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Mineralogy of kaolin clays in different forest ecosystems of southern Western Ghats, India

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Random X-ray powder diffraction (XRD) and scanning electron microscopy (SEM) were used to identify 1 : 1 clay minerals in soils of five different forest ecosystems such as moist deciduous forests, evergreen forests, shola forests, grasslands and scrub jungles in the southern Western Ghats, India. The study sites experience a humid tropical climate with intense leaching and weathering, except scrub jungle which lies in the rain shadow area of the Western Ghats. XRD analyses of air-dried samples, confirmatory tests using formamide intercalation and SEM could establish kaolinite–halloysite coexistence in clay fractions of three different ecosystems of the Western Ghats. Earlier studies on clay mineralogy in the region failed to establish such coexistence because of the relative metastable nature of halloysite with respect to kaolinite. The identification of soil systems with metastable minerals like halloysite presents interesting possibilities of further studies vis-à-vis soil genesis and management in the tropics.

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Keywords: Forest ecosystems, halloysite, kaolinite, scanning electron microscopy, X-ray diffraction.

SOILS of the humid tropical region undergo intense weathering due to the prevailing conditions of high rainfall and temperature. Conditions of such intense leaching provide a favourable environment for development of kaolin clays, a general group covering kaolinite, halloysite and intermediate forms along with specific minerals such as dickite and nacrite[1]. In geological environments kaolin minerals are formed in a number of different ways such as direct crystallization from solution, replacement, crystallization from colloidal gels and weathering of layer or non-layer silicates[2].

Halloysite and kaolinite are the most frequent and abundant kaolin minerals in tropical regions, where intense drainage and hot climate induce a monosialitization process[3]. These two minerals are formed together or independently as weathering products of alumino-silicates depending on geologic conditions such as climate, topography, degree of leaching, etc. or geochemical conditions like activities of Al, H_4SiO_4, Na^+, K^+ and H^+. Structural modelling and microscopic observations reveal that transformations between the two minerals after their formation lead to a decrease in the relative abundance of halloysite with respect to kaolinite[4]. The rate of transformation of halloysite to kaolinite can be explained in terms of Gibbs free energy and activation energy:

The Gibbs free energy of formation (ΔG_f^0) for halloysite and kaolinite is –898.4 and –902.9 kcal mol^{-1} respectively[5]. Assuming a chemical composition of $Al_2Si_2O_5(OH)_4$ for both the minerals, a transformation reaction of halloysite to kaolinite can be depicted as follows

$$\underset{\text{(Halloysite)}}{Al_2Si_2O_5(OH)_4} \leftrightarrow \underset{\text{(Kaolinite)}}{Al_2Si_2O_5(OH)_4}$$

ΔG_r^0 (Gibbs free energy of reaction)
$= \sum\Delta G_f^0$ (products) $- \sum\Delta G_f^0$ (reactants)
$= \sum\Delta G_f^0$ (Gibbs free energy of formation of kaolinites) $- \sum\Delta G_f^0$ (Gibbs free energy of formation of halloysites)
$= -902.9 - (-898.4)$
$= -4.5$ kcal.

Since $\Delta G_r^0 < 0$, the forward reaction will proceed spontaneously promoting kaolinite formation. From a solution chemistry point of view, a supersaturated solution of alumino-silicates with molar Gibbs free energy greater than both minerals will precipitate halloysite or kaolinite, whereas a solution saturated with kaolinite will crystallize only the latter mineral (Figure 1).

However, the question remains as to how long this transformation takes. This being a kinetics problem is best described by activation energy (ΔH) rather than Gibbs free energy alone. The magnitude of ΔH depends on reaction mechanism and temperature is a rate-determining factor in any such mechanism. Thermodynamic and kinetic simulation studies suggest that halloysite grains are metastable with respect to kaolinite and are replaced by the latter mineral with progressive weathering[4]. Any natural ecosystem in the humid tropics (intense weathering conditions) that obstructs this transformation presents interesting possibilities of in-depth studies.

Early works on mineralogy of soils in the region[6–9] have indicated the presence of kaolinite, goethite and quartz along with some 2 : 1 clays as the dominant minerals, but failed to detect any halloysites. However, these works largely concentrated on agricultural systems and no attempts have been made till date to understand soil mineralogical make-up of different forest types in the region, leading to omissions of some important minerals. To overcome this deficiency, the present study focused on forest soils and made a preliminary attempt to identify systems with kaolinite–halloysite coexistence, the two major kaolin minerals, in the hot humid tropics of the southern Western Ghats. The detection of such metastable minerals will provide good inputs for models of soil genesis in the tropics.

