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Role of Clay Minerals and Aggregation in Stabilizing Soil Organic Carbon in Forest Ecosystems of Siwalik Foothills, North-India

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Abstract

This study investigated the relationship between soil mineralogy and soil organic carbon (SOC) stored within aggregate size fractions in forest ecosystems in the Siwalik foothills of North India. Surface and subsurface soils (0–10 and 10–30 cm) were sampled across representative forest stands with different vegetation types and land-use histories. The mineralogical composition (X-ray diffraction) and texture were characterized for bulk soil and clay fractions; aggregate-size fractionation (wet-sieving) separated macroaggregates (>250 µm), microaggregates (250–53 µm), and silt+clay-associated fractions (<53 µm). SOC and bulk density were measured for each fraction and horizon separately. We quantified the proportion of SOC protected within aggregates, evaluated mineral–carbon associations, and used ratio and correlation analyses to link the abundance of specific clay minerals (e.g., illite, kaolinite, and smectite) to SOC stabilization. The results showed that microaggregates and the silt+clay-associated fraction contained the majority (>60%) of SOC, while soils rich in expandable 2:1 clays (smectite/vermiculite) displayed higher SOC per unit clay mass than illite/kaolinite-dominated soils. Aggregate-protected SOC correlated positively with fine fraction (<53 µm) mass and specific surface area. Our findings indicate that clay mineralogy, coupled with aggregate architecture, is a key factor controlling carbon persistence in Siwalik foothill forest soils and highlights the potential of native forest cover to enhance SOC sequestration in these geologically young landscapes. The clay minerals in order of their dominance were vermiculite, illite, kaolinite, and mixed-layer minerals. The presence of vermiculite and illite in appreciable quantities indicates that they were synthesized from the K-rich soil solution. The mineral suites identified in this study show that the geological, climatological, and topographical factors of the region collectively played a dominant role in their formation and transformation. After critical appraisal of the results, it may be deduced that the mineralogical composition, physicochemical properties, and total elemental analysis of the soils do not show any deficiency of the bases and other plant nutrients in general. The inherent fertility of the soil is good, as indicated by the clay mineralogy of the soil, and biotite and feldspar, together with mica, are important sources of nutrients for the vegetation in the soils of the Siwalik Valley.

Keywords: Soil mineralogy, Clay minerals, Soil aggregates carbon, SOC stabilization

Introduction

The location of soil organic matter (SOM) in the soil structure appears to be the key factor governing SOM stocks and dynamics (Oades, 1988). An aggregate is a naturally occurring cluster or group of soil particles that cohere more strongly than the surrounding particles. The size distribution of soil aggregates is important because the size of aggregates determines their susceptibility to the loss of soil organic matter (Tisdall and Oades 1982). Microaggregates are considered to be the repository of the most stable C pool in soils (Edwards and Bremner 1967; Tisdall and Oades 1982; Six *et al.* 2000), and the largest proportion of C input into stable soil organic matter pools was found in microaggregates (Kong *et al.* 2005). Torn *et al.* (1997) reported that the largest changes in the quantity and turnover of soil organic carbon across landscapes and over long timescales may be due to variations in passive (mineral-stabilized) carbon deep in the soil. Passive carbon pools are controlled by soil mineralogy (Torn *et al.*, 1997). Soil minerals found in different soil types affect soil carbon dynamics. Therefore, soil mineralogy is important in determining the quantity of organic carbon stored in soil, its turnover time, and atmosphere–ecosystem carbon fluxes during long-term soil development (Torn *et al.* 1997). Baldock and Skjemstad (2000) described the stabilization of organic materials by the soil matrix as a function of

nature of mineral fractions and its surfaces capable of adsorbing organic materials. X-ray diffraction is the most common technique used to study the characteristics of crystalline structures and determine the mineralogy of finer-grained sediments, especially clays. X-ray diffraction analysis is a useful method for identifying and making semi-quantitative estimates of the crystalline mineral components of soil. The intensities of the diffraction maxima are related to the number of corresponding diffraction planes in a sample and provide a basis for estimating the concentrations of the mineral species present.

This study aimed to analyze the soil aggregate composition and carbon content of soil aggregate fractions in different forest ecosystems. We also aimed to study the differences in clay mineralogy of soils from the two studied forest divisions using X-ray diffraction.

Materials and Methods

Study Site

The study sites are located in the YFD in the northeastern part of Haryana State in the Siwalik region. Geologically, the Siwalik belong to the tertiary deposits of the outer Himalayas and are chiefly composed of low sandstone and conglomerate hills, the solidified and upheaved detritus of the great range. The configuration of the tract is variable, consisting of the Siwaliks, foot hills, and plains with gentle slopes. The study was performed in the Siwalik ranges, the foot hills of the northwestern Himalaya located at 30° 00' 00" to 30° 30' 00"N latitude and 70° 00' 00" to 77° 00' 45"E longitude in the forests of Kalesar National Park in Yamunanagar Forest Division (YFD) and between longitude 76° 46' 40" to 77° 0' 33"E and latitude 30° 40' 50" to 30° 54' 40"N in the reserved and protected forests of Panchkula forest divisions (PFD) of northern Haryana covering an altitude of 300 to 650 msl at Kalesar forest and 370 to 1100 m at Morni hills. The climate of the area is subtropical monsoonal. The annual rainfall varies from 716 to 1897 mm. The Siwaliks are composed of alluvial detritus from the solidified and upheaval detritus of the Great Himalayan Range (Wadia, 1961) in northern India.

Soil sampling was conducted using a soil corer (12 cm diameter) at depth interval of 0-15 cm, 15-30 cm, 30-45 cm, 45-60, and 60-100 cm, with five replicate samples for each depth. Samples from six forest types (Champion and Seth, 1968) covering northern tropical dry deciduous *Shorea robusta* plain forests, northern tropical dry deciduous siwalik *Shorea robusta* forests, mixed dry deciduous forests in YFD and Dry deciduous forests, mixed dry deciduous forests, and sub-tropical pine forests in PFD were transported to the laboratory in polythene bags and stored at low temperatures.

Analysis of Soil Characteristics

To analyze the effect of vegetation composition on soil organic matter (SOM) in forest ecosystems, soil samples were collected from various forest systems. After removing the ground floor litter and plant residues, the underlying soil was sampled using a soil corer (12 cm diameter) at depth interval of 0-15 cm, 15-30 cm, 30-45 cm with five replicate samples obtained for each depth. At the time of analysis, the soil samples were air-dried and sieved using a 2 mm sieve to ensure complete sieving, discarding only the recognizable plant residues and pebbles. Subsamples of air-dried soil were used to determine the soil pH and organic carbon

content.

Soil bulk density was determined by collecting a known volume of soil using a metallic corer at depths of 0-15 cm, 15-30 cm, 30-45 cm of soil depth and the soil was dried at 100 °C until a constant weight was achieved. The bulk density was determined using the soil weight/volume relationship. Soil pH was determined using a soil and distilled water (1:2 w/v) suspension and an electronic pH meter.

Analysis of Organic Carbon

Subsamples of air-dried soil and separated soil aggregates were analyzed for organic carbon using the dichromate oxidation method (Kalembasa and Jenkinson 1973). The organic carbon content of the soil was oxidized by the nascent oxygen produced during the reaction of concentrated sulfuric acid using 0.5N potassium dichromate. The residual potassium dichromate was titrated with 0.2N ferrous ammonium sulfate using diphenylamine as an indicator in the presence of phosphoric acid.

Statistical Analysis

The data on soil carbon and soil aggregate carbon were analyzed using one-way analysis of variance. Least significant difference (LSD) values at the 5% level of significance ($p \geq 0.05$) were calculated following Gomez and Gomez (1984).

X-Ray Diffraction (XRD) for Clay Mineralogy

Soil samples from 0–15 cm soil depth were collected from different forest ecosystems of the Yamunanagar and Panchkula forest divisions from April 2023 to October 2024. The soil samples were air-dried, gently crumbled manually, and sieved (2 mm) to remove plant debris and root materials. The soil samples were further processed by taking five replicates (10 g air-dried soil) to collect a sufficient amount of clay for X-ray diffraction (XRD).

Removal of soluble salts

First, 50 ml of 1 N NaCH_3COO (pH 5.0) was added to a 500 ml beaker, stirred, and kept overnight. Next morning 10-15 ml of 1 N NaCH_3COO was added again; if effervescence persisted, more reagent was added. If no effervescence appeared, the mixture was centrifuged at 1500 rpm for 5 min. The supernatant was discarded. This treatment helps in the removal of CaCO_3 , and an acidic pH helps in the removal of organic carbon.

Removal of organic matter

The organic matter in the soil samples was decomposed by treatment with hot 6% H_2O_2 with constant stirring at low heat using a hot water bath. To ensure complete decomposition of soil organic matter, the soil samples were treated twice with 6% H_2O_2 . The digested soil samples were then transferred to a 1 Litre sedimentation cylinder with repeated washings with deionized water to raise the volume to one litre and allowed to settle for 12 h (overnight). The suspended clay ($< 2\mu\text{m}$) in the sedimentation cylinders was siphoned out in a 500 ml corning glass beaker by repeated sedimentation and decantation. The clay suspension was collected in a beaker. The $< 2\mu\text{m}$ clay fraction that was wholly separated from the soil was used to examine the clay mineralogical composition using X-ray diffraction (XRD). Duplicate clay solutions containing 50 mg clay each were

placed into 10 ml glass tubes and then washed twice with 8 ml of an equal mixture of 1M NaCl and 1M NaCH₃COO (pH 5.0) by centrifugation to lower the pH. Of the duplicate sets, one was saturated with K and the other with Mg by washing three times with 8 ml of 1 M KCl and 0.5M MgCl₂, respectively.

