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MINERAL MAKEUP OF CLAYS OF FOUR SOIL PEDONS FROM WESTERN HIMACHAL PRADESH

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The mineralogy of clay fraction ($< 2\ \mu$) of four soil pedons from western districts of Himachal Pradesh was evaluated by X-ray diffraction, per cent K_2O and CEC (Ca/Mg) data. The XRD patterns are characterised by normally broad and asymmetrical peaks often having well-developed shoulders, which are interpreted in terms of interstratification of expandible layer minerals present in these soil clays with illite. All the soil clays are predominated by illite. Expandible layer minerals (smeclite plus vermiculie) and kaolinite are present as next major minerals constituting 17-30% and 15-32% of the total, except Nauni pedon (P12) wherein kaolinite contributes only 5% to all the clay size minerals and quartz is as high as 10%. Chlorite is present in highest amounts (12-15%) in Dhaula Kuan pedon (P14) but almost nil in IIc and Al horizons of Bhota (P1) and Rishikesh (P3) pedons, respectively. Mean weathering index varies between 8.2 in Bhota (P1) and Rishikesh (P3) pedons to 7.4 in Nauni pedon.

Key Words: Mineralogy; Clays; Soil Pedons; X-ray Diffraction; Smectile; Vermiculite; Kaolinite; Chlorite

Introduction

A thorough knowldege of mineralogical composition of clay fraction is a prerequisite for improvement of fertilizer use efficiency, management of irrigation practices and proper understanding of soil development/degradation processes. Mineralogy also plays a pivotal role in placing the soils in classification system wherein genetic considerations are important. In Soil Taxonomy (Classification System of USDA) mineralogy has been included at the family level, a category which represents properties important for plant growth, but some orders, e.g., vertisols and oxisols are defined in terms of properties directly dependent on mineralogy.

A survey of the literature, however, reveals that information on the clay mineralogy of Himachal Pradesh soils is quite meagre and sporadic[1-4]. The present study was, therefore, undertaken to evaluate the mineral makeup of soil clays of four soil pedons from Western Himachal Pradesh.

Materials and Methods

Data on location, taxonomic units, morphological characteristics, particle size distribution, chemical and physico-chemical properties on these pedons have been presented[5]. For mineralogical analysis, the soil samples (< 2 mm) were

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Table I

Semi-quantitative mineralogical composition of clays

Depth (cm)	Horizon	Illite	Vermicultie	Smectite	Chlorite	Kaolinite	Quartz	K_2O (%)	CEC ca/mg me/100 g	Mean weathering index
				Pedon 1, BHOTA (Typic Hapludalfs)						
0-15	Al	38	10	12	10	22	8	3.80	28.4	8.1
42-78	Bt2	43	10	10	2	30	5	4.10	27.7	8.2
110-175	IIc	48	8	10	Tr	32	2	4.70	26.6	8.2
				Pedon 3, RISHIKESH (Fluventic Eutrochrepts)						
0-12	Al	43	15	15	ND	25	2	4.30	34.7	8.2
48-82	IIB2	40	15	13	5	22	5	3.90	31.9	8.1
115-150	IIIC	40	12	10	10	20	8	4.00	29.2	8.0
				Pedor 12, NAUNI (Typic Udorthents)						
0-15	Al	55	15	10	5	5	10	4.5	29.5	7.5
35-75	C	55	25	Tr	5	5	10	4.1	30.2	7.4
				Pedon 14, DHAULAKUAN (Typic Hapludalfs)						
0-15	Al	48	17	Tr	15	18	2	4.9	26.2	8.0
30-70	Bt	43	22	Tr	12	18	5	4.3	27.6	8.0
90-130	IIC2	45	10	10	12	15	8	4.1	26.6	7.9
			17-30%			15-32%				

pretreated with NaOAc (*p*H 5.0), 30% H_2O_2 and dithionite citrate-bicarbonate (DCB) and the clay ($< 2\ \mu$) was fractionated by the method described by Jackson[6].

Two parallel oriented specimens one Mg-saturated and the other K-saturated, were prepared on glass slides for X-ray diffraction analysis. The Mg-saturated specimen was X-rayed before and after glycerol solvation, whilst the K-saturated samples were X-rayed after airdrying, heating at 300 °C for one hour and then at 550 °C, again for one hour.

