouse and seven cuttings of biomass were harvested periodically. Measured quantity of deionized water was applied to the pots depending on the amount of evapo-transpiration which was calculated by daily weighing method. Twenty seeds of Sudan grass (*Sorghum vulgare* var. sudanensis) were sown in each pot. After 12 days of sowing, thinning was done to retain eight healthy plants per pot to ensure enough dry matter production and potassium removal from the soil. In order to study the effect of different levels of K exhaustion on potassium dynamics, pots were divided into four sets, receiving different potassium doses as fertilizer. Before sowing the crop, optimum doses of nitrogen (50 mg /kg), phosphorous (30 mg /kg), copper (2 mg /kg), manganese (5 mg /kg), iron (10 mg /kg), and zinc (5 mg /kg) were applied as basal and these nutrients were also supplied at the same rate after 1st, 2nd, and 3rd cutting. Potassium was applied at the rate of 0, 50, 100, and 200 mg /kg before starting the experiment and after each of the first three cuttings. Urea, di-ammonium phosphate and potassium chloride served as source of N and P, and K respectively, while the micronutrients were applied in their sulphate form. Treatment combinations were:

Type of soils (Inceptisol, Vertisol, and Alfisol)	: 3
Potassium treatments	: 4
Total number of treatments	: 3×4 = 12
Replication	: 6
Total number of pots	: 72

Sudan grass was harvested 7 times (totally 280 days) at a height of about three centimeters above soil level at 4-6 weeks intervals. After each cutting, small quantity of representative soil sample was also collected from each pot with a tube auger. After air drying, the soil samples were crushed using a wooden mortar and pestle and passed through a 2 mm round hole sieve and kept for chemical analysis. A 30 g of soil sample was treated chemically to remove cementing materials like carbonate, organic matter, free iron oxide and free silica by following the various pretreatments as outlined by Jackson (1967). Sand fraction was separated by wet-sieving (300 mesh) of the dispersed soil and dry weight of that was calculated. The upper 10 cm mixture of silt + clay was siphoned off at 8 h intervals for separation of clay from mixture. Silt fraction was dried and weighed and finally each fraction was expressed as a percentage of the total to obtain particle size distribution. The clay suspension (in NaCl) containing sufficient clay to give 10 mL of 2 % clay, was washed first with distilled water to remove excess of NaCl and was then saturated with Mg and K using normal solution of $MgCl_2$ and KCl by repeated centrifugation. The Mg and K saturated clays were then made electrolyte free using distilled water and alcohol, and were stored as 2 % clay suspension. Parallel oriented-aggregate clay specimens were prepared by taking 2 mL of the 2 % suspension on 2.5 cm x 4.0 cm glass slides, in duplicate, on a level surface and air dried These slides were then subjected to X-ray diffraction analysis by using Cu-K_α radiation and Ni filter in a X-ray diffractometer (Philips PW 1710). The different treatments given to the clay specimen prior to analysis as follows: (i) K-saturated air dried, (ii) K-saturated heated to 550°C, (iii) Mg saturated and (iv) Mg-saturated glycerol solvated.

From the X-ray diffractograms, the Θ values were obtained, which were used in calculating “d-spacing”, and were used in characterizing the soil clays, into its component clay minerals. X-ray profile was fitted using automated powder diffractometery software package and peak area was calculated from the fitted profile. A semi-quantitative estimation of the minerals was done by following the procedure as described by Jackson (1956). For semi-quantitative estimation, the peak size of the first order basal reflection is determined. The peak area is divided by a factor for each clay mineral to give the corrected intensity (Table 1). After measuring the peak area of any particular mineral and division of area by its own correction factor, all the areas were added up and made up to 100. The amount of conversion coefficient will be 100 / sum of peak areas, and with multiplication of peak area of the mineral into conversion coefficient, the per cent of that mineral can be obtained. After correction of area, the amounts of minerals were estimated in proportion to their corrected area.

Results and discussions

Quantitative analysis of clay minerals in soil is more difficult to carry out than qualitative analysis. The reflections of mixed-layer minerals are usually found between those of the pure compounds and this further complicates the interpretation of data. X-ray diffraction analysis was carried out on soil clay samples subjected to the following treatments: (a) K-saturated and air dried; (b) K-saturated and heated at 550°C; (c) Mg-saturated and air dried, and (d) Mg-saturated and glycerol solvated. To obtain glycerol solvated samples,

Table 1. Correction factor for peak intensity.