The southern Western Ghats is a part of the South Indian Precambrian terrain dominated by low-grade metamorphic and gneissic rocks. Five different forest ecosystems representing diverse climate, altitude and vegetation mix were selected in the Kerala part of the Western Ghats (southern Western Ghats) to study their soil kaolin mineralogy (Figure 2). The general characters of the selected ecosystems are:

1. Moist deciduous forests: Deciduous mixed vegetation which sheds leaves and opens canopy cover during January–April. Located at 62 m amsl with moderate to steep slopes. The area receives >2000 mm annual rainfall. Soil temperature and moisture regime of the study region are isohyperthermic and ustic respectively (soil temperature and moisture moisture regime classification according to the Soil Survey Staff, 1975 protocols[10]).
2. Evergreen forest: Study site located at 600 m amsl. It receives >2000 mm annual rainfall and supports a closed canopy and thick undergrowth. Soil temperature regime is isohyperthermic and moisture regime ustic.
3. Shola forest: Unique montane vegetation occupying temperate habitats in tropical latitudes. Study site is located at an elevation of 1920 m amsl and receives an annual rainfall between 2000 and 3000 mm. Air temperature in the coldest month goes up to 5–6°C and the area experiences a dry period for 4–5 months (December–April).
4. Scrub jungle: Lying in the rain shadow region of the Western Ghats, scrub jungles support thorny vegetation. The study site is located at 800 m amsl with

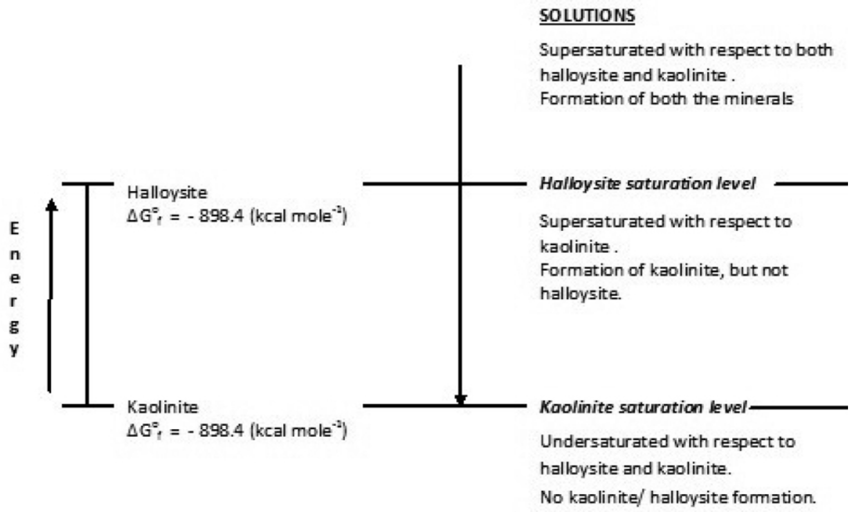


Figure 1. Relative thermodynamic stability of kaolinite and halloysite in soil systems.