X-ray diffraction analysis was performed using Phillips X-ray diffractometer with CoK_∞ radiation at a scanning speed of 1°2 θ/min and a time constant of 4. Semiquantitative estimation of minerals was worked out by comparing the relative intensities of diagnostic diffraction maxima, K_2O content, and CEC data (Ca/Mg) of the clay fractions. The K-content was determined by flamephotometry, after decomposition of the sample with $HF:HClO_4$.

Results and Discussion

X-ray Diffractometry

The XRD patterns in the 7Å region are quite dissimilar as is evident in Figs 1 to 5 and Table I. They are discussed below:

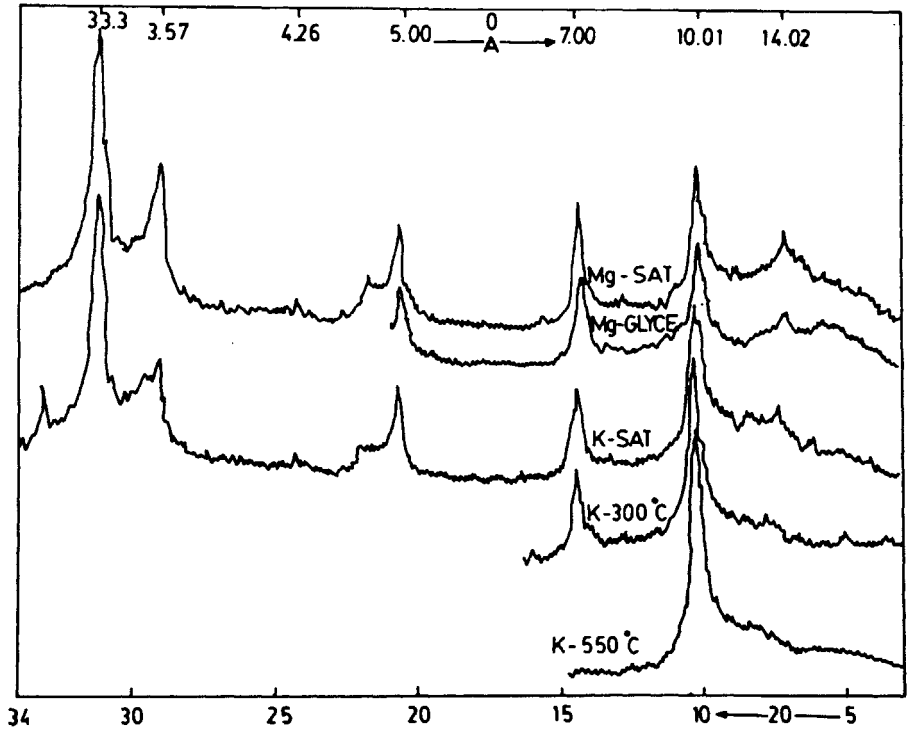

Fig. 1 X-Ray diffractograms of clay, B2t horizon, pedon

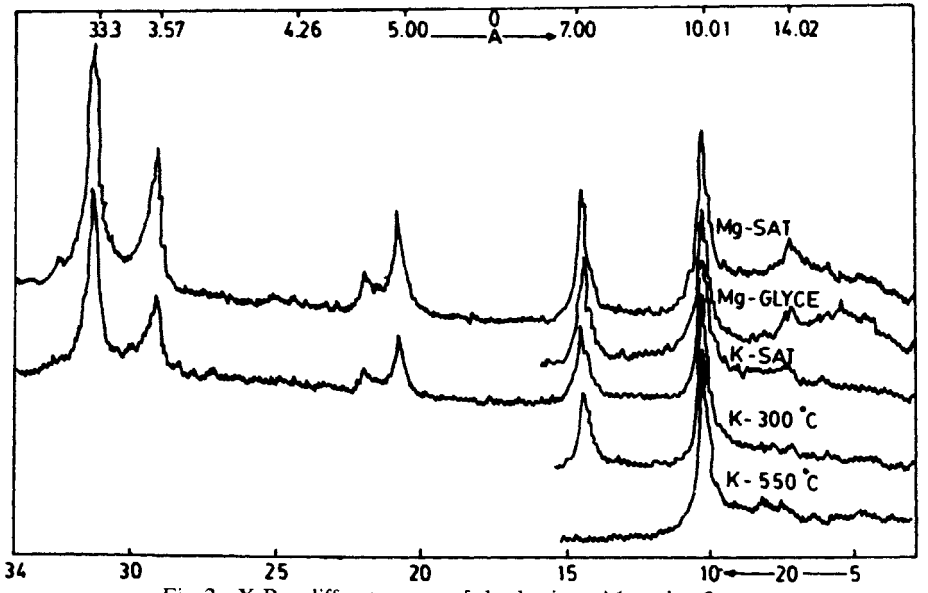

Fig. 2 X-Ray diffractograms of clay horizon A1, pedon 3

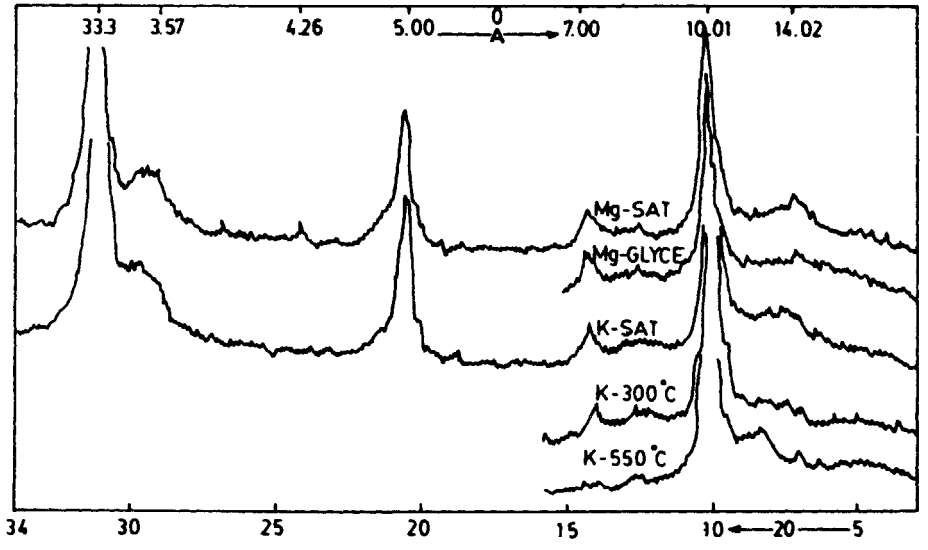

Fig. 3 X-Ray diffractograms of clay, Al horizon, pedon 12

10Å Illite Peak

The X-ray diffraction patterns of Mg-saturated sample shows asymmetry of the (001) illite peak towards low 2-θ angle side and broadening of (002) peak. On solvating with glycerol the Mg-saturated specimen exhibits quite different feature from each other in that (i) the 10Å peak develops into a more symmetrical form without showing either a shift in d-spacing or any shoulder on either side of the peak (Figs 2&3; Al samples of pedon 3 & pedon 12 and

