

# Mineralogical transformations under fire in the montane grassland systems of the southern Western Ghats, India

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Forest systems in the Western Ghats region have undergone significant transformations over the past century and wild fires are considered as a major factor for such modifications. Apart from natural fires, prescribed fire is also adopted as a management strategy in some of the forest types in the region. The present study evaluated the impact of prescribed fires on soil components in the high-altitude montane grassland systems of the southern Western Ghats. Fire-induced mineral transformations were assessed using X-ray diffraction (XRD), thermogravimetry (TGA), differential thermogravimetry (DTA), differential scanning calorimetry (DSC) and thermodynamic parameters. Organic carbon content which determines key soil functions was found to reduce from 1.96% before fire and stabilize at 1.48% in soils following fire. XRD and TGA–DTA analyses indicated that major changes in soil during fire occurred between 70°C and 110°C, 250°C and 320°C as well as 430°C and 500°C, corresponding to loss of interlayer water from phyllosilicate minerals, modification of gibbsite to amorphous minerals and goethite to hematite, and transformation of kaolinite to metakaolinite respectively. Thermodynamic parameters ( $\Delta H$ ,  $\Delta S$  and  $\Delta G$ ) estimated from the DSC curves showed that such transformation had positive enthalpy ( $\Delta H$ ) and Gibbs free energy change ( $\Delta G$ ) values; hence they are not spontaneous or reversible by themselves.

**Keywords:** Clay minerals, forest fire, montane grassland systems, thermodynamics.

FORESTS in the humid tropical regions are highly vulnerable to wildfires and such fire-induced changes are considered an important part of the landscape dynamics<sup>1</sup>. Besides natural fires, carefully planned, prescribed fires are also adopted in some forest systems as an important management strategy to counter the spread of high-intensity fires<sup>2</sup>, prepare sites for planting or seeding of forest species, control competing vegetation<sup>3</sup>, control pests<sup>4</sup>, improve habitat quality needed for wildlife, increase forest stand accessibility and preserve plant species that are dependent on fire. During a fire, along with burning

of vegetation and litter layers, the topsoil layers also get heated by the combined effects of combustion and heat transfer<sup>5</sup>. Such sharp temperature gradients in the surface soil layers lead to changes in biological, physical, chemical and mineralogical properties of soils<sup>6</sup>, and the extent of such effects depends on the type of fire (intensity and duration), land slope and post-fire weather conditions<sup>7,8</sup>. In the humid tropical region, higher fire frequency and intense precipitation pose a potential risk of increasing nutrient loss and reducing soil fertility by erosion, thereby limiting recovery of soil health<sup>9,10</sup>.

Combustion of vegetation produces intense heat, sufficient to alter or completely change soil minerals and organic matter. Conditions during fires may be suitable for decomposition, dehydration or collapse of soil minerals that play a vital role in soil processes and functions. For example, fire temperatures above 200°C alter goethite, a common soil mineral, to hematite or maghemite. Soils in the humid tropics have abundance of kaolin which is thermally decomposed around 420–550°C, producing amorphous aluminosilicates and thereby having a negative impact on these 1 : 1 clay minerals<sup>11</sup>. Expanding phyllosilicates like montmorillonite are reported to irreversibly change or get thermally decomposed at high temperatures<sup>12</sup>. Dehydroxylation of 2 : 1 octahedral and interlayer hydroxide sheets may lead to decomposition of minerals such as chlorite and chlorite–vermiculite<sup>13</sup>. Other aluminosilicate minerals, such as illites and vermiculite, may also be impacted by high-intensity fire, either due to dehydration of interlayer cations or as a result of the release of ions such as  $K^+$ ,  $Ca^{2+}$  and  $Mg^{2+}$ , leading to a reduction in their 001 *d*-spacing. Reports indicate that such mineral alterations and transformations in the soil following forest fires will persist over longer periods of time<sup>11</sup>. Though fires are frequent and widespread in the forest systems of India, their impacts on the soil have been scarcely studied.

