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## Late Holocene aridification recorded in the stable carbon and nitrogen isotope composition of soils from Nainital, Lesser Himalaya

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### ABSTRACT

This study incorporates micromorphology, geochemistry, and stable carbon and nitrogen isotope compositions of soil organic matter ( $\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$ ) in a radiocarbon-dated Holocene Alfisol section from Nainital, northwest Himalaya, India to understand pedogenic and vegetation response to changing Indian Summer Monsoon (ISM). Soil micromorphology suggests this soil to be an Alfisol that formed in semiarid to humid conditions. Extensive illuviation with thick clay pedofeatures found in the Bt-horizon suggests wet conditions and intense monsoon during early to middle Holocene (9.0–4.6 ka). The  $\delta^{13}\text{C}$  varies from  $-14.2\text{‰}$  to  $-22.6\text{‰}$  and reflects abrupt changes in the proportions of C3 and C4 plants in the ecosystem during late Holocene. An average  $\delta^{13}\text{C}$  of  $-21.6 \pm 1.0\text{‰}$  during early to middle Holocene (9.0–4.6 ka) indicates that the vegetation was dominated by C3 plants growth under hot humid and strong monsoonal conditions. On the other hand,  $\delta^{13}\text{C}$  ( $-16.7 \pm 2.1\text{‰}$ ) during late Holocene ( $\sim 4\text{--}1$  ka) suggests distinct vegetation shift towards increasing C4 plants abundance related to aridification (weak ISM) at  $\sim 4$  ka. The pattern of  $\delta^{15}\text{N}$  variability in the soil is similar to that observed for the  $\delta^{13}\text{C}$  variability in the profile. The  $\delta^{15}\text{N}$  varies from  $+4.4\%$  to  $+8.3\%$ , with significantly more positive values during late Holocene (weaker monsoon) compared to early to middle Holocene (stronger monsoon), consistent with the relationship between  $\delta^{15}\text{N}$  and mean annual precipitation observed on a global scale. Our study demonstrates a strong and rapid adjustment of vegetation in response to ISM variability during Holocene.

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### 1. Introduction

Indian Summer Monsoon (ISM) is a dynamic climate system, which affects climate throughout south Asia (Prasad et al., 2014). The intensity of ISM is mainly regulated by the latitudinal location of the Intertropical Convergence Zone (ITCZ). It was argued that gradual southward migration of the ITCZ resulted in a decrease in ISM intensity (Fleitmann et al., 2003; Zorzi et al., 2015). Long-term trends in ISM precipitation are controlled by summer insolation, which affects the latitudinal position of the ITCZ and in turn the ISM intensity (Fleitmann et al., 2003). On the other hand, short-term millennial/centennial scale fluctuations in ISM precipitation are controlled by several factors such as (i) glacial events in the North Atlantic (Gupta et al., 2003), (ii) changes in the Indian Ocean Dipole (IOD) and El Niño Southern Oscillation (ENSO) (Fisher et al., 2008; MacDonald, 2011), and (iii) the Indo-Pacific warm pool (Prasad

et al., 2014). In general, a hot and humid climate persisted during early Holocene (9–7 ka) that gradually changed to a more arid climate towards the present-day (Sarkar et al., 2016). The Holocene has also witnessed short-term/abrupt climate changes (Gupta et al., 2003). An archeologically significant event, i.e., deurbanization of the Indus Valley Civilization around 4 ka, has been linked to the onset of an arid phase at 4.2–4.0 ka due to weakening of ISM (Staubwasser et al., 2003; Berkelhammer et al., 2012; Ponton et al., 2012; Dixit et al., 2014a; Zorzi et al., 2015). Recent soil geomorphic studies of the Indo-Gangetic Plains (IGP) provide information on paleoclimatic changes over the last 13.5 ka marked by two humid phases punctuated by arid-semiarid conditions (Srivastava et al., 2015, 2016). Despite the significant information from polygenetic soils of the IGP now available, there is no complete Holocene climatic record for ISM reconstructions based on soils developed in the northwest (NW) Himalaya.