II C2 sample, pedon 14); (ii) the 10Å peak sharpenes with some shoulders on the low angle side of the peak (Figs 1 and 5; all the samples of pedon 1 and Bt sample of pedon 14); and (iii) the 10Å-illite peak further broadens forming a long tail with or without slight shift of the peak towards low 2θ-angle side (Fig. not shown; II B2 sample, pedon 3, Al sample, pedon 14). This is similar to the XRD interpretation made by Weaver[7] and Hower and Mowett[8], suggesting that illite in the above three categories of samples contains < 10, 10-15 and > 15 per cent expandible layers, respectively.

An exception to above is the C sample, pedon 12 (Fig. 4) which contains almost discrete form of illite and wherein the 10Å illite peak neigher shows any asymmetry in the Mg-saturated specimen nor is there any change in either the sharpness of the intensity on glycerol solvation of Mg-saturated specimen.

Patterns in the > 10Å Regions

The X-ray diffractograms of all the samples in the Mg-saturated state show a 14Å maximum, which on solvating with glycerol display either (i) a plateau bridging the 14Å-18Å d-spacings with a small part of the maximum remaining at 14Å (Figs 1 & 3; Al & Bt2 samples of pedon 1; II B2 and IIIC samples of pedon 3, Al sample of pedon 12 and II C2 samples of pedon 14) or (ii) two separate maxima, one at 14Å and the other at 18Å with a plateau bridging these two maxima (Fig. 2; II C sample of pedon 1 and Al sample of pedon 3), or (iii) the 14Å peak remains intact without any significant change either in intensity or in *d*-spacing (Figs 4 & 5; C sample of pedon 12; Al and Bt samples of pedon 14).