Montane grasslands, lying at an altitude of 2000–2600 m amsl, are specific systems of the southern Western Ghats predominated by grasses along with a mix of herbaceous and shrubby plants. Representative grassland systems for the study were selected at Eravikulam National Park, Idukki, Kerala, India. The area sustains the largest population of the endangered ungulate, Nilgiri

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tahr (*Hemitragus hylocrius*, Ogliby), endemic to South India. Fire is regularly set as a prescribed management strategy in these ecosystems to induce fresh grass sprouts for Nilgiri tahr, and as a fire break to prevent spread of wild fire in the forests during summer. Though a multitude of benefits of wild fires on the forest ecosystem have been assessed, their effects on soil have been rarely evaluated in these systems.

In montane rain forest ecoregion of the southern Western Ghats, soils undergo intense weathering and erosion under the prevailing topography and climate, and therefore become a qualitatively scarce resource. The protection of this scarce resource is essential for provisioning ecosystem services, hydrological cycle, carbon storage and nutrient cycle<sup>14</sup>. Knowledge of soil degradation processes and their context in specific systems will help us design practices focused on soil health protection.

## Materials and methods

The Eravikulam National Park is a protected area in the high ranges of the southern Western Ghats (10°05'–10°20'N, 77°0'–77°10'E), covering an area of about 97 sq. km. The area is occupied by high-grade supracrustal rocks, gneisses and granulites, and has an undulating terrain with an elevation of about 2000 m amsl. Soils are loamy sands belonging to Typic Dystrustepts (USDA classification). Mean annual precipitation and temperature are in the range 2300–3000 mm year<sup>-1</sup> and 13–27°C respectively.

Prescribed fire was applied to 50 ha of montane grasslands on 16 December 2016, as part of the management plan. Temperature at soil surface during the fire was measured using a portable thermocouple fitted with a data logger. Surface soil samples (0–5 cm depth) were collected at 50 m interval from four points in the burned sites. The soil samples were thoroughly mixed to obtain representative composite sample. The samples were collected from the site before fire (BF; date of collection – 14 December 2016), as well as one, three and six months after burning (1B, 3B and 6B; dates of collection – 20 January, 26 April and 20 July 2017 respectively), to assess chemical changes in the soil following fire. The study site was disturbed by pre-monsoon showers (April 2017) and the southwest monsoon (June–July 2017) before the collection of 3B and 6B samples respectively. The collected samples were air-dried, slightly powdered, sieved (2 mm sieve) and stored at room temperature for further analysis.

Soil reaction (pH) and electrical conductivity (EC) were estimated in 1 : 2.5 soil : water suspension. Available N was estimated by alkaline permanganometry<sup>15</sup>, NH<sub>4</sub>F + HCl extracted phosphorus (available P) by colorimetry, neutral normal NH<sub>4</sub>OAc extractable potassium (available K) by flame photometry<sup>16</sup>, and oxidizable organic carbon content by wet oxidation method<sup>17</sup>.

Mineralogical analyses, thermogravimetric analysis (TGA) and differential scanning calorimetric (DSC) analysis were carried out in BF and 1B samples only, as the 3B and 6B samples could have rain-induced artefacts. X-ray diffraction (XRD) analyses were done after treatment of the sample with hydrogen peroxide and sodium citrate–bicarbonate–dithionite to remove organic matter and iron oxides respectively<sup>18</sup>. Silt (0.02–0.002 mm) and total clay (<0.002 mm) fractions were extracted from the soil by centrifugation and randomly oriented fractions were studied by XRD analysis (Bruker AXS D8 Advance) with Ni-filter, Cu-K<sub>α</sub> radiation at a scanning speed of 2°2θ/min. Identification of minerals in the silt and clay fractions was done according to the criteria laid down by Jackson<sup>18</sup>. TGA of the soil samples was performed (Perkin Elmer, Diamond TGA/DTA Analyser) in the temperature range from ambient to +800°C.