Soils/paleosols serve as one of the most important terrestrial archives that record the prevailing climatic signatures during the formation of soils in the past (Retallack, 2001). Micromorphology of the soils and paleosols deals with description and interpretation of

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the diagnostic pedogenic features such as microstructures, clay coatings, birefringence fabric, bioturbation, pedogenic calcium carbonate, and iron and manganese oxides mottles/concretions to understand (paleo)pedogenic processes and the prevailing paleo-environmental conditions (Sheldon and Tabor, 2009; Srivastava et al., 2016). In addition, soil geochemistry can be used to determine the degree of chemical weathering and leaching, which strongly depends on the mean annual precipitation (MAP) and mean annual temperature (MAT) of the prevailing climate (Retallack, 2001; Sheldon and Tabor, 2009).

The stable carbon isotope composition ( $\delta^{13}\text{C}$ ) of soil organic matter is routinely used as a proxy to reconstruct paleovegetation of a region, which in turn is a function of local climatic parameters (e.g., Boutton et al., 1998; Nordt et al., 2002; Bowman et al., 2004). These interpretations are based on the premise that abundances of C3 (Calvin cycle) and C4 (Hatch-Slack cycle) plant species are strongly dependent on the prevailing climate (Cerling et al., 1989; Pagani et al., 1999). The C3 and C4 types of vegetation are marked by distinctly different  $\delta^{13}\text{C}$  values and thus  $\delta^{13}\text{C}$  of soil organic matter can be used as a proxy for ISM intensity (Galy et al., 2008; Sanyal et al., 2010). Similarly, few studies have attempted to use soil  $\delta^{15}\text{N}$  values in paleoenvironmental studies in terms of availability of nutrients, water, and vegetation (Amundson et al., 2003; Bustamante et al., 2004; Liu and Liu, 2017). It is clear that both  $\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$  of plants are greatly affected by MAP and therefore C and N isotopic composition of soil organic matter may provide clues to ISM variability, which in turn affects regional vegetation patterns.

In this study, we have investigated key macro- and micro-morphological features, bulk soil geochemistry, and  $\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$  variability in one ~2 m deep Alfisol section from Nainital, Lesser Himalaya to understand pedogenic response to changing Holocene climate. This soil was formed from the eroded alluvium derived from surrounding hills of sedimentary rocks mainly shales/pelites. Our major goal was to use  $\delta^{13}\text{C}$  of soil organic matter as a proxy to infer response of vegetation to Holocene climate variability that is in turn related to the intensity of ISM. Our study clearly documents weakening of ISM at ~4 ka leading towards an arid climate persisting until 1 ka, which possibly triggered deurbanization of the Indus Valley Civilization in the northwestern part of the Indian subcontinent.

## 2. Study site

The study site is located in Nainital (29°23'1.3N, 79°27'E; 2262 m a.s.l.), Uttarakhand, India (Fig. 1). It is a part of the Lesser Himalaya located between the Main Boundary Thrust in the south and Main Central Thrust in the north (Valdiya, 1988). The Krol Formation of Proterozoic age is the most important unit in our study area, which predominantly comprises carbonate sediments (Valdiya, 1988). The other Tal Formation in the area comprises carbonaceous shale. Our study site is located on a stable upland region near Tiffin Top with alluvium derived from surrounding hills (Fig. 1). Present-day climate of the area is monsoonal with three seasons as rainy (mid-June to mid-September), winter (November to February) and summer (April to mid-June). The MAP and MAT are ~1100 mm and 25 °C, respectively (Indian Meteorological Department (IMD); <http://www.imd.gov.in/>). The present-day vegetation of the Lesser Himalaya is mainly dominated by C3 plants such as Banj-oak (*Quercus leucotrichophora* A. Campus), Tilonj-oak (*Quercus floribunda* Lindl. ex Rehder), Kharsu-oak (*Quercus semecarpifolia* J.E. Smith) and Chir-pine (*Pinus roxburghii* Sarg) species (Kharkwal and Rawat, 2010). The local vegetation also comprises of mixed-broadleaved (*Q. leucotrichophora*, *Quercus glauca* Thumb, *Pyrus pashia* Ham., *Myrica esculanta* Linn. and

*Rhododendron arborium* Sm. *Q. leucotrichophora*), shrub (*Pyracantha crenulata* M. Roemer), herb (*Anaphalis contorta* Hook. f) and grass (*Chrysopogon Serrulatus*) species, which typically occur in cooler climate (Ram et al., 2004).