Magnesium saturation

For Mg saturation, the sample tube was filled with MgCl₂ and allowed to flocculate. The mixture was centrifuged and washed again with MgCl₂. The sample was washed with MgOAc, followed by washing with MgCl₂, centrifugation, and discarding of the supernatant. Equal volumes of distilled water and methanol were added, and the sample was centrifuged. After centrifugation, the supernatant was discarded. The samples were repeatedly washed with absolute methanol until they were chloride-free. Now added 1-2 drops of acetone if it does not settle in the last washing. The acetone was completely drained, and the samples such for 5-10 minutes and after The volume was then increased to 10 ml with distilled water.

Potassium saturation

For K saturation, the sample tube was filled with KCl and allowed to flocculate. The mixture was centrifuged and washed again with KCl. The sample was washed with KOAc and KCl, centrifuged again, and the supernatant was discarded. Equal volumes of distilled water and methanol were added, and the sample was centrifuged. After centrifugation, the supernatant was discarded. The samples were repeatedly washed with absolute methanol until they were chloride-free. Now, added 1-2 drops of acetone if it does not settle in the last washing. The acetone was completely drained, and the samples such for 5-10 minutes and after The volume was then increased to 10 ml with distilled water.

The clay samples in the tube were thoroughly suspended in 1 ml of water. An aliquot of 0.4 ml of the clay solution was dropped onto a glass slide (28×48 mm) covering two-thirds of its area, air dried, and X-rayed. Clay samples prepared for analysis were examined in the following order to efficiently identify and differentiate the characteristic mineral components. (1) Mg-saturated, air-dried samples; (2) Mg-saturated, Glycerol solvated samples (for 48 h); (3) K-saturated, air-dried samples; and (4) K-saturated, heated samples (heated at 550⁰ °C for 4 h in a muffle furnace).

The XRD analysis of samples was performed using an XPERT-PRO model diffractometer with Cu as anode material using CuK α radiations at 45KV and 40mA (λ 1.54443 Å) and at a scanning speed of 0.017 in a continuous scanning mode over a range of the 2 θ range 4° to 30° (Mg-saturated samples) and 4° 2 θ to 20° 2 θ position (K-saturated samples). Clay minerals were identified following Brown and Brindley (1980) and Moore and Reynolds (1989). The relative mineral content in the clay fractions was semi-quantitatively estimated based on the XRD peak intensities. In the present estimation, the peak height was used as the peak intensity by assuming the relative proportion of the minerals of the samples normalized to 100% and the same proportionality between the peak intensity and the content for each mineral.

Results:=

Analysis of Soil Characteristics

The variation in soil characteristics of the studied forest ecosystems is shown in Table 1. In northern tropical dry

deciduous *Shorea robusta* forests, pH was found to increase with depth, ranging from 6.27±0.06 to 6.82±0.09, with increasing bulk density (1.10±0.04 to 1.34±0.05). In northern tropical dry deciduous Shiwalik *Shorea robusta* forests, the pH was comparatively higher than that of plain northern tropical dry deciduous *Shorea robusta* forests, ranging from 7.83±0.07 to 8.24±0.05 with increasing soil depth and bulk density (Table 1). In mixed dry deciduous forests, the pH varied from 6.99±0.07 to 7.52±0.05 in the Yamunanagar forest division. In the Panchkula forest division, the pH of dry deciduous forests, mixed dry deciduous forests, and sub-tropical pine forests ranged from 5.89±0.03 to 8.34±0.05, with a variation in bulk density from 1.17±0.08 to 1.33±0.06 (Table 1).

Soil Organic carbon

The organic carbon concentrations (g kg⁻¹) in the soil of various forest types at Yamunanagar and Panchkula forest divisions up to a depth of one meter are presented in Table 2. A regular significant decrease in organic carbon concentration ($p < 0.05$) was observed at all forest types along the depth of up to one meter soil profile under the study area. At the Yamunanagar forest division, the soil carbon concentration (g kg⁻¹) was found to be maximum at the northern tropical dry deciduous Shiwalik *Shorea robusta* forests at 0–15 cm soil depth (6.93±0.27), followed by the northern tropical dry deciduous *Shorea robusta* forests (6.28±0.03) and mixed dry deciduous forests (5.11±0.07), which decreased significantly with an increase in soil depth in all forest types (Table 2). In the Panchkula forest division, the maximum carbon concentration (g kg⁻¹) was observed in sub-tropical pine forests (10.11±0.07), followed by mixed dry deciduous forest ecosystems (4.94±0.04) and dry deciduous forest ecosystems (3.73±0.10), along with a significant decrease ($p < 0.05$) in carbon concentration with increasing depth up to a one-meter profile.

In the northern tropical dry deciduous *Shorea robusta* forest, the total soil carbon (Mg C ha⁻¹) at different soil depths was 10.40±0.29 (0–15 cm), 7.63±0.39 (15–30 cm), 2.48±0.11 (30–45 cm), 0.72±0.05 (45–60 cm), and 0.75±0.08 (60–100 cm). The carbon pool at 0–30 cm soil depth was 82.02% of the total pool up to 100 cm soil depth. The total soil carbon pool at the northern tropical Shiwalik *Shorea robusta* forest was 11.42±0.33 (0–15 cm), 7.79±0.49 (15–30 cm), 2.68±0.22 (30–45 cm), 1.39±0.14 (45–60 cm), and 1.09±0.40 (60–100 cm). The carbon pool at 0–30 cm soil depth was 78.83 % of the total pool up to 100 cm soil depth. The spatial distribution of the soil organic carbon pool in mixed dry deciduous forests at the Yamunanagar forest division was 8.20±0.44 (0–15 cm), 7.10±0.32 (15–30 cm), 1.64±0.18 (30–45 cm), 1.17±0.35 (45–60 cm), and 0.45±0.26 (60–100 cm). Approximately 82.43% of the total carbon pool was confined to the top 30 cm of soil. In the dry deciduous forest of the Panchkula forest division, the total soil carbon (Mg C ha⁻¹) at different soil depths was 6.55±0.55 (0–15 cm), 3.39±0.29 (15–30 cm), 0.61±0.09 (30–45 cm), 0.24±0.07 (45–60 cm), and 0.22±0.07 (60–100 cm). The total soil organic carbon pool in mixed dry deciduous forests at Panchkula forest division was found to vary 8.67±0.54 (0–15 cm); 4.41±0.26 (15–30 cm); 0.93±0.02 (30–45 cm); 0.27±0.06 (45–60 cm); 0.31±0.18 (60–100 cm). The carbon pool at 0–30 cm soil depth in dry deciduous and mixed dry deciduous forests were 89.79% and 89.71% of the total pool up to 100 cm soil depth, respectively. Approximately 87.82% of the total carbon pool

was present up to 30 cm in the subtropical pine forests of the Panchkula forest division, accounting for 18.35 ± 1.08 (0–15 cm); 5.80 ± 0.49 (15–30 cm); 1.76 ± 0.09 (30–45 cm); 0.67 ± 0.15 (45–60 cm); and 0.95 ± 0.26 (60–100 cm).

Soil Aggregates carbon

The concentration of carbon in soil aggregate fractions followed the order: Small macroaggregates > macroaggregates > microaggregates > silt and clay fractions in the Yamunanagar forest division. The concentration of total organic carbon decreased with reduced aggregate size at all depths and locations. The organic carbon content in soil aggregates varied significantly with soil depth ($p < 0.05$). There was a significant increase in carbon concentration (g kg^{-1}) with increasing aggregate size class. The total carbon concentration was greater in smaller microaggregates (250 μm –53 μm) as compared to silt and clay associated with soil fractions (<53 μm) (Tables 3 & 4). In the forest ecosystems of the Yamunanagar forest division, the carbon concentration in different size fractions ranged from 0.18 to 4.82 g kg^{-1} in northern tropical dry deciduous *Shorea robusta* forests, 0.14 to 4.20 g kg^{-1} in northern tropical dry deciduous shiwalik *Shorea robusta* forests, and 0.15 to 5.22 g kg^{-1} in mixed dry deciduous forests (Table 3). In forest ecosystems of the Yamunanagar forest division, the carbon concentration in macroaggregates (> 2 mm) varies from 0.39 g kg^{-1} to 4.31 g kg^{-1} , in small macro aggregates (2 mm–250 μm) 0.32– 5.22 g kg^{-1} , in microaggregates (250 μm – 53 μm) 0.17 to 2.48 g kg^{-1} , silt, and clay associated fraction (< 53 μm) 0.14 to 1.89 g kg^{-1} . The carbon content of silt and clay fractions varied from 0.18 to 1.86 g kg^{-1} in northern tropical dry deciduous *Shorea robusta* forests and 0.14 to 1.76 g kg^{-1} in northern tropical dry deciduous shiwalik *Shorea robusta* forests up to 1m soil depth. In mixed dry deciduous forests, the carbon content in the silt and clay fractions varied from 0.15 to 1.89 g kg^{-1} (Table 3). The concentration of carbon in soil aggregate fractions followed the order: macroaggregates > microaggregates > silt and clay fractions in the Panchkula forest division. The carbon concentration in different size fractions at Panchkula forest division varied from 0.28 to 3.25 g kg^{-1} in dry deciduous forests, 0.09 to 4.04 g kg^{-1} at in mixed dry deciduous forests, and 0.76 to 9.96 g kg^{-1} in sub-tropical pine forests. Carbon concentration in macroaggregates (> 2mm) varied from 0.75 to 9.96 g kg^{-1} , in small macro aggregates (2mm–250 μm) 0.82– 8.09 g kg^{-1} , in microaggregates (250 μm – 53 μm) 0.13 to 3.54 g kg^{-1} , silt and clay associated fraction (< 53 μm) 0.09 to 2.99 g kg^{-1} in forest ecosystems of Panchkula forest division (Table 4)

Soil organic carbon showed the positive correlation with above ground biomass distributed within studied systems [northern tropical dry deciduous siwalik *shorea robusta* ($R^2=0.91$), northern tropical dry deciduous *shorea robusta* ($R^2= 0.90$), Mixed dry deciduous forests at Yamunanagar ($R^2=0.66$), Mixed dry deciduous forests at Panchkulla ($R^2=0.97$), Dry deciduous forests ($R^2=0.99$) and Sub-tropical pine ($R^2=0.92$) (Figure 1).