Heat-induced transformations of the samples were studied by DSC (Mettler Toledo DSC 822e) in the temperature range –130°C to 450°C. Thermodynamic parameters such as change in enthalpy (ΔH), entropy (ΔS) and Gibbs free energy (ΔG) were calculated from the DSC curves using modified Gibbs–Helmholtz equation<sup>19</sup>

$$\Delta G^{\circ}(T) = \Delta H_m^{\circ}[1 - (T/T_m)] + \Delta C_p [(T - T_m) - T \ln (T/T_m)], \quad (1)$$

$$\Delta H^{\circ}(T) = \Delta H_m^{\circ} + \Delta C_p(T - T_m), \quad (2)$$

$$\Delta S^{\circ}(T) = \Delta S_m^{\circ} + \Delta C_p \ln(T/T_m), \quad (3)$$

where  $T$  is the temperature of interest,  $T_m$  the transition midpoint temperature (midpoint of transition peak in the DSC curve),  $\Delta G^{\circ}$  the standard free energy change or the difference in Gibbs energy between different states (reactant and product),  $\Delta H^{\circ}$  the enthalpy change at the temperature at which  $\Delta G^{\circ}$  is evaluated;  $\Delta S^{\circ}$  the entropy change at the temperature at which  $\Delta G^{\circ}$  is evaluated;  $\Delta H_m^{\circ}$  the transition enthalpy change (transition peak area in DSC curve) and  $\Delta C_p$  is the heat capacity change (shift in baseline of the DSC curve).

## Results and discussion

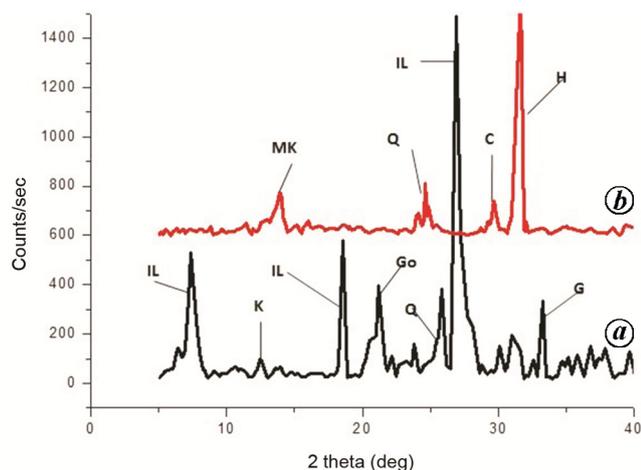
### *Fire-induced chemical changes in grassland soils*

Soil pH and EC were found to be substantially modified by fire. They increased from 5.76 to 5.81 and from 0.14 to 0.38 dS/m respectively (Table 1). The increase in soil reaction (pH) is an indication of improvement of alkalinities in soil released from oxidized organic matter<sup>20</sup>. Burning of organic residues in soils and plant litter produces soluble salts of Ca and K, thereby increasing EC<sup>21</sup>. However, the poorly ordered acid-forming soluble

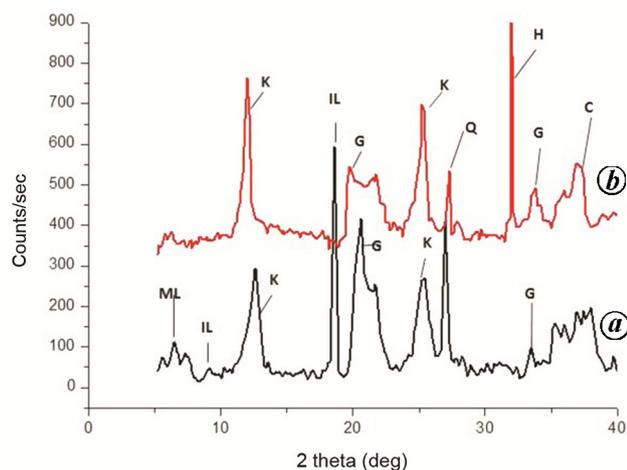
**Table 1.** Effect of fire on selected chemical properties of soil in the montane grassland system of the southern Western Ghats