A 14Å XRD maximum from a Mg-saturated and air-dried specimen may arise due to vermiculite, chlorite, smectite or an interstratification of these components with each other or with mica. If mica is a component then smectite

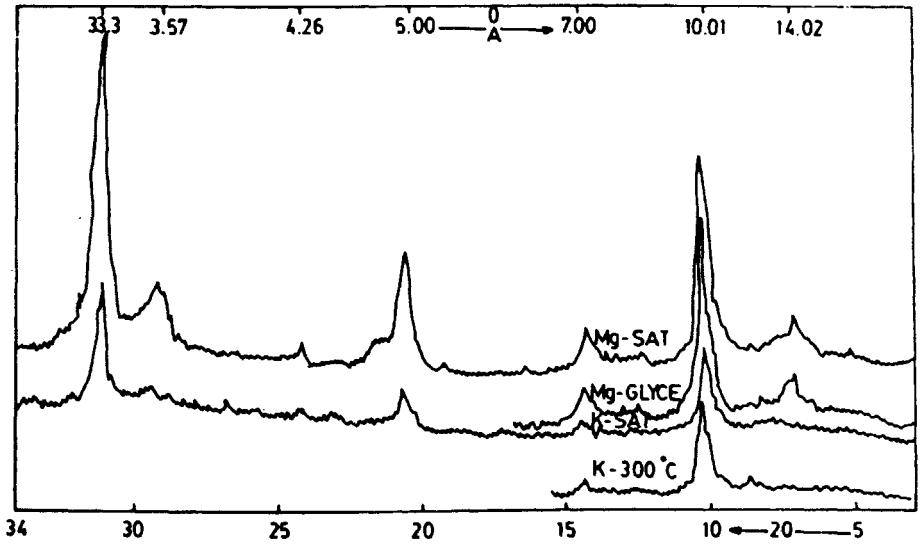

Fig. 4 X-Ray diffractograms of clay, C horizon, pedon 12

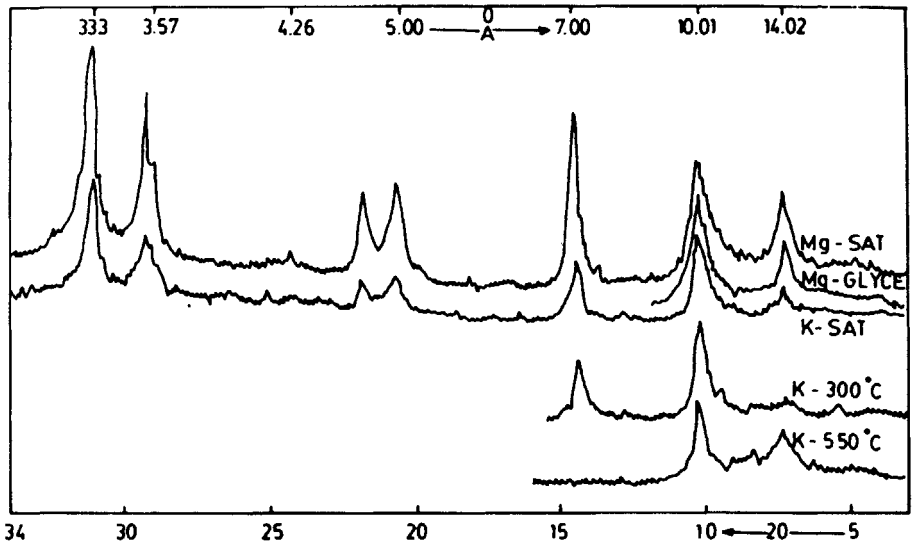

Fig. 5 X-Ray diffractograms of clay, Bt horizon, pedon 14

alone or in combination with vermiculite and/or chlorite must be present as one of the components to give rise to a peak at 18Å d-spacing in the glycerol solvated specimen.

In all the clay samples of category (i) above, the 14Å maximum even in the Mg-saturated state is quite broad. A common feature of all the XRD patterns is that either there is high background in between the 10-14Å maxima or some feeble maxima are developed in this region. This indicates some sort of irregular interstratification. The d-spacings of the first four (00 1) reflections do not form an integral series suggesting that the mineral is an irregular mixed-layer. Irregular mixed layering is also indicated by the asymmetry of the 14Å maximum.