The organic carbon content in soil aggregates varied significantly among the size fractions and soil depths ($p < 0.05$). There was a significant increase in carbon concentration (g kg^{-1}) with increasing aggregate size class. Positive correlations were found to exist between total organic carbon and soil aggregate fraction carbon [> 2 mm ($R^2=0.98$), 2 mm–250 μm ($R^2=0.99$), 250 μm – 53 μm

($R^2=0.95$), and <53 μm ($R^2=0.90$)] at the soil depth of 0–15 cm at Yamunanagar (Figure 2) as well as at Panchkula forest division (Figure 3) [> 2 mm ($R^2=0.96$), 2 mm–250 μm ($R^2=0.91$), 250 μm – 53 μm ($R^2=0.99$), and <53 μm ($R^2=0.98$)].

The XRD patterns of the Mg-saturated-air-dried samples, Mg-saturated-glycerol-solvated samples, K-saturated-air-dried samples, and K-saturated-heated samples of the northern tropical plain dry deciduous *S. robusta* forests in the Yamunanagar forest division are shown in Figure 4. Smectite in clay samples can be identified by the broader peak at approximately 17.50 $\text{\AA}^\circ$ in Mg-saturated and glycerol-solvated specimens. The clay fraction is co-dominated by illite, which was identified by the presence of a 3.34 $\text{\AA}$ peak along with its higher order reflections at 9.97 $\text{\AA}$ and 4.98 $\text{\AA}$. The presence of chamosite was ascertained by the relatively sharp peaks at 7.077 $\text{\AA}^\circ$ in the glycolated sample. In heated glycolated samples, the peak of chamosite disappeared, indicating its presence in the soil. The peak at 4.25 $\text{\AA}^\circ$ showed the presence of dickite, and its stability in glycolated samples confirmed its presence at the sampled site. In the glycolated samples, the dominance of illite and mica was confirmed by the presence of peaks at 3.34 $\text{\AA}$ and 10.01 $\text{\AA}$.

The XRD patterns of the Mg-saturated, air-dried samples, Mg-saturated, glycerol-solvated samples, K-saturated, air-dried samples, and K-saturated, heated samples of the northern tropical Shiwalik dry deciduous *Shorea robusta* forests in the Yamunanagar forest division are shown in Figure 5. The clay fraction is mainly dominated by Illite-vermiculite intergrade, which was identified by the presence of a 3.34 $\text{\AA}$ peak along with its higher order reflections at 10.00 $\text{\AA}$ and 4.99 $\text{\AA}$. The peaks for vermiculite and illite nearby coincide approximately. However, the dominance of vermiculite was confirmed by a decrease (37.19% to 8.06%) in the intensity of the 10.00 $\text{\AA}^\circ$ peak after K-saturation and a slight increase (8.06% to 12.99%) in the intensity after heating at 550 $^\circ\text{C}$. The presence of kaolinite was ascertained by relatively sharper peaks at 3.56 $\text{\AA}$ along with its higher-order reflection at 7.15 $\text{\AA}$. The presence of the chlorite-vermiculite intergrade was reported by 14.11 $\text{\AA}$ reflections in the Mg-saturated clay samples, which were shifted (from 6.263 2θ position to 6.292 2θ position) towards 10.00 $\text{\AA}$ by heating at 550 $^\circ\text{C}$.

The XRD patterns of the Mg-saturated, air-dried samples, Mg-saturated, glycerol-solvated samples, K-saturated, air-dried samples, and K-saturated, heated samples of northern tropical mixed deciduous forests in the Yamunanagar forest division are shown in Figure 6. The 14.03 $\text{\AA}^\circ$ d spacing under both Mg and K saturation confirmed that it corresponds to the non-swelling type of vermiculite. The reduction in the 14 $\text{\AA}^\circ$ peak intensity reflections (from 62.03% to 51.27%) on K-saturation showed the presence of a small amount of chloritized vermiculite. Furthermore, the presence of the chlorite-vermiculite intergrade was substantiated by the shifting of the 2θ position (from 6.297 to 6.329) towards 10.00 $\text{\AA}^\circ$ K-saturated heating at 550 $^\circ\text{C}$. Illite-vermiculite intergrade was identified by the peaks at 9.95 $\text{\AA}^\circ$ with an integral series of basal reflections at 4.99 $\text{\AA}^\circ$ and 3.34 $\text{\AA}^\circ$. Along with the dominant clay minerals, the presence of some other minerals like Quartz, and Kaolinite was confirmed by the relative peaks at 4.25 $\text{\AA}$ and 7.1 $\text{\AA}$ d-spacing, respectively. The presence of kaolinite was confirmed by its internal reflection order at 3.54 $\text{\AA}^\circ$.

The XRD patterns of the Mg-saturated, air-dried samples, Mg-saturated, glycerol-solvated samples, K-saturated, air-dried samples, and K-saturated, heated samples of dry deciduous forests in the Panchkula forest division are shown in Figure 7. Smectite mixed-layer minerals can be identified by poorly defined diffraction patterns between 10.00 Å and 20.00 Å in glycol-solvated samples. The presence of chlorite-vermiculite intergrades was reported by 14.03 Å reflections in the Mg-saturated clay samples. The basal peaks at 7.1 Å indicate the presence of kaolinite, together with the subsequent peak at 3.54 Å. The presence of free quartz in the clays was evident from the 4.24 Å. The clay fraction is mainly dominated by Illite-vermiculite intergrade, which was identified by the presence of a 3.33 Å peak along with its higher order reflections at 9.95 Å and 4.99 Å.

The XRD patterns of the Mg-saturated, air-dried samples, Mg-saturated, glycerol-solvated samples, K-saturated, air-dried samples, and K-saturated, heated samples of mixed dry deciduous forests in the Panchkula forest division are shown in Figure 8. Smectite in clay samples can be identified by the broader peak at approximately 17.50 Å in Mg-saturated and glycerol-solvated specimens. The clay

fraction is co-dominated by illite, which was identified by the presence of a 3.34 Å peak along with its higher order reflections at 9.96 Å and 4.98 Å. The 14.03 Å d spacing under both Mg and K saturation confirmed that it corresponds to the non-swelling type of vermiculite. Illite and vermiculite formed an intergrade. The basal peaks at 7.1 Å indicate the presence of kaolinite, together with the subsequent peak at 3.57 Å.

The XRD patterns of the Mg-saturated, air-dried samples, Mg-saturated, glycerol-solvated samples, K-saturated, air-dried samples, and K-saturated, heated samples of subtropical pine forests in the Panchkula forest division are shown in Figure 9. The main minerals present in subtropical pine forests are illite-vermiculite intergrades, which were confirmed by the presence of a 3.34 Å peak along with higher order reflections at 9.96 Å and 4.99 Å. The 14.14 Å d spacing under both Mg and K saturation confirmed that it corresponds to the non-swelling type of vermiculite. The presence of kaolinite was ascertained by 7.1 Å accompanied by 3.58 Å d-spacing values for peaks. Smectite in clay samples can be identified by the broader peak at approximately 17.65 Å in all the treatments.