Parameters	BF	1B	3B	6B
pH	5.76	6.17	5.73	5.65
EC	0.14	0.65	0.26	0.18
Organic carbon (%)	1.98	1.48	1.42	1.46
Available N (kg/ha)	326.00	338.00	328.00	324.00
Available P (kg/ha)	24.97	32.25	28.42	26.71
Available K (kg/ha)	328.27	635.41	390.36	357.92

BF, Before fire; 1B, One month after burning; 3B, Three months after burning; 6B, Six months after burning.



**Figure 1.** XRD patterns of randomly oriented silt-sized fractions of soils from montane grassland systems of the southern Western Ghats. *a*, Silt fractions in soil before fire (BF); *b*, Silt fractions in soil after fire (IB). C, Calcite; Go, Goethite; G, Gibbsite; H, Haematite; IL, Illite; K, Kaolinite; MK, Metakaolinite; Q, Quartz.



**Figure 2.** XRD patterns of randomly oriented clay-sized fractions from montane grassland systems of the southern Western Ghats. *a*, Clay fractions in soil before fire (BF); *b*, Clay fractions in soil after fire.

forms of Fe and Al minerals formed by dehydroxylation (discussed later) of the parent crystalline minerals during fire neutralize the bases and even reduce the pH compared to the original in the long run. Leaching of bases during rainfall further reduces the soil pH. Organic

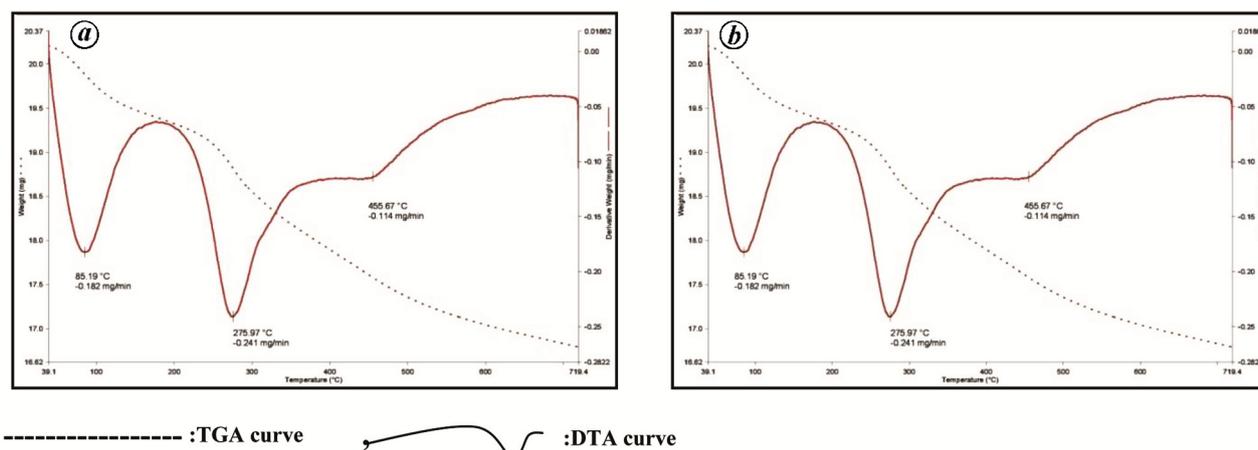
carbon, an important indicator of soil health, decreases substantially following fire from 1.98% (in BF) to 1.46% (in 6B). A combined effect of fire-induced carbon oxidation and humic acid transformation effectively reduces the oxidizable and labile carbon forms in soil<sup>7</sup>. Carbon being the energy source of microbial activities, such decline in organic carbon pool will seriously affect the soil food web and thereby soil processes and functions, leading to its rapid degradation.