## 3. Materials and methods

In this study, a ~2 m soil profile was excavated near Tiffin Top, Nainital (Fig. 1). The soil profile was explored for field characteristics following standard procedures (Schoeneberger et al., 2012). About 500 g of bulk soil samples were collected at a regular interval of 15 cm along the profile, for laboratory analyses (Fig. 2). Six undisturbed soil blocks were collected in metal boxes (Kubiéna Box) for micromorphological study. The unconsolidated nature of the soils required impregnation with polyester resin after air drying to procure hard rock like blocks to obtain the thin sections (Murphy, 1986). The thin sections were studied using a Nikon 100 POL microscope (40–400× magnification) for fine detailing of the soil fabric, groundmass, b-fabric, and pedofeatures, following the guidelines of Stoops et al. (2010). The soil pH and electrical conductivity (EC) measurements were carried out using Eutech pHScan2 and Eutech TDSscan4 meter, respectively, on a 1:5 ratio of sample to MilliQ water.

The radiocarbon dates on bulk soil organic carbon retrieved from three samples – from top, middle, and bottom part of the soil profile – were obtained using the radiocarbon facility of Birbal Sahni Institute of Paleosciences (BSIP), Lucknow, India following the methods described by Farooqui et al. (2016). The bulk soil samples (~150 g), free of rootlets (manually cleaned) were treated with 10% HCl to remove carbonates. After repeated rinsing with distilled water for pH neutralization, the soil samples were dried at ~80 °C, and then combusted at 900 °C in a continuous flow of pure oxygen for about 1 h. The resulting CO<sub>2</sub> was collected and converted to acetylene and then benzene using standard catalysts and procedures. The radioactivity was counted in a Liquid Scintillation Counter (Quantulus 1220). Calibrated ages (cal yr BP) were obtained using OxCal v4.3.2 and IntCal13 data base (Reimer et al., 2013) at 95.4% probability.

Major oxides and selected trace element concentrations were determined on pressed powder pellets analyzed using a Rigaku ZSX primus II wave-length dispersive X-ray fluorescence spectrometer (WD-XRF) at IIT Kanpur. Calibration curves were obtained from regressions of more than 20 international rock standards from United States Geological Survey, Centre de Recherches Pétrographique et Géochimiques, Nancy, and Canadian Certified Reference Materials Project (CCRMP). Two CCRMP standards, LKSD-2 (Lake Sediments, Ontario, Canada) and STSD-4 (Stream Sediments, Ontario, Canada), were analyzed with each batch of unknown samples to determine the accuracy of analyses.

Stable isotope ratio measurements of carbon ( $\delta^{13}\text{C}$ ) and nitrogen ( $\delta^{15}\text{N}$ ) of bulk soil organic matter were carried out using the continuous-flow stable isotope ratio mass spectrometry facility (CF-IRMS) available at IIT Kanpur. For  $\delta^{13}\text{C}$  analyses, about 5–10 mg of carbonate-free samples (using 0.6N HCl at 70–80 °C for 2 h) were combusted in a Flash 2000 elemental analyzer at 1020 °C in an oxygenated environment and the evolved CO<sub>2</sub> was analyzed online in a Delta V Plus IRMS. Nitrogen isotope measurements were done using 15–30 mg of (un-treated) bulk samples combusted in the elemental analyzer followed by isotopic analysis of N<sub>2</sub> gas in IRMS. Data normalizations (Paul et al., 2007) to Vienna Pee Dee Belemnite (VPDB) for carbon and to AIR for nitrogen stable isotope reference scales were done by analyzing International Atomic Energy Agency standards (LSVEC, NBS-19, and CH-3 for carbon; USGS-26, IAEA-NO-3, USGS-35, and IAEA-N-2 for nitrogen). Analytical precision was monitored by analyzing Oxalic Acid (IAEA-C8)

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