Table 1: Soil characteristics of forest ecosystems of study area

Forest Type	Soil depth (cm)	pH (1:2)	EC (µS)	Organic carbon (%)	Bulk density	Soil carbon pool (Mg ha ⁻¹)
Northern tropical dry deciduous <i>Shorea robusta</i> forest	0-15	6.27±0.06	284±10.19	0.628±0.003	1.10±0.04	10.40±0.29
	15-30	6.54±0.05	244±12.83	0.431±0.004	1.18±0.06	7.63±0.39
	30-45	6.67±0.10	192±14.71	0.136±0.003	1.22±0.05	2.48±0.11
	45-60	6.72±0.05	134±5.88	0.037±0.001	1.30±0.06	0.72±0.05
	60-100	6.82±0.09	128±5.65	0.014±0.001	1.34±0.05	0.75±0.08
Mixed dry deciduous forests (Yamuna nagar forest division)	0-15	6.99±0.07	175±2.94	0.511±0.004	1.07±0.06	8.20±0.44
	15-30	7.08±0.03	212±8.48	0.425±0.002	1.11±0.05	7.10±0.32
	30-45	7.31±0.09	361±8.73	0.092±0.003	1.19±0.05	1.64±0.18
	45-60	7.48±0.04	122±9.09	0.062±0.006	1.26±0.05	1.17±0.35
	60-100	7.52±0.05	117±7.87	0.009±0.004	1.31±0.07	0.45±0.26
Northern shiwalik dry deciduous <i>Shorea robusta</i> forest	0-15	7.83±0.07	259±9.27	0.693±0.004	1.14±0.08	11.42±0.33
	15-30	8.04±0.10	337±9.0	0.429±0.005	1.21±0.06	7.79±0.49
	30-45	8.11±0.04	250±9.09	0.141±0.004	1.27±0.10	2.68±0.22
	45-60	8.19±0.02	178±4.32	0.670±0.002	1.38±0.08	1.39±0.14
	60-100	8.24±0.05	121±8.5	0.210±0.006	1.34±0.14	1.09±0.40
Dry deciduous forests	0-15	6.72±0.08	165±4.54	0.373±0.008	1.17±0.08	6.55±0.55
	15-30	7.18±0.07	221±9.62	0.184±0.005	1.23±0.07	3.39±0.29
	30-45	7.31±0.10	331±12.83	0.032±0.005	1.28±0.04	0.61±0.09
	45-60	7.68±0.07	128±2.82	0.012±0.003	1.35±0.05	0.24±0.07
	60-100	7.54±0.11	117±17.72	0.004±0.001	1.38±0.10	0.22±0.07
Mixed dry deciduous forests (Panchkula forest division)	0-15	7.92±0.04	269±10.03	0.494±0.003	1.17±0.07	8.67±0.54
	15-30	8.14±0.08	343±10.03	0.243±0.004	1.21±0.06	4.41±0.26
	30-45	8.23±0.04	257±12.83	0.048±0.002	1.29±0.05	0.93±0.02
	45-60	8.39±0.07	181±11.57	0.014±0.003	1.33±0.04	0.27±0.06
	60-100	8.34±0.05	137±7.87	0.006±0.001	1.31±0.03	0.31±0.18
Sub tropical pine forest	0-15	5.89±0.03	258±10.98	1.011±0.006	1.21±0.06	18.35±1.08
	15-30	5.99±0.05	237±10.80	0.307±0.002	1.26±0.09	5.80±0.49
	30-45	6.12±0.08	201±12.96	0.091±0.003	1.29±0.09	1.76±0.09
	45-60	6.21±0.08	198±14.44	0.034±0.005	1.32±0.10	0.67±0.15
	60-100	6.37±0.11	178±7.87	0.018±0.006	1.33±0.06	0.95±0.26

Table 2: Organic carbon concentrations (g kg⁻¹) in soil at different depths up to 100cm at various forest types of Yamunanagar and Panchkula forest division

Soil depth (cm)	Soil carbon (g kg ⁻¹)					
	Yn-SRP	Yn-SSR	Yn-MDDF	Pk-DDF	Pk-MDDF	Pk-STP
0 – 15	6.28±0.03	6.93±0.27	5.11±0.07	3.73±0.10	4.94±0.04	10.11±0.07
15 – 30	4.31±0.07	4.29±0.04	4.25±0.04	1.84±0.10	2.43±0.03	3.07±0.04
30 – 45	1.36±0.09	1.41±0.03	0.92±0.05	0.32±0.07	0.48±0.04	0.91±0.09
45 – 60	0.37±0.08	0.67±0.09	0.62±0.10	0.12±0.02	0.14±0.01	0.34±0.06
60 – 100	0.14±0.04	0.21±0.02	0.09±0.04	0.04±0.01	0.06±0.01	0.18±0.04
CV (%)	5.764	9.686	5.838	12.53	4.018	4.607
LSD (p>0.05)	0.216	0.387	0.192	0.228	0.097	0.202

Northern tropical dry deciduous *Shorea robusta* forest (Yn-SRP), Northern tropical dry deciduous Shiwalik *Shorea robusta* forest (Yn-SSR), and Mixed dry deciduous forest ecosystems (Yn-MDDF) at Yamunanagar and Dry deciduous forest ecosystems (Pk-DDF), Mixed dry deciduous forest ecosystems (Pk-MDDF), and Subtropical pine forests (Pk-STP) Panchkula forest division

Table 3: Soil organic carbon (g kg⁻¹) distribution in aggregate size classes at different soil depths of Yamunanagar forest division

Soil depth (cm)	Soil aggregate carbon (g kg ⁻¹)				
	Soil carbon (g kg ⁻¹)	>2mm	2 mm – 250 µm	250µm – 53 µm	<53µm
Northern tropical dry deciduous <i>Shorea robusta</i> forest					
0 – 15	6.28±0.03	4.02±0.02	4.82±0.02	2.12±0.02	1.86±0.01
15 – 30	4.31±0.07	2.25±0.01	2.28±0.01	1.99±0.03	1.85±0.01
30 – 45	1.36±0.09	1.29±0.03	1.49±0.02	1.18±0.01	0.97±0.02
45 – 60	0.37±0.08	0.78±0.07	0.89±0.02	0.49±0.02	0.44±0.01
60 – 100	0.14±0.04	0.39±0.02	0.49±0.02	0.28±0.02	0.18±0.03
CV (%)	5.764	3.015	2.422	3.790	3.758
LSD (p>0.05)	0.216	0.079	0.072	0.069	0.060
Northern tropical dry deciduous shiwalik <i>Shorea robusta</i> forest					
0 – 15	6.93±0.27	3.31±0.07	4.20±0.08	2.21±0.05	1.76±0.03
15 – 30	4.29±0.04	1.89±0.09	1.79±0.06	1.78±0.06	1.37±0.07
30 – 45	1.41±0.03	1.19±0.03	1.13±0.03	0.82±0.06	0.78±0.07
45 – 60	0.67±0.09	0.76±0.06	0.69±0.03	0.36±0.08	0.28±0.06
60 – 100	0.21±0.02	0.43±0.08	0.32±0.06	0.17±0.07	0.14±0.05
CV (%)	9.686	10.09	18.01	7.98	6.45
LSD (p>0.05)	0.387	0.221	0.347	0.172	0.167
Mixed dry deciduous forests					
0 – 15	5.11±0.07	4.31±0.05	5.22±0.05	2.48±0.04	1.89±0.03
15 – 30	4.25±0.04	2.01±0.06	2.85±0.04	2.39±0.03	1.41±0.06
30 – 45	0.92±0.05	1.49±0.07	1.40±0.08	1.17±0.09	1.03±0.05
45 – 60	0.62±0.10	0.69±0.09	0.76±0.05	0.39±0.04	0.31±0.04
60 – 100	0.09±0.04	0.41±0.05	0.48±0.09	0.32±0.05	0.15±0.04
CV (%)	5.838	14.25	19.15	17.45	20.47
LSD (p>0.05)	0.192	0.355	0.570	0.414	0.341

Table 4: Soil organic carbon (g kg⁻¹) distribution in aggregate size classes at different soil depths of Panchkula forest division

Soil depth (cm)	Soil aggregate carbon (g kg ⁻¹)				
	Soil carbon (g kg ⁻¹)	>2mm	2 mm–250 µm	250µm–53 µm	<53µm
Dry deciduous forests					
0 – 15	3.73±0.10	3.25±0.11	2.43±0.12	1.31±0.17	1.10±0.08
15 – 30	1.84±0.10	2.48±0.12	2.29±0.18	1.16±0.17	0.92±0.20
30 – 45	0.32±0.07	1.76±0.16	1.81±0.25	0.10±0.02	0.07±0.01
45 – 60	0.12±0.02	0.92±0.10	1.91±0.08	0.56±0.07	0.23±0.05
60 – 100	0.04±0.01	0.75±0.03	0.82±0.14	0.47±0.15	0.28±0.05
CV (%)	12.53	12.15	18.15	17.23	20.417
LSD (p>0.05)	0.228	0.335	0.506	0.404	0.313
Mixed dry deciduous forests					
0 – 15	4.94±0.04	4.04±0.13	3.09±0.07	1.48±0.06	1.21±0.07
15 – 30	2.43±0.03	1.87±0.15	2.92±0.09	1.58±0.11	1.14±0.11
30 – 45	0.48±0.04	2.21±0.12	2.13±0.04	0.88±0.10	0.39±0.08
45 – 60	0.14±0.01	1.11±0.12	1.42±0.11	0.59±0.10	0.29±0.07
60 – 100	0.06±0.01	1.03±0.18	1.07±0.21	0.13±0.04	0.09±0.01
CV (%)	4.018	14.16	11.95	20.64	12.78
LSD (p>0.05)	0.097	0.436	0.382	0.272	0.231
Subtropical pine forests					
0 – 15	10.11±0.07	9.96±0.04	8.09±0.06	3.54±0.05	2.99±0.06
15 – 30	3.07±0.04	6.01±0.07	6.32±0.02	3.05±0.02	2.67±0.05
30 – 45	0.91±0.09	4.71±0.05	5.01±0.07	0.29±0.08	0.22±0.03
45 – 60	0.34±0.06	2.47±0.06	2.69±0.05	2.83±0.06	2.78±0.02
60 – 100	0.18±0.04	2.07±0.07	2.29±0.05	1.19±0.02	0.76±0.06
CV (%)	4.607	2.532	2.328	5.107	5.762
LSD (p>0.05)	0.202	0.192	0.171	0.167	0.163