In unburned soils, the silt fraction was found to be dominated by illite, kaolinites, goethite, quartz and gibbsite, while the clay fraction had predominance of kaolinite, illite, gibbsite and kaolinite–hydroxy-interlayered vermiculites (hereafter mixed layer aluminosilicate minerals) (Figures 1 and 2). In a humid tropical climate and under an acid weathering environment, formation of 1:1 type minerals (e.g. kaolinite), hydroxy–aluminium interlayering and interstratification of 2:1 and 1:1 layers to form mixed layer aluminosilicate minerals are the primary reactions. The results indicate that fire affects the nature of these soil minerals and presumably their interaction with nutrients. TGA–DTA curves show that major transformations of the soil occur in the temperature range 70–110°C, 250–320°C and 430–500°C (Figure 3 *a* and *b*).

#### *Fire-induced mineralogical changes in grassland soils*

Following fire, the mineralogical composition of silt fraction changed to metakaolinite, calcite, haematite and quartz. Disappearance of mixed-layer aluminosilicate minerals, illites and appearance of peaks representing amorphous gibbsite, calcite and haematite were the dominant changes in the clay fraction. The results indicate that fire affects the nature of soil minerals and presumably their interaction with soil nutrients.

Soils in the humid tropics are usually dominated by kaolinites and sesquioxide minerals<sup>22</sup>. However, forest soils in the region are relatively young and have abundance of mica group minerals and mixed-layer aluminosilicates along with kaolinites<sup>23</sup>. Under high temperatures induced by fire, dehydroxylation and recrystallization occur in a stepwise manner leading to the alteration



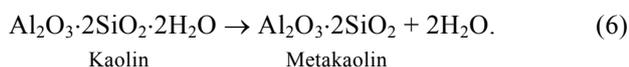
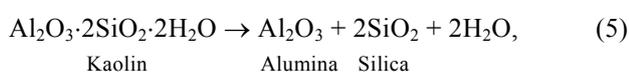
**Figure 3.** Thermogravimetric and differential thermal analysis curves of soil samples from montane grassland systems of the southern Western Ghats. *a*, Soil before fire; *b*, soil after fire.

of minerals in both silt and clay fractions<sup>24</sup>. Earlier studies have reported that heating temperatures of about 200°C causes dehydration and alters gibbsite to complex crystalline and amorphous alumina mineral assemblages<sup>25,26</sup>.

Gibbsite → boehmite → amorphous alumina. (4)

At 300°C, goethite alters to partly ordered haematites<sup>27</sup>. The presence of goethite in the samples before fire and its relatively lower XRD peak intensities or absence in the burned samples confirms results of earlier studies that a good portion of goethite got converted to haematite under high temperature conditions due to fire. Haematite formation from goethite following fire may be by way of dehydroxylation of the crystalline goethite or dissolution of goethite and reprecipitation through an intermediate phase such as hydrohaematite in soil solution<sup>28</sup>.

The temperature due to fire in the study site was recorded to rise up to 800°C with a mean temperature of 450–600°C. Such intense temperatures above 600°C provide sufficient energy to modify kaolin to a mixture of independent amorphous alumina and amorphous silica or metakaolins<sup>29</sup>



Calcite, another major mineral phase that developed in the soil after burning, is attributed to the transformation of calcium oxides in the vegetative tissue following incineration<sup>30,31</sup>. The loss of crystalline 2:1 minerals such as mixed-layer aluminosilicate minerals and illites from the clay fractions during fire may seriously impair the ecosystem functions of soil, as these expanding clay minerals provide a wide range of services from nutrient buffering to cation exchange capacity of soils.