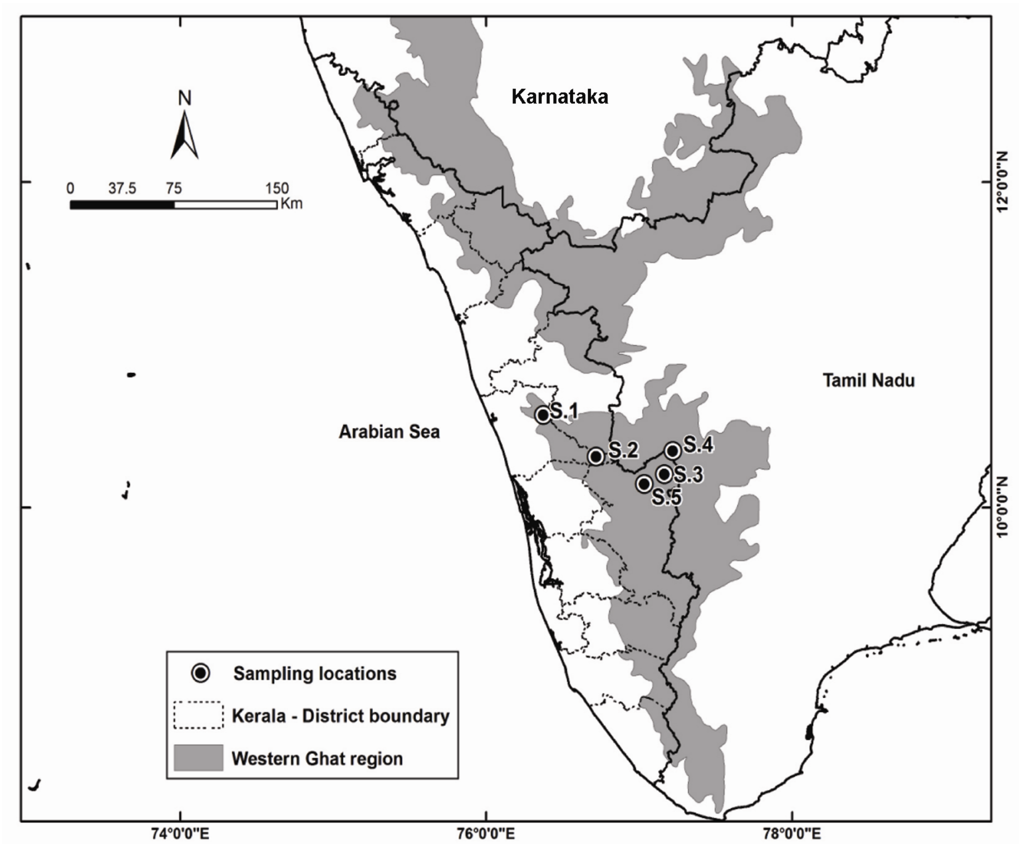


Figure 2. Map showing sampling locations in the Southern Western Ghats. S.1, Moist deciduous forest; S.2, Evergreen forest; S.3, Shola forest; S.4, Scrub jungle; S.5, Grassland.

thermic soil temperature and ustic soil moisture regimes. It receives an annual rainfall of <500 mm.

5. Grassland: Montane grasslands with predominance of grasses mixed with herbaceous and shrubby species. The study site lies at an altitude of 2120 m amsl, receives an annual rainfall of >2000 mm and has an isohyperthermic soil temperature and ustic soil moisture regime.

Composite soil samples (0–30 cm depth) were collected from the selected ecosystems. These samples were air-dried, sieved and subjected to physico-chemical analyses.

Table 1. Selected physical and chemical properties of soil

Ecosystem	Particle-size (USDA) distribution (% of <2 mm)			Textural class	pH (1 : 2.5)		Organic carbon (%)	CEC NH_4OAc (cmol (p+) kg^{-1})	Base saturation (NH_4OAc) (%)
	Sand	Silt	Clay		H_2O	KCl			
Moist deciduous forest	78	10	12	Loamy sand	5.6	5.4	2.2	17.5	49.9
Evergreen forest	81	11	8	Loamy	5.1	4.8	1.8	13.1	32.4
Shola forest	92	4	4	Sandy	5.2	4.4	5.7	21.7	52.7
Scrub jungle	90	5	5	Sandy	6.5	5.4	1.1	20.1	40.9
Grassland	86	9	5	Sandy	5.4	5.1	5.7	24.8	50.2

Particle size distribution was determined by Bouyoucos method after removal of organic carbon and free iron oxides[11]. Next pH, organic carbon, cation exchange capacity (CEC) and extractable bases were determined on the total fine earth fraction (<2 mm) following standard methods[12].

Mineralogical analyses of the soils were done after treatment with hydrogen peroxide and sodium citrate–bicarbonate–dithionite to remove organic matter and iron oxides respectively[13]. Total clay (<0.002 mm) was extracted from the soils by centrifugation and oriented clay fractions were subjected to X-ray diffraction (XRD) analysis using a Philips diffractometer with Ni-filtered, Cu-Kα radiation at a scanning speed of 2° 2θ/min. Formamide intercalation test was carried out and clay samples were scanned in the diffractometer – 1 h, 4 h and 10 days after treatment to differentiate between halloysite and kaolinite[14]. Scanning electron microscopic (SEM) analyses were conducted after mounting clay particles on an aluminum stub with LEIT-C conductive carbon cement, coated with gold and examined with a FEI QUANTA 200 SEM machine.