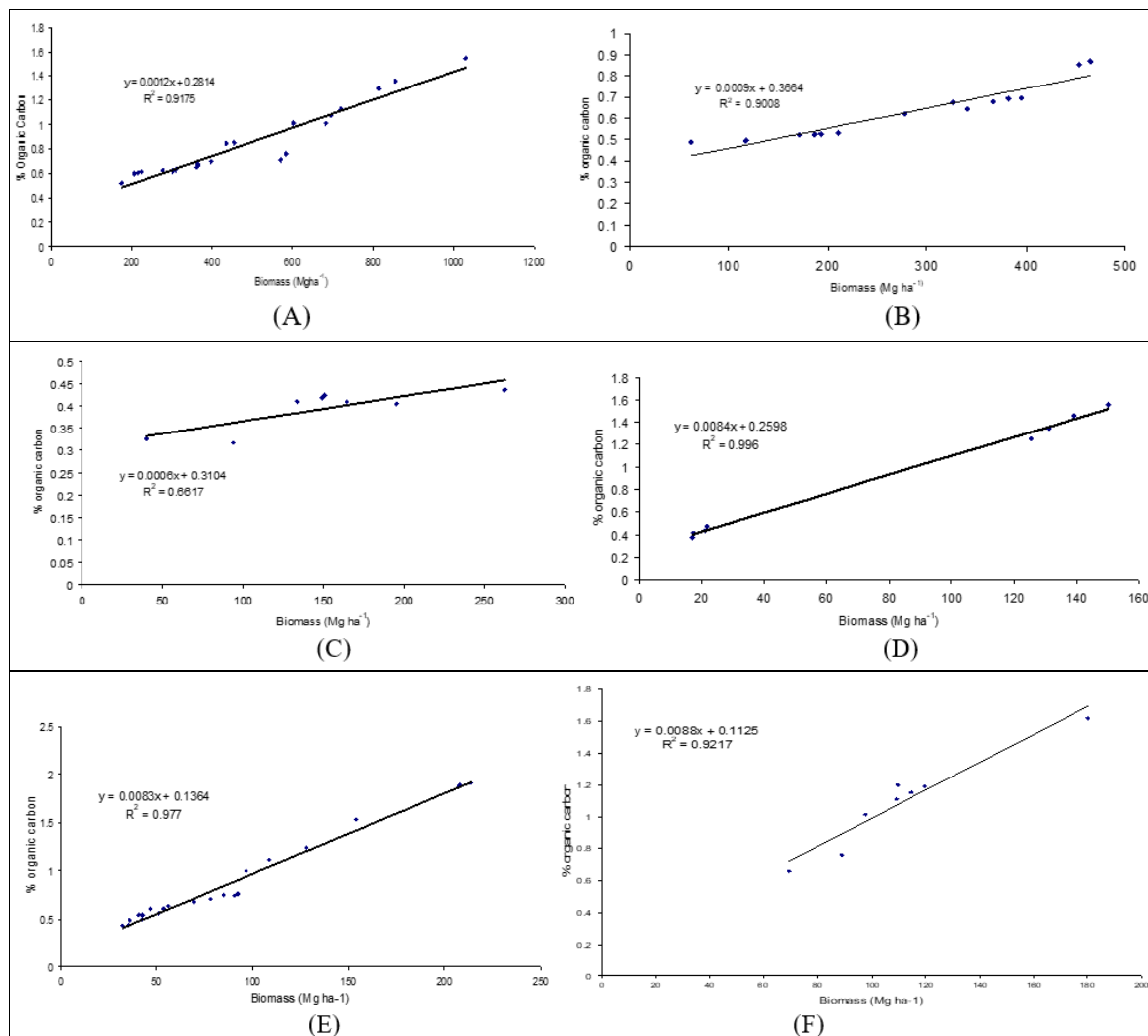
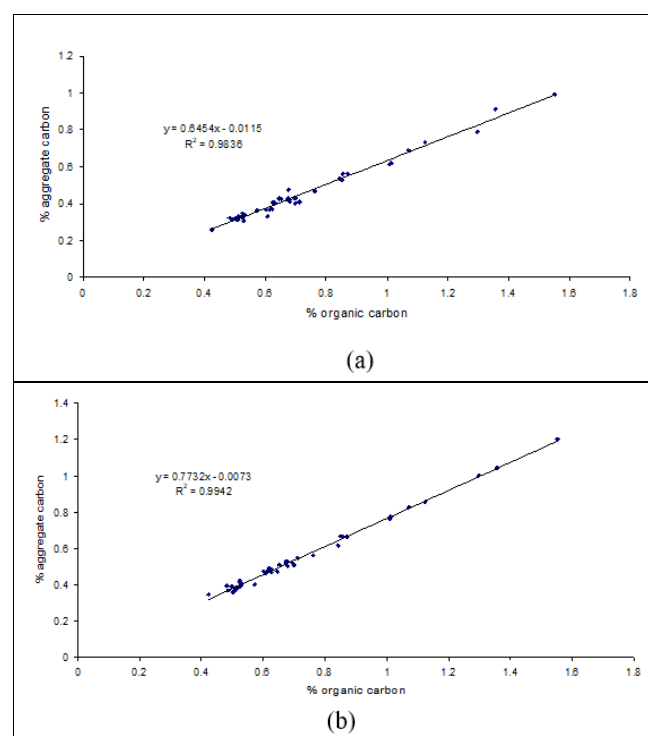


Fig 1: Relationship between total organic carbon (0-15cm) of soil (%) and above ground biomass (Mg ha⁻¹) (A) Northern tropical dry deciduous *Shorea robusta* forests, (B) Northern tropical dry deciduous shiwalik *shorea robusta* forest, (C) Mixed dry deciduous forest at Yamunanagar forest division, (D) Dry deciduous forest, (E) Mixed dry deciduous forests, (F) Sub-tropical pine forest at Panchkula forest division



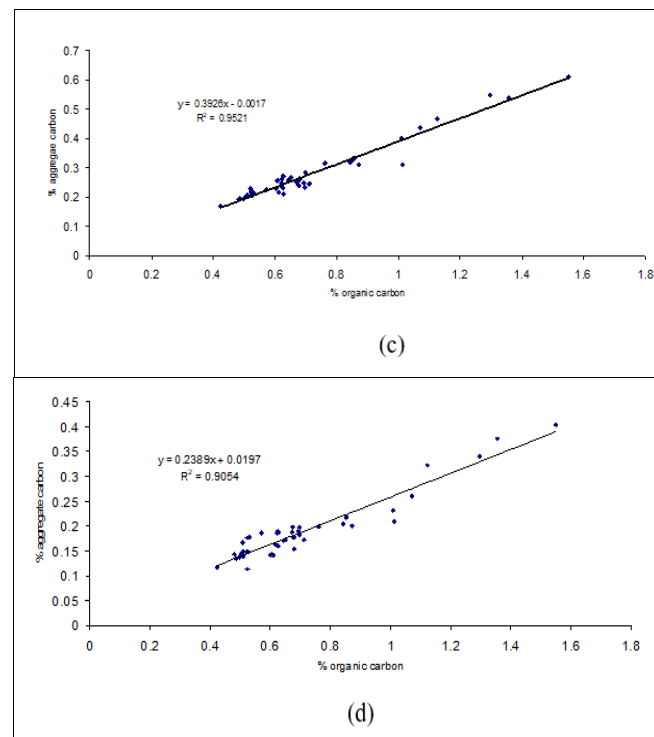
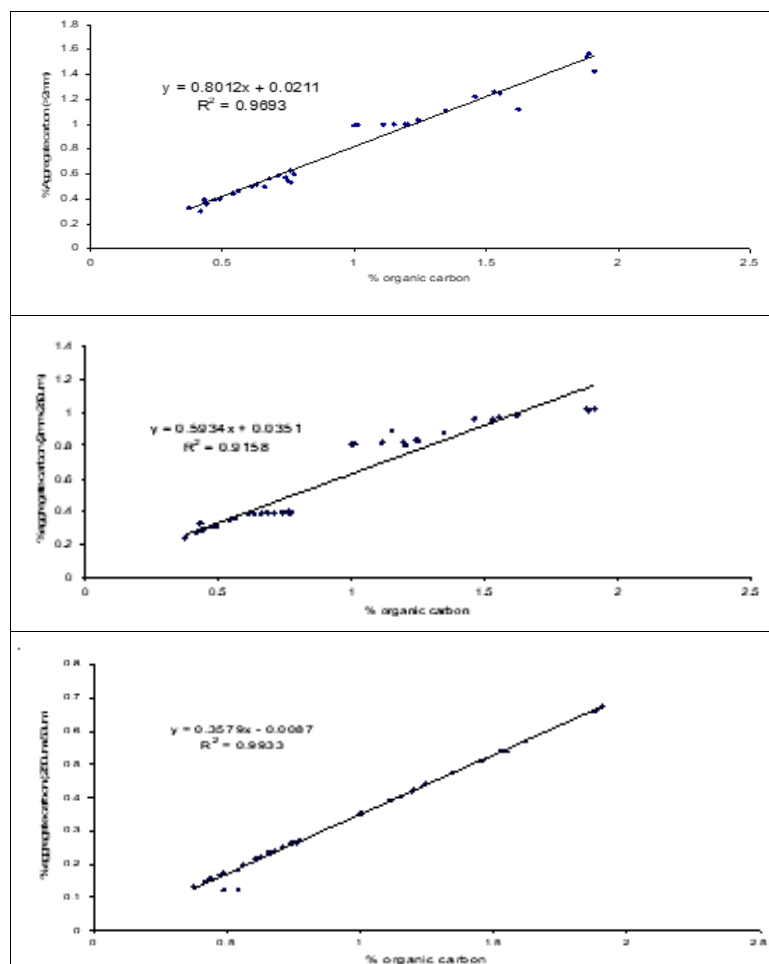


Fig 2: Relationship between total organic carbon of soil and aggregate carbon at 0-15cm depth (a) >2mm (b) 2mm-250um (c) 250um-53um (d) <53um at Yamunanagar forest division