### *Fire-induced thermogravimetric changes in grassland soils*

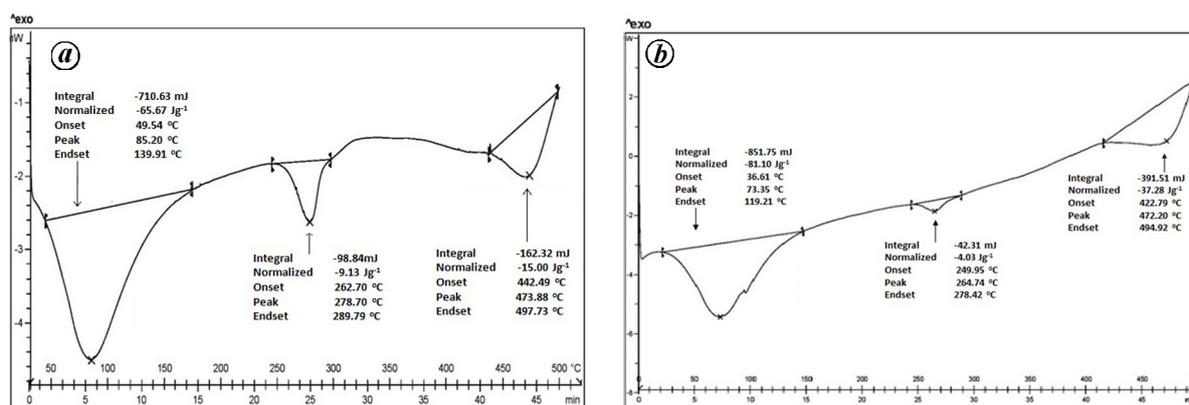
During fire, the first chemical reaction was found to occur around 70–110°C, which can be attributed to the loss of interlayer water molecules from 2:1 type minerals (illites or mixed-layer aluminosilicate clay minerals), and is an endothermic process. The interlayer water in 2:1 layer minerals can be either ‘bound water’ or ‘unbound water’, a significant portion of which gets removed at ~110°C (ref. 32). The second endothermic peak was observed in the temperature range 250–320°C, corresponding to the dehydroxylation of goethite to form haematite. Transformation of gibbsite to amorphous alumina and complete oxidation of soil organic matter also occur around this temperature range. Kaolinite transformation to metakaolinites occurring at ~550°C is represented by the third endothermic peak in the TGA spectrum. Maximum weight loss was found in the temperature range 250–320°C corresponding to three major transformation processes (gibbsite to amorphous silica, goethite to haematite, oxidation of organic matter) and minimum during kaolinite to metakaolinite transformation. Kaolinite being low activity, non-expanding clay holds less water and cations ( $\text{K}^+$ ,  $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$ ) in its lattice and higher temperature is required to disrupt the structure leading to its transformation.

TGA spectra indicated that unburned soil samples lost 16.89% of its weight when heated in a temperature range of ambient to 800°C (Table 2). These values are close to the theoretical dehydroxylation related weight loss value for kaolinite (14%) at 520°C. On the other hand, TGA spectra of burned soils had a weight loss that was 43.5% lesser than the unburned soil in the same temperature range. The greater weight loss in the TGA spectra of unburned samples than burned soils can be attributed to the loss of strongly mineral-adsorbed water in the former between 110°C and 400°C (ref. 33). In the present study,

**Table 2.** Effect of fire on thermogravimetric and thermodynamic properties of montane grassland soils in the southern Western Ghats

Sample	Weight loss (%)	DTG peak		Thermodynamic parameters		
		Temperature (°C)	Rate of decomposition (mg/min)	$\Delta H$ (J/g)	$\Delta S$ ( $10^{-5}$ J/K)	$\Delta G$ (J/g)
BF	16.89	85.19	0.18	65.67	-1.25	65.67
		275.97	0.24	9.13	-0.47	9.13
		455.67	0.11	15.00	-0.25	15.00
1B	9.58	82.23	0.09	81.10	-1.55	81.10
		275.46	0.09	4.03	-0.33	4.03
		455.27	0.08	37.28	0.67	37.27

BF, Before fire; 1B, One month after burning.