Crystallinity of 1 : 1 minerals was assessed from relative crystallinity and crystallinity index (CI). Relative crystallinity of minerals was evaluated from the intensity of 0.70 nm peak as follows

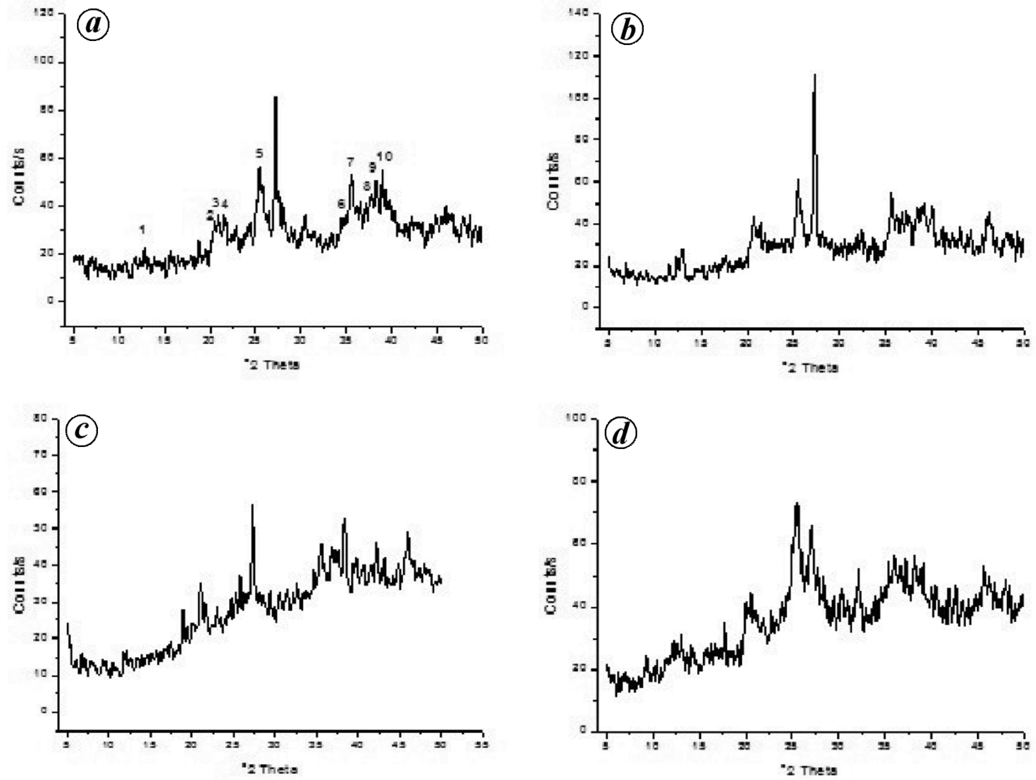
$$\text{Relative crystallinity} = \frac{\text{Peak height (0.70 nm peak)}}{\text{Width at half peak height}}.$$

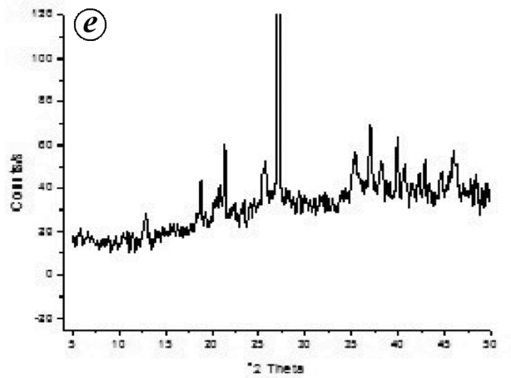
CI was calculated by Hinckley's method. This index is defined as the ratio of sum of heights of the reflections (110) and (111) measured from inter-peak background, and height of (110) peak measured from the general background. Hinckley's crystallinity index is a dimensionless number varying between 0.2 and 1.5, with larger values indicating greater crystallinity.

In general, the soils were found to be acidic (pH (1 : 2.5 :: soil : H_2O) < 5.5) due to high rainfall and leaching of bases common in humid tropical conditions. However, scrub jungle lying in the rain shadow regions of the Western Ghats had nearly neutral soil reaction (pH (1 : 2.5 :: soil : H_2O) = 6.5). Shola forests, scrub jungle and grassland systems had sand fractions approximating 90% and retained appreciable amounts of weatherable minerals in them (data not shown). The ΔpH values (ΔpH = pH (KCl) – pH (H_2O)) were negative in all the soils, indicating the presence of variable charge minerals like gibbsite or sesquioxides[15]. The CEC values ranged from 13.1 to 24.8 cmol (p+) kg^{-1} and organic carbon content from 1.1% in scrub jungle to 5.7% in grasslands and shola forests (Table 1).