Since the 14Å peak, though partially, migrates and merges to increase the intensity of the 10Å-illite peaks on K-saturation and further on heating at 300 °C and 550 °C, expandible layer must be involved in the interstratification system. Migration of 14Å peak towards the low 2 θ-angle side to form a 14-18Å plateau on glycerol solvation suggests that smectite is present. The CEC (Ca/Mg) data show that smectite & vermiculite are the main expanding layer minerals is these soil clays. According to Brown and McEvan[9] a single peak intermediate between the d-spacings characteristics of the two minerals develops only when the distance between the two d-spacings is small (10Å & 14Å and in the Mg-saturated state). When widely separated (10Å & 18Å in Mg-glycerol solvated state) two separate maxima can appear. Cradwick and Wilson[10], however, with the help of calculated curves showed that the development of a peak also depends on the degree of segregation of the component layers in the irregularly interstratified system. They showed that any component would be able to give rise to a separate peak only when one of the components is present in

segregated form in the interstratified system. If the segregated component is not present in sufficiently good amount, a single peak at an intermediate d-spacing rather than a separate peak due to the component would develop, and increasing randomisation of the components leads to peaks of reduced intensity and in extreme cases to their elimination. Thus, a small peak ~ 14Å with a 14Å-18Å plateau in case of samples of category (i) and two small peaks at 14Å and 18Å with a plateau bridging these two spacings clearly suggest that the samples of category (i) are less segregated then those of category (ii), and that in the former smectite layers are completely randomised. This phenomenon of segregation and randomisation has been reported earlier[11-13] while studying the interstratification phenomenon in detail in the soils of Western UP. The category (iii) samples show neither expansion nor any reduction of the intensity of 14Å maximum on glycerol solvation. However, the peak migrates partially or completely on K-saturation and heating at 300 °C to strengthen the 10Å illite peak. This suggests that though smectite is beyond the XRD detection limits, vermiculite constitutes a significant proportion of these clays (Table I). All the Mg-saturated samples show a plateau or quite a good amount of shoulders in between the 10Å and 14Å peaks. This suggests that this category of samples also contains interstratified minerals involving illite and vermiculite components, though with some interlayering of the vermiculite layers as discussed below.

10Å-14Å Stable r-nase

The XRD patterns show that the 14Å maximum does not collapse completely on K-saturation or K-300 °C heating in many of the samples (Fig. 5, Table I); some of the samples even exhibit an increase in intensity to more than three times its original value on K-550 °C heat treatment (Fig. 5). This effect agrees with the observation of Brindley and Ali[14] and Brindley and Youell[15] that some chlorites show an increase in the first order and a decrease in the second order basal reflection as a result of heating at 600 °C. The increase in intensity of 10Å-14Å peaks on K-550 °C heating is indicative of the presence of pedogenic chloride or 2:1-2:2 intergrades in these soil clays. The shape, intensity and d-spacing of these 10Å-14Å maxima very widely depending upon whether the sample contains fully developed chlorite represented by a 14Å maximum (Fig. 5) or partial hydroxy interlayering, the latter displaying either a peak (Fig. 3) or a plateau in between 10Å-14Å *d*-spacings. According to Brown and McEvan[9], a peak intermediate to 10Å-14Å d-spacings and broadening of 10Å peak on K-550 °C heating are the characteristics of 2:1-2:2 intergrades[6] and a hump or plateau is formed only when chloritisation of expandible layers is partial.

7Å Peak

A 7Å-XRD peak in the Mg-saturated and air-dried specimen may arise as the 002 reflection of 14Å expandible minerals (smectite and vermiculite), stable layer minerals (chlorite) or due to Kaolinite (001 reflection). Since 14Å expandible layer minerals are known to migrate to 10Å completely on K-300 °C heating, the 14Å peak, owing to pedogenic nature of chlorite, gets strengthened only after 550 °C heating; so also, the 7Å the 14Å kaolinite peaks collapse only

after 500 °C heating and the 14Å peak does not exist in some of the K-300 °C heated specimens. In such cases the 7Å peak in the K-300 °C heated specimen is used for the quantification of kaolinites in those soil clays.

Summary

The mineral makeup of the clay fractions shows that easily weatherable primary minerals such as pyroxines, amphiboles and feldspars are absent and the clays do contain only the clay minerals. This together with a quite high content of expanding layer minerals suggests that pedogenesis has affected the soils of this mountainous transect of Himachal Pradesh. The main weathering index calculated on the basis of semi-quantitative mineralogical composition of clay ($<0.2\ \mu$) ranged from 7.9 to 8.2 except in Nauni pedon (P12), wherein these values remain as low as 7.4. This shows clearly that Nauni pedon is in the initial stage of weathering, whilst Bhota (P1) Rishikesh (P3) and Dhaulakuan (P14) pedons have entered the intermediate stage.

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