**Figure 4.** Differential scanning calorimetry spectra of soil samples from montane grassland systems of the southern Western Ghats. *a*, Soil before fire; *b*, Soil after fire.

soils had reached temperatures up to  $\sim 800^{\circ}\text{C}$  during fire that are sufficient enough to cause dehydroxylation, and loss of water and volatile components<sup>34</sup>.

#### Thermodynamics of fire-affected soil transformations

Three endothermic peaks have been observed in the DSC curve corresponding to transformations that are obvious in the TGA–DTA spectra. The endothermic peaks in the DSC spectra between  $49.54^{\circ}\text{C}$  and  $139.91^{\circ}\text{C}$ ,  $262.7^{\circ}\text{C}$  and  $289.79^{\circ}\text{C}$  as well as  $442.49^{\circ}\text{C}$  and  $497.73^{\circ}\text{C}$  correspond to weight loss points in TGA and confirm dehydroxylation and recrystallization processes in the soil (Figure 4 *a* and *b*). The characteristic midpoint transition temperature ( $T_m$ ) for transition or transformation during burning was found to be  $85.20^{\circ}\text{C}$ ,  $278.70^{\circ}\text{C}$  and  $473.88^{\circ}\text{C}$  corresponding to the loss of interlayer water, dehydroxylation reactions and metakaolinite formation respectively. Burned soil (1B) already deprived of most of the components gave peaks at these temperatures and showed DSC peak intensities lower than in the unburned soils. The Gibbs free energy values were found to be maximum at  $85.20^{\circ}\text{C}$  in both BF and 1B, corresponding to release of hygroscopic moisture from the soil. The positive values for free energy ( $\Delta G$ ) and enthalpy ( $\Delta H$ ) and

negative values for entropy ( $\Delta S$ ) for the soil mineral transition or transformation processes show that these reactions are non-spontaneous and irreversible in the soil. Under natural conditions these processes occur at an infinitesimally small rate and take millions of years to be completed. However, fire imparts a large amount of energy into the soil system and witness irreversible transformation of mineral phases within a short span of time (positive Gibbs free energy). It can be concluded that frequent fires at the same location will accumulate transformed minerals like metakaolin and inert resistant minerals like haematite (minerals at the end of a weathering sequence), and drastically deplete the soil health.

#### Conclusion

Influence of fire on the soil system depends on its severity. Soils in the montane grassland systems show temporary nutrient enrichment after burning, but revert to a lower level than the original contents within a short span of six months. During fire, major changes were found to occur between  $70^{\circ}\text{C}$  and  $110^{\circ}\text{C}$ ,  $250^{\circ}\text{C}$  and  $320^{\circ}\text{C}$  as well as  $430^{\circ}\text{C}$  and  $500^{\circ}\text{C}$ . Between  $70^{\circ}\text{C}$  and  $110^{\circ}\text{C}$ , phyllosilicate minerals lost water molecules through an endothermic process. Major transformations were found to occur around  $250\text{--}320^{\circ}\text{C}$ , which include transformation

of gibbsite to amorphous minerals, goethite to haematite and oxidation of organic matter producing substantial weight loss in the samples. Kaolinite, a major soil mineral in the tropical soils, was altered between  $\sim 430^{\circ}\text{C}$  and  $500^{\circ}\text{C}$  to metakaolinite during fire. Thermodynamic parameters estimated from DSC curves indicate that these soil modifications have positive  $\Delta H$  and  $\Delta G$  values and hence are not by themselves feasible or reversible under natural conditions. The large amount of heat energy infused by fire accelerated the processes which would have otherwise occurred at an infinitesimally slow pace under natural conditions. Hence, continuous use of fire as a management strategy will reduce active organic fractions and accumulate transformed minerals like amorphous alumina, haematite and metakaolin in the pristine ecosystems of the southern Western Ghats within a short period, thereby depleting the soil of its natural composition and its capacity to support ecosystem services.

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