XRD patterns of oriented samples of total clays showed prominent 0.7 nm peaks indicating the presence of kaolinite/halloysite in all the systems (Figure 3). Formamide intercalation-induced peak shifts from 0.70 nm to 0.80–1.0 nm (001 diffraction shift) within 1 h of treatment were taken as confirmation for presence of halloysite in the samples. As such, kaolinite–halloysite coexistence was found in three of the five ecosystems: shola forest, scrub jungle and grassland systems (Figure 4). Moist deciduous and evergreen forests indicated the presence of disordered kaolinites, but no halloysites.

Diffractograms of clay fractions from shola forests exhibited well-defined XRD maxima at 1.02 and 0.52 nm, 1 h after intercalation with formamide, corresponding to 001 and 002 basal diffraction peaks of hydrated halloysites. A diffusion of XRD peaks between 0.7 and 1.0 nm was observed 4 h after formamide treatment, indicating structural degradation of halloysites similar to that described elsewhere[16]. Reinforcement of 1.0 nm peaks after 10 days of formamide treatment indicates kaolinite in the samples as more energy is apparently required for interlayer penetration of this mineral. Previous studies on kaolin minerals show that the energy requirement for intercalation of formamide varies as hydrated halloysite < dehydrated halloysite < kaolinite. SEM analyses of the clay samples further confirmed the presence of tubular halloysites and platy kaolinites in soils of this system (Figure 5). Shola forest lying at an altitude of 1600–2100 m amsl has a cool climate restricting weathering and transformation of minerals common in humid tropics. As activation energy for kaolinite to halloysite transformation is temperature-dependent, climatic conditions which fail to provide sufficient activation energy for reaction mechanisms may slow down the process and along with ecosystem-supported geochemical conditions will





Peak number	*hkl*	*d*-spacing
1	001	7.21
2	020	4.48
3	110	4.39
4	111	4.20
5	002	3.59
6	130	2.57
7	131	2.50
8	003	2.39
9	202	2.35
10	131	2.30

Figure 3. Randomly oriented X-ray diffraction (XRD) patterns of clay samples from different ecosystems. ***a***, Moist deciduous forests; ***b***, Shola forests; ***c***, Evergreen forests; ***d***, Scrub jungle; ***e***, Grasslands.

retain metastable mineral phases for relatively longer geological time periods[4].

Scrub jungle lies in the rain shadow area of the Western Ghats and represents a large number of plants and animals unique to the thorny vegetation. Geologically the area is comprised of gneissic metamorphic rocks from the Archean shield. Rainfall acts as a limiting factor for weathering in the region. The reduced leaching and weathering action in the study area was evident from sandy texture, near-neutral pH and 50% base saturation in its soil environment. A prominent basal diffraction 001 peak was observed at 0.81 nm (001 *hkl*) immediately (<1 h) after intercalation with formamide, confirming the presence of dehydrated halloysite in the clay samples. Retention of 0.70 nm peak even after 4 h of formamide treatment along with tailing of the peak towards 0.10 nm with time confirms the presence of kaolinite in these soils. Presence of kaolinites in the samples also led to a disintegration of 0.70 nm peak and subsequent strengthening of 0.81 nm peak, 10 days after formamide intercalation. Earlier studies[11] had confirmed that the time lapse of kaolinite in reinforcing the 0.81 nm peak by formamide intercalation was likely influenced by sample crystallinity, whereas complex formation by halloysite was rapid and complete, irrespective of differences in crystallinity or morphology of clay samples. A 0.10 nm peak