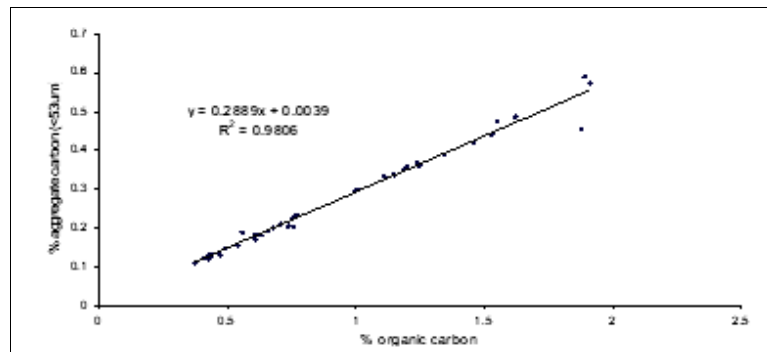


Fig 3: Relationship between total organic carbon of soil and aggregate carbon at 0-15cm depth (a) >2mm (b) 2mm-250um (c) 250um-53um (d) <53um at Panchkula forest division

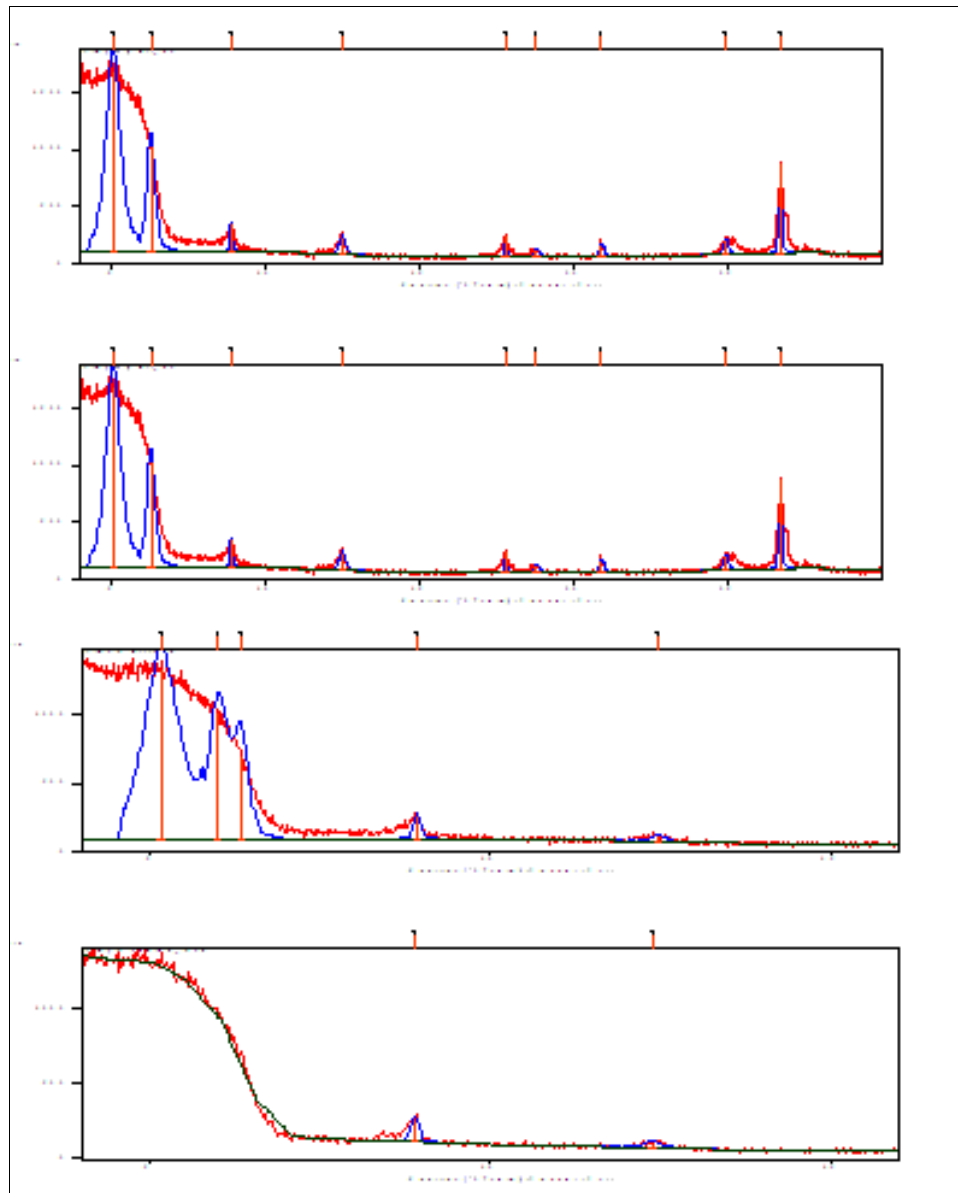


Fig 4: X-ray diffraction pattern of (A) Mg-air saturated, (B) Mg- Glycolated, (C) K-air saturated, (D) K-heated clay samples of northern tropical dry deciduous *Shorea robusta* forests of Yamunanagar forest division (I-Vm = Illite-vermiculite intergrade; CI-Vm = Chlorite-vermiculite intergrade; Ch= Chamosite); Kaolinite =K).

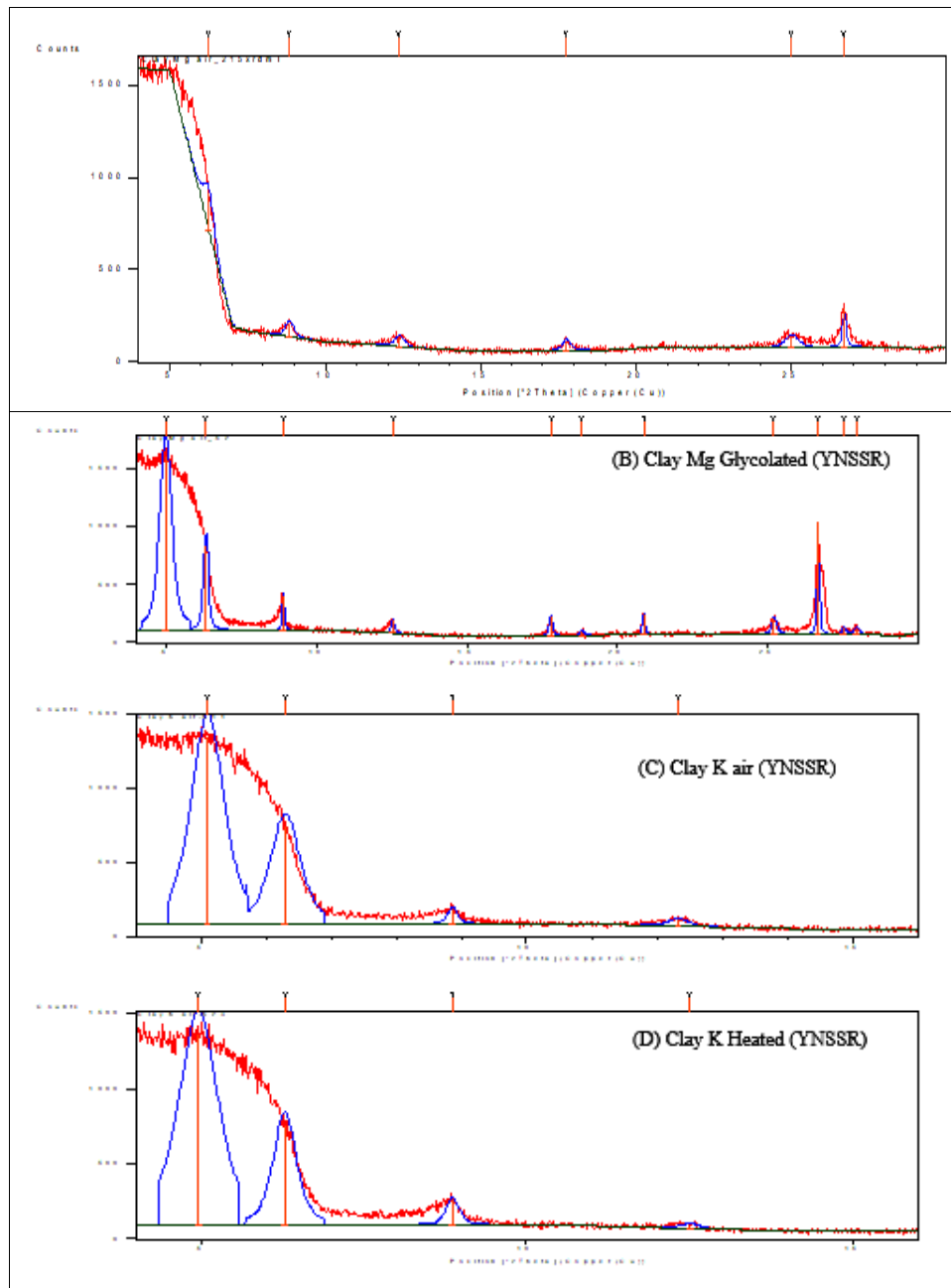
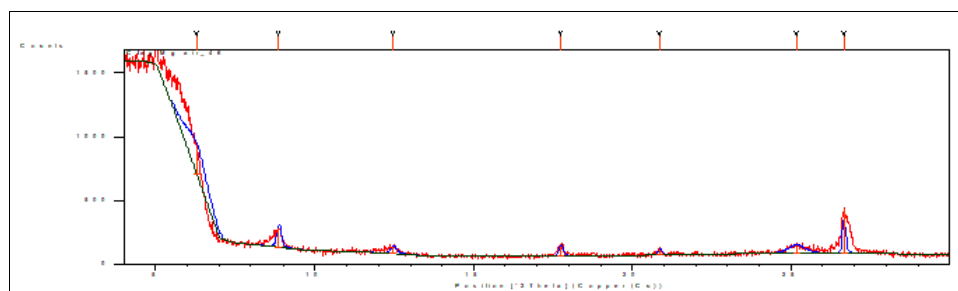


Fig 5: X-ray diffraction pattern of (A) Mg-air saturated, (B) Mg- Glycolated, (C) K-air saturated, (D) K-heated clay samples of northern tropical dry deciduous shiwalik *Shorea robusta* forests of Yamunanagar forest division (I-Vm = Illite-vermiculite intergrade; CI-Vm = Chalorite-vermiculite intergrade; Ch= Chamosite; Kaolinite =K; Sm = Smectite).



