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Transport pathways of black carbon to a high mountain Himalayan lake during late Holocene: Inferences from nitrogen isotopes of black carbon

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A B S T R A C T

Historically, forest fires have played a significant role in the production and distribution of black carbon (BC), including its deposition in water bodies. BC can reach to water bodies through two main pathways: (i) wet and dry atmospheric deposition and (ii) transportation of soil BC via surface runoff. Identifying the transport pathways of BC after fire has proven to be a challenging endeavour. This study aimed to decipher the pathway of BC transportation to a lake (Wular Lake, Kashmir Valley, India) by utilizing nitrogen isotopic composition of BC ($\delta^{15}N_{BC}$) from a sediment core spanning 3744 years. The $\delta^{15}N_{BC}$ record demonstrate that terrestrial N dynamics in the Kashmir Valley were predominantly influenced by shifts in climate condition during the late Holocene. The observed variations indicated lower $\delta^{15}N_{BC}$, indicative of dominance of atmospheric transportation of BC to the lake, during relatively drier periods with higher forest fire activity. In contrast, higher $\delta^{15}N_{BC}$, suggesting a dominance of soil BC transportation via runoff, aligned with relative wetter periods of low forest fire activity.

1. Introduction

Forest fires generate a significant amount of pyrogenic material called black carbon (BC) through incomplete combustion of terrestrial biomass (Kondo et al., 2011; Saleh et al., 2014; Liu et al., 2023). The pyrogenic material comprises of elemental or graphite carbon (or soot) to charcoal, char, and partially carbonized plant tissue (Masiello, 2004; Schmidt and Noack, 2000; Wang et al., 2019) and hence likely to contain carbon (C) and nitrogen (N) along with other elements (Bird and Ascough, 2012; Mukherjee and Kumar, 2021). A study showed an incorporation of N in the recalcitrant structure of the pyrogenic material at mid (500–600 °C) and high (900 °C) temperature pyrolysis instead of loss of N, which occurred at lower temperature pyrolysis (McBeath et al., 2015). Previous works on BC have demonstrated that BC retains valuable information regarding fire events, climate change, and vegetation shifts along with C and N cycling in terrestrial ecosystems over geological time scales (Lim and Cachier, 1996; Wang et al., 2005, 2019; Rahman et al., 2021; Verma et al., 2023). Paleoclimatologists have successfully inferred precipitation patterns during the Holocene by analyzing C isotopes of BC (Wang et al., 2013; Verma et al., 2023). Recently, a study focused on utilizing nitrogen isotopes of BC ($\delta^{15}N_{BC}$) to gain insights into changes in terrestrial N dynamics in China and was found to be influenced by climate change and impact of human activities in the region (Wang et al., 2019).

In general, $\delta^{15}N_{BC}$ could represent $\delta^{15}N$ of the plants, which in turn largely depends on climate (Craine et al., 2009; Wang et al., 2019). Usually, lower $\delta^{15}N$ were noticed for wetter climate plants and higher $\delta^{15}N$ with drier climate plants (Garten, 1996