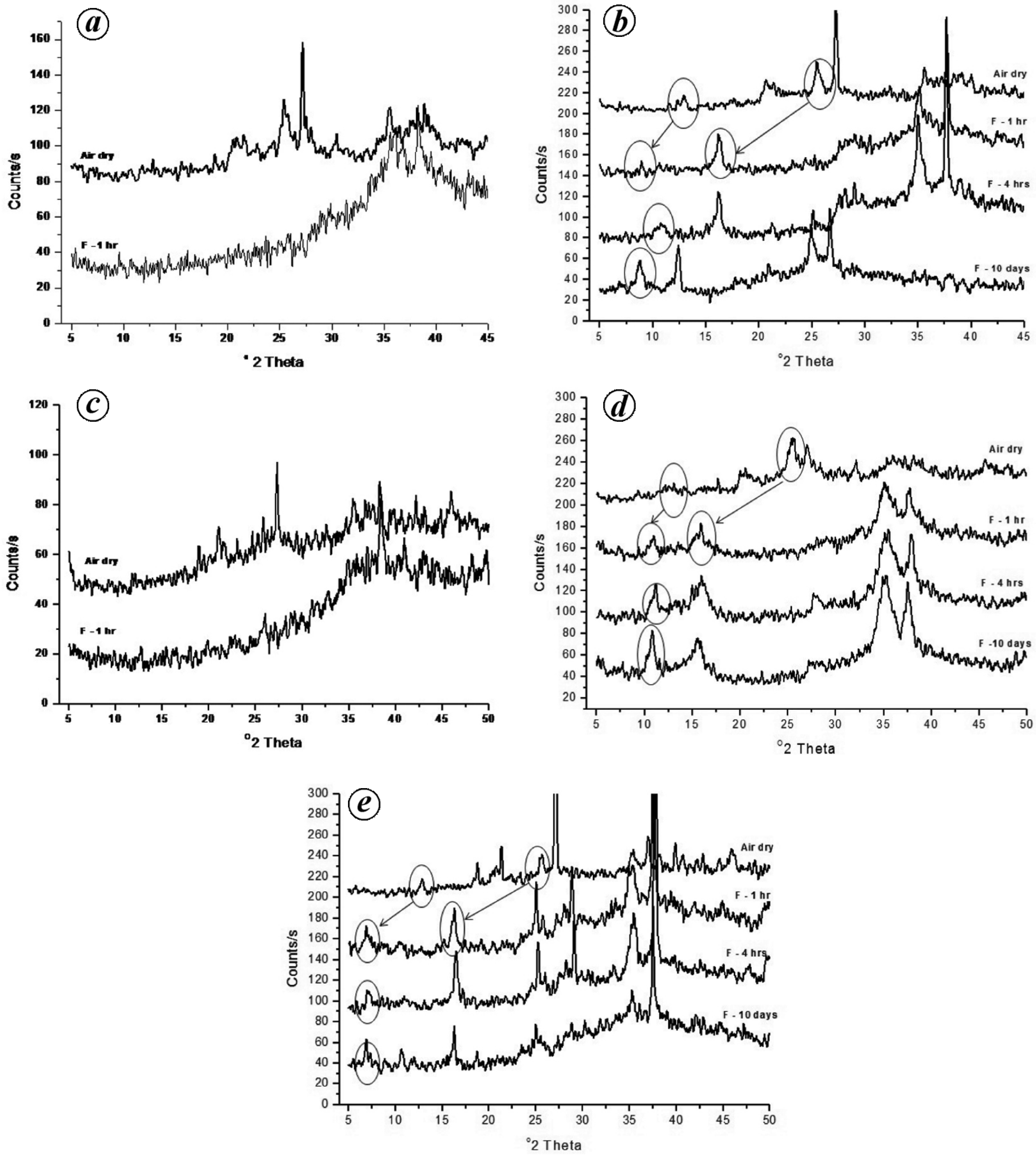


Figure 4. Randomly oriented XRD patterns of formamide intercalated clay samples from different ecosystems. ***a***, Moist deciduous forests; ***b***, Shola forest; ***c***, Evergreen forest; ***d***, Scrub jungle; ***e***, Grassland. Air dry, Diffraction peaks of air-dried samples; F–1 h, Diffraction peaks within 1 h of formamide treatment; F–4 h, Diffraction peaks after 4 h of formamide treatment; F–10 days, Diffraction peaks after 10 days of formamide treatment.

detected in air-dried clay samples of scrub jungle was found to persist throughout the formamide treatments (1 h, 4 h and 10 days) without change, indicating mica minerals. Studies[17] show that crystallization of kaolinites and halloysites occurs only in leaching solutions containing silica in excess of about 5 ppm. Scrub jungles with a low leaching environment could easily maintain these silica concentrations in their soil solutions for longer periods and thereby support halloysite–kaolinite coexistence.