Mineral	Factor
Illite	1
Pyrophillite	1
Kaolinite	1
Chlorite	1
Vermiculite	3
Montmorillonite	4
Mixed layer minerals	2

the Mg-saturated clay samples were sprayed with 10% glycerol solution in ethanol. These pre-treatments are necessary for distinguishing the various clay minerals present in a clay sample. The X-ray diffractogram of the samples obtained after various pretreatments are presented in this chapter and the c-spacing [d (00l)] representing prominent reflections of the clay mineral present in each soil are described below:

X-ray diffractogram of Inceptisol clay (Mg-saturated and glycerol solvated) showed four peaks at 19, 14.4, 10, and 7.1 Å, which indicated the presence of smectite, vermiculite, and kaolinite in the clay. The peak of 7.1 Å suggested the presence of kaolinite which was confirmed by the diffractogram of the same sample obtained after pretreatment of K-saturated and heated at 550°C. The clay samples which have been separated from the original Inceptisol and from the same soil after exhausting the soil K to high level by Sudan grass were subjected to this type of analysis.

Vertisol clay contained predominantly smectite as shown by a broad peak in the X-ray diffractogram of Mg-saturated and air-dried sample at 14.9 Å, and Mg-saturated and glycerol solvated sample at 17.6 Å. This peak shifted to 12.5 Å on K-saturation (air dried). The other peak in Mg-saturated and glycerol solvated sample at 10.1 and 7.1 Å showed the presence of Illite and kaolinite as confirmed from the diffractogram of the same sample treated with K and heated at 550°C. The general patterns shown by clay sample from highly K exhausted Vertisol was almost same with the original soil but the differences in the semi-quantitative estimates are presented in Table 2.

The clay from Alfisol exhibited three peaks at 13.6, 10.2 and 7.1 Å with Mg-saturated and glycerol solvated clay which indicated the presence of vermiculite, Illite, and kaolinite in the sample. The decrease in Illite content due to K depletion was high in Vertisol (46%), medium in Alfisol (33%) and low in Inceptisol (14%). It is evident from the data presented in Table 2 that permanent removal of K from Illite-dominant soils leads to the degradation of Illite. From the semi-quantitative analysis it is confirmed that percentage of Illite decreased due to its exhaustion by plant uptake of K. Although there was an increase in percentage of kaolinite, vermiculite and smectite, it is not certain that the Illite has transformed to kaolinite, vermiculite and smectite. Increase in the calculated value of percentage of kaolinite might be due to the fact that intensity of x-ray diffraction depends on the mass fractions of a crystalline component among the total crystalline materials. Since the percentage of Illite decreased due to its apparent weathering the calculated mass fractions of the other minerals can be expected to increase. So keeping in mind the short duration of the K release a portion of Illite might have transformed to vermiculite only.

Table 2. Semi-quantitative estimation of clay minerals in clay fraction of different soils (%).

Soil	Mineral			
	Kaolinite	Illite	Smectite	Vermiculite
Iceptisol (Delhi)	9	66	16	10
- moderately Exhausted	11	61	17	11
- Highly Exhausted	12	57	18	13
Vertisol (Bhopal)	4	43	47	5
- moderately Exhausted	10	34	50	7
- Highly Exhausted	13	23	56	8
Alfisol (Bangalore)	91	9	-	-
- moderately Exhausted	93	8	-	-
- Highly Exhausted	94	6	-	-

This however needs to be verified by another set of corroborative evidences. In intensively cultivated soils, large amounts of K are removed from the soil and if this results in K deficit in the soil in the absence of K fertilizer application, a degradation of clay minerals, especially Illite, is a distinct possibility. Therefore in intensively cropped soils having Illite as the main clay mineral, K fertilizer application is not only a question of plant nutrition, but also of clay conservation. In all Indian soils after exhaustive cropping there was a decrease in intensity of peak at 10 Å indicating strongly of degradation of Illite due to permanent removal of K ions by plants from inter-layers of clay lattice.

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