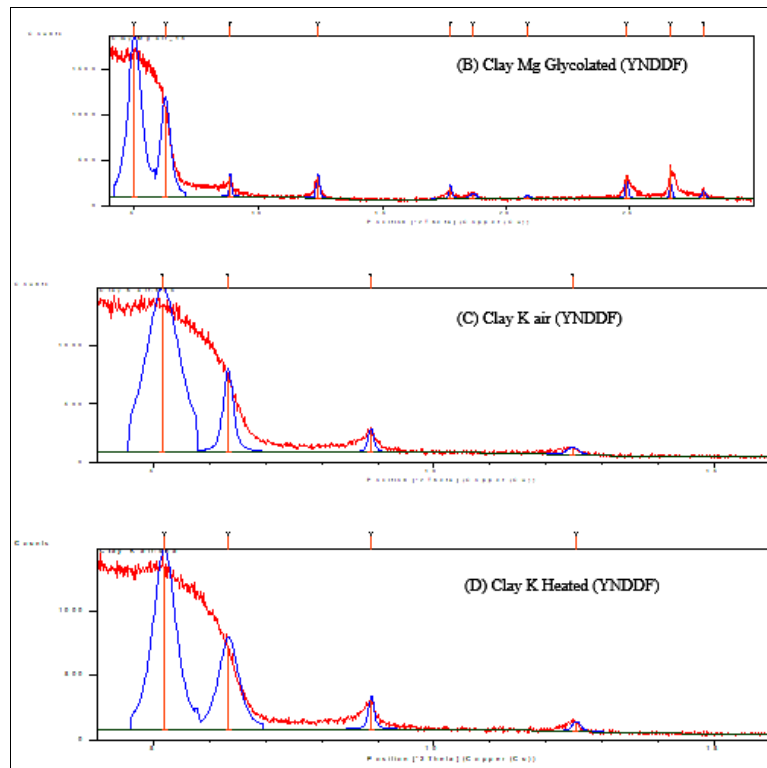
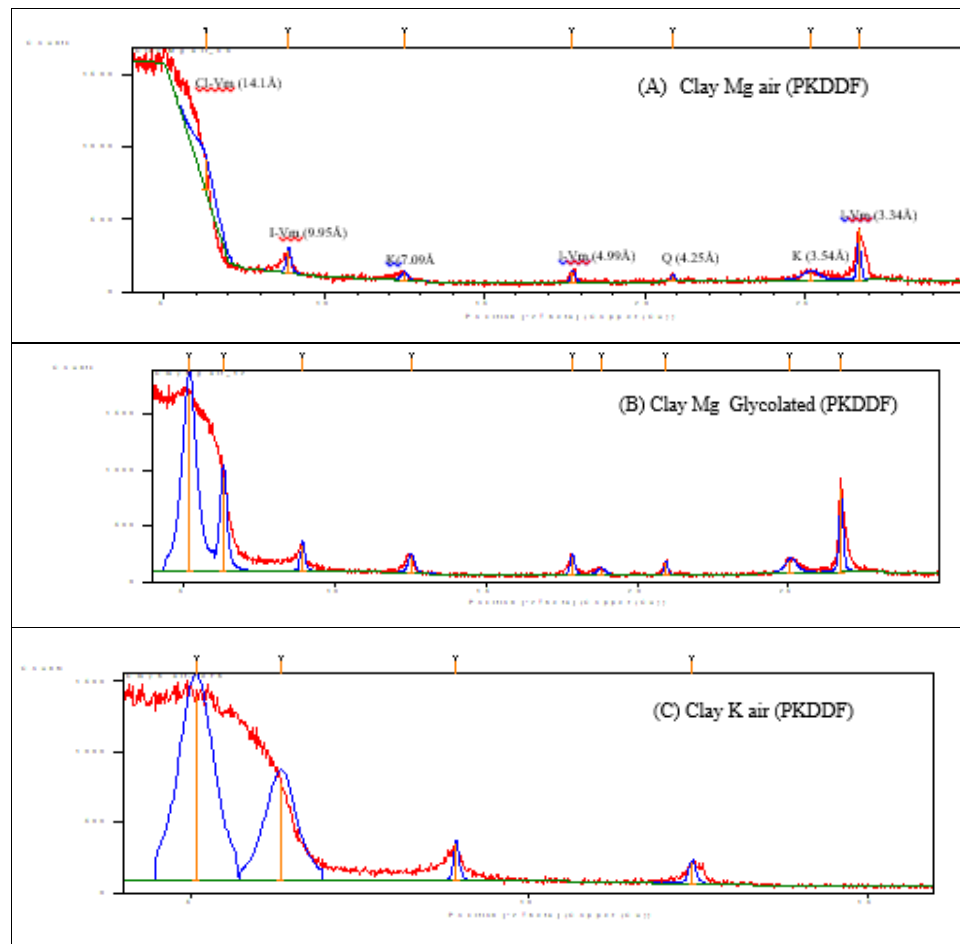


Fig 6: X-ray diffraction pattern of (A) Mg-air saturated, (B) Mg- Glycolated, (C) K-air saturated, (D) K-heated clay samples of mixed dry deciduous forests of Yamunanagar forest division (I-Vm = Illite-vermiculite intergrade; CI-Vm = Chalarite-vermiculite intergrade; Ch= Chasmosite; Kaolinite =K; Sm = Smectite).



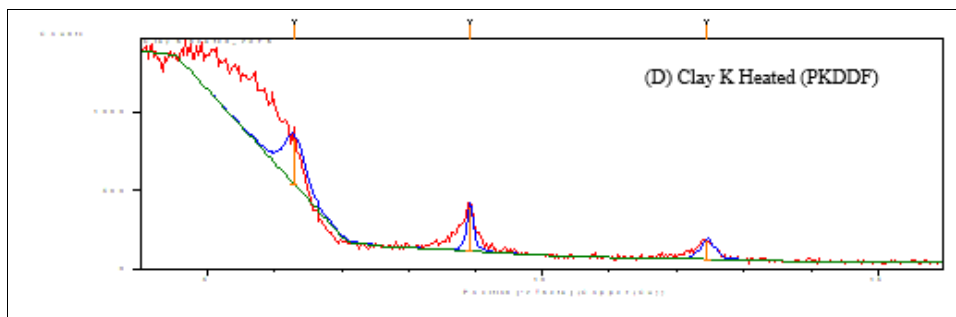


Fig 7: X-ray diffraction pattern of (A) Mg-air saturated, (B) Mg- Glycolated, (C) K-air saturated, (D) K-heated clay samples of dry deciduous forests of Panchkula forest division (I-Vm = Illite-vermiculite intergrade; CI-Vm = Chalarite-vermiculite intergrade; Ch= Chasmosite; Kaolinite =K; Sm = Smectite).