Kaolin minerals in grassland soils were characterized as hydrated halloysites and well-ordered kaolinites. Air-dried, random-oriented clay samples from this system exhibited single strong diffraction peak without splitting at 0.72 nm (001 *hkl*) and 18–22° 2θ (0.45 and 0.42 nm corresponding to 020 and 111 *hkl*), indicating the presence of well-oriented kaolinite. Unlike XRD peaks of total clays from this system, diffractograms of shola, scrub jungle and deciduous systems had given broadly spreading peaks in 18–22° 2θ range corresponding to disordered kaolinites in those soils. Presence of hydrated halloysite was established by a prominent sharp peak at 0.10 nm immediately after formamide intercalation. After 4 h of formamide treatment, the 0.10 nm peaks got diffused indicating loss of crystallinity and stability of halloysite.

Table 2. Crystallinity of 1 : 1 clays in forest ecosystems of Kerala part of the Western Ghats

Ecosystem	Relative crystallinity	Hinckeley's crystallinity index
Moist deciduous forest	11.0	0.65
Evergreen forest	32.0	0.68
Shola forest	16.3	1.06
Scrub jungle	32.5	–
Grassland	25.0	1.07

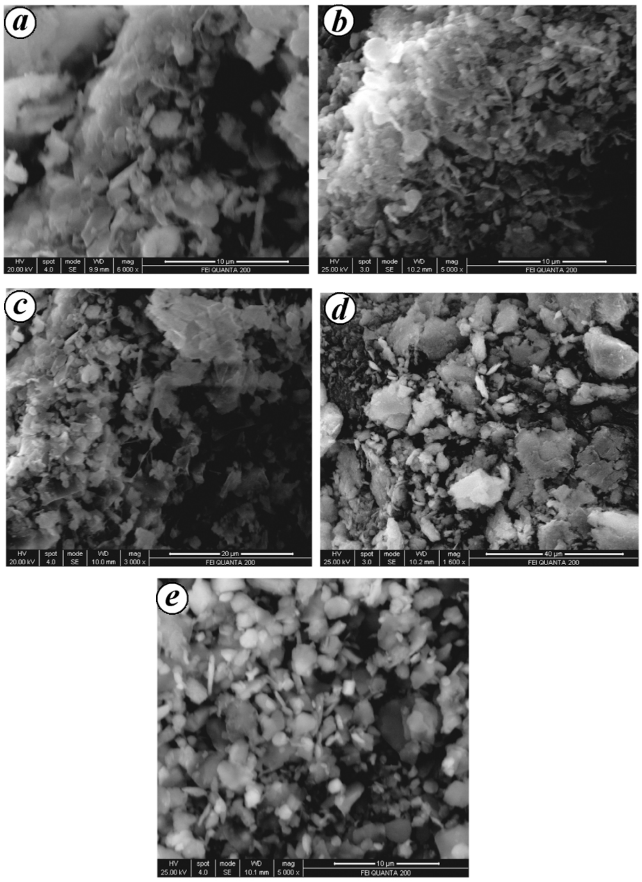


Figure 5. Scanning electron microscopic images of clay fractions from different forest systems showing the presence of halloysite. ***a***, Moist deciduous forests; ***b***, Shola forest; ***c***, Evergreen forest; ***d***, Scrub jungle; ***e***, Grassland.

No halloysites were detected in moist deciduous and evergreen forests. However, clays from these soils were rich in disordered kaolinites. SEM analysis of clay samples from moist deciduous and evergreen forests showed hexagonal and platy kaolinite, whereas shola, grassland and scrub jungle gave tubular halloysites along with kaolinites.

Results from XRD and SEM analyses do not clearly point out the variation of clay crystallinity in different systems. This was overcome by determining relative crystallinity and CI of the different kaolin clays (Table 2). Owing to the broadly spreading 02 l, 11 l bands (18–22° 2θ), the disordered kaolin of evergreen forests could not be evaluated by Hinckley's method. Relative crystallinity of kaolin minerals in the ecosystems was found to be in the order scrub jungle > evergreen forest > grasslands > shola forest > moist deciduous forest. The relative crystallinity and CI give a good indication of the degree of mineral weathering in natural systems. As halloysites are a metastable phase in kaolinite formation, the presence of this mineral can be taken as a reasonable indicator of system-induced restricted weathering in the tropics. The observed higher Hinckley CI values in ecosystems with kaolinite–halloysite (shola forest and grassland systems) in the present study further confirm this hypothetical deduction.