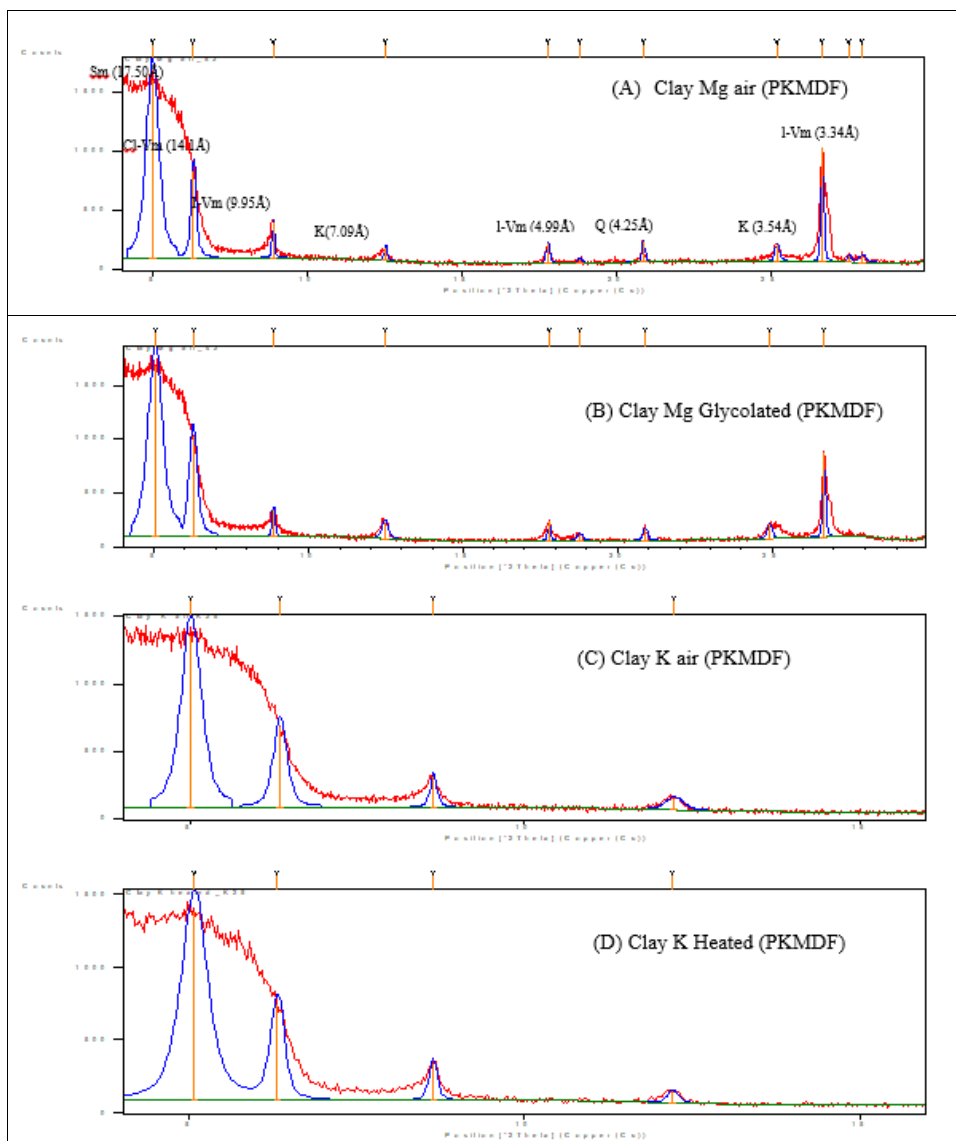


Fig 8: X-ray diffraction pattern of (A) Mg-air saturated, (B) Mg- Glycolated, (C) K-air saturated, (D) K-heated clay samples of mixed dry deciduous forests at Panchkula forest division (I-Vm = Illite-vermiculite intergrade; CI-Vm = Chalarite-vermiculite intergrade; Ch= Chasmosite; Kaolinite =K; Sm = Smectite).

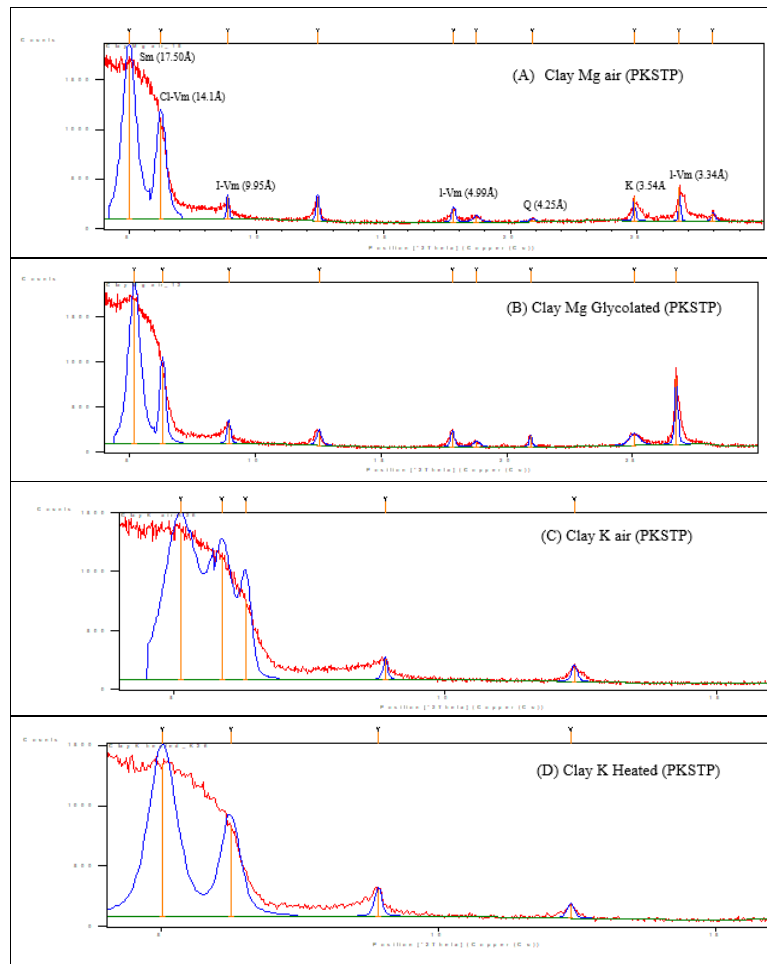


Fig 9: X-ray diffraction pattern of (A) Mg-air saturated, (B) Mg- Glycolated, (C) K-air saturated, (D) K-heated clay samples of Sub tropical pine forests of Panchkula forest division (I-Vm = Illite-vermiculite intergrade; CI-Vm = Chalorite-vermiculite intergrade; Ch= Chasmosite; Kaolinite =K; Sm = Smectite).

Discussion

Knowledge of minerals and their transformations helps in understanding the distribution of forest types and their biogeochemistry. Minerals, specifically clay minerals, endow soils with unique surface charge chemistry and reactivity. This provides chemical homeostasis to terrestrial biogeochemical cycles, eventually influencing forest growth and regeneration. Comprehension of mineralogy is fundamental to both the conventional approach of proper understanding and the assessment of weathering, pedogenesis, soil management, and potentiality for different uses and terrestrial acidification load assessment of ecosystems and global climate change. For the reasons mentioned earlier, the basic properties of soils and the importance of specialized silviculture operations for sustainable forest management necessitate accurate identification, characterization, and knowledge of the soil mineralogical constitution. X-ray powder diffraction is the most common technique used to study the characteristics of crystalline structures and to determine the mineralogy of finer-grained sediments, especially clays. X-ray diffraction analysis is a useful method for identifying and making semi-quantitative estimates of the crystalline mineral components of soil. The angles of diffraction, which are affected by the different sample treatments, are distinctive for a particular mineral and help to identify that mineral. The intensities of the diffraction maxima are related to the number of corresponding diffraction planes in a sample and provide a basis for estimating the concentrations of the mineral species present.

The presence of smectite and vermiculite-illite intergrade indicates that the parent material has not yet been fully weathered and that soil formation has not yet reached maturity (Sahu and Dash 1991). Clays are aluminosilicates that carry a negative charge. It acts as a binding agent between the soil particles. It provides soil elasticity and cohesion of soil particles. The result is a network of capillary pores that hold moisture against the force of gravity. They contribute many benefits to the physical, chemical, and biological properties of soil. It increases (i) soil cation exchange capacity, (ii) acts as a binding agent for the non-clay components, (iii) provides elasticity, (iv) reduces nutrient loss through leaching, and (v) enhances soil buffering capacity (resistance to change in pH) (NCDA & CS Agronomic Division 1999). XRD analysis is a valuable tool for gaining insight into the interactions between clay minerals and organic matter (Pereira *et al.* 2008). Clay mineral phases identified by the decomposition of XRD patterns by direct measurement of d-spacing provided a realistic analysis of the clay phase present in complex soil samples (Right and Elsass 1996; Siddiqui *et al.* 2009). The direction of the d-spacing shift and its magnitude depend on the nature of the crystal surface (Tettenhorst and Roberson 1973).

The union of clay minerals and organic substances in soil to form clay-organic complexes is a synthesis vital to the continuance of life and is less understood than photosynthesis (Manjaiah *et al.* 2010). Understanding the mechanisms of formation and properties of clay organic complexes may facilitate the development of forest

management systems that increase long-term carbon stability and sequestration in soils. Soil minerals are known to play a stabilizing role in organic matter and the behavior of clayey soils. Organic matter in soil is partially associated with minerals. This association renders soil organic matter less susceptible to biodegradation than when it is free of metals. Therefore, these associations are believed to be controlling factors for carbon storage in the soil (Baldock and Skjemstad 2000). Minerals are naturally occurring homogeneous inorganic solids with a definite chemical composition and specific repeating arrangements of atoms. The most common minerals found in soils are silicates, including quartz, feldspar, and mica. The primary silicate mineral composition depends on the composition of the parent material and the extent of weathering. Secondary silicates, such as kaolinite and smectite, are formed due to the weathering of primary silicates and are the most important in the clay fraction. The mineralogy of the clay samples from various forest ecosystems in the Yamunanagar and Panchkula forest divisions, as interpreted by XRD patterns, showed that the clay mineral assemblage was dominated by illite-vermiculite intergrade, smectite accompanied by interstratified Quartz, Illite, and Kaolinite in varying concentrations, and negligible proportions of chlorite, muscovite, and chamosite. Clay of all the studied systems, representing the alluvium, is a mixture of identical clay mineral suites- vermiculite, illite, kaolinite, and mixed layer minerals with a very small proportion of accessory non-clay free quartz. No major differences in the peak intensities of the diffractograms showed a uniform distribution of clay minerals throughout all the forest ecosystems of the Siwalik region. This might be due to the almost uniform climatic conditions and similar parent material in the study area.

The transformation and alteration of minerals into other minerals is assumed to have followed the sequence of mica → illite → mixed layer minerals → vermiculite → kaolinite, under a comparatively wet moisture regime (Eswaran and Bin 1978). Although the effects of vegetation on the alteration and neo-synthesis of minerals are difficult to separate from those of the climate, the presence of high organic matter and high organic acids (Singhal *et al.* 1976; Sharma 1982; Singhal and Sharma 1983), wet moisture regime, rich floristic diversity, and root biomass suggest that natural vegetation has a definite role in defining the clay mineralogy of forested regions. The natural vegetation in the studied forests seems to play an important role in the alteration of quartz and neo-synthesis of vermiculite and/or kaolinite. The humid conditions during the rainy season and prolonged dry periods during summer and winter in the studied area are the major factors influencing the clay mineralogy of the forested areas of the Shiwaliks. Thus, soil mineralogy and its spatial variability can lead to a better understanding of how minerals regulate soil carbon stability.

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