Despite earnest efforts of researchers, a pertinent question remains as to why there are no extremely weathered Oxisols (USDA soil taxonomy system) in the humid tropics even after millions of years of intense weathering, the answer to which lies in the proper understanding of genesis and mineral transformation kinetics in the tropics[9]. Ecosystems with metastable phases of minerals can be considered as young systems which provide valuable inputs of early mineral conversions while developing models of soil genesis in the tropics. Further scientific analyses of these systems may provide us a clue on restrictions in degradation of Ultisols to Oxisols with time, even in intense weathering conditions of the humid tropics. Knowledge of soil physico-chemical conditions and ion balances supported by these systems for formation and maintenance of metastable phases like halloysites will also help us in the scientific management of highly utilized and exposed soils of the humid tropics.

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Yar tsa Gunbu [*Ophiocordyceps sinensis* (Berk.) G.H. Sung *et al.*]: the issue of its sustainability

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Any resource of immense value and key relevance to rural livelihood as the main cash source, invariably runs the risk of being overexploited, more so when it remains a common property resource. The current harvest pressure on caterpillar fungus, Yar tsa Gunbu (*Ophiocordyceps sinensis* (Berk.) G.H. Sung *et al.*) serves, as a prime example. The ever-increasing demand for the commodity in the international markets and concomitantly its ever-increasing price, hovering at present at US$ 20,000 per kg, have resulted in not just its rampant exploitation, but also in the degradation of the very habitat, thus endangering its future. The present study conducted across nine broad landscapes in 110 villages and 2511 harvesters within Pithoragarh district, Central Himalaya, highlights the socio-economic changes brought forth by the harvesting of the 'green gold' and discusses the prospects of future availability of the species. The study also provides suggestions for evolving sound mechanisms to lessen the pressure on the species.

Keywords: *Cordyceps sinensis*, economy, harvest pressure, sustainability, traditional belief system.

THE Latin etymology of *Cordyceps sinensis* (Berk.) G.H. Sung *et al.* (= *Cordyceps sinensis* (Berk.) Sacc.), Ophiocordycipitaceae (Ascomycetes) is as follows: *cord* – 'club', *ceps* – 'head' and *sinensis* – 'Chinese'. The mushroom is also called the 'caterpillar fungus', and, more frequently Yar tsa Gunbu, which translates as 'winter worm, summer grass', or locally in Kumaun and Garhwal Himalaya as 'keera ghaas', referring to the larva (syn. keera) and the emergent fruiting body that appears as sprouting grass (syn. ghaas). *Cordyceps* is a genus of mostly entomophagous flask fungi (Pyrenomycetes, Ascomycotina) belonging to the family Ophiocordycipitaceae. The British mycologist Berkeley[1] first described it in 1843 as *Sphaeria sinensis* Berk. Later in 1878, Saccardo[2] renamed it as *Cordyceps sinensis*. Based on molecular phylogenetic study, Sung *et al*.[3] separated the mega genus *Cordyceps* into four genera, viz. *Cordyceps* (40 spp.), *Ophiocordyceps* (146 spp.), *Metacordyceps* (6 spp.) and *Elaphocordyceps* (21 spp.), while the remaining 175 spp. were left in the *Cordyceps* group. As a result, *C. sinensis* was transferred to *Ophiocordyceps*, and hence renamed *Ophiocordyceps sinensis*. However, for convenience of the readers, the most commonly used taxa – *Cordyceps* is used in this communication.

C. sinensis parasitizes various grass root-boring *Thitarodes* (previously *Hepialus*) caterpillars, which hatch as 'ghost moths' when not preempted by *Cordyceps*. The genus *Thitarodes* comprises around 51 species, out of which about 40 species have been identified as being the host of *C. sinensis*[4]. Uninfected larvae typically hibernate deeper in the soil than infected ones; apparently the fungus is able to force the infected host to locate itself nearer the soil surface in a position more favourable to its fruiting. The hyphae of the mycelium develop inside the body of the insect, first feeding on less vital parts before taking over the complete organism. Eventually the insect is completely mummified and emptied of nutrients, and all that remains is the exoskeleton filled and coated with *Cordyceps mycelium*. In spring, the mushroom (the fruiting body or stroma) develops out of the head of the exoskeleton just above the eyes. The slender, brown, club-shaped fruiting body then emerges from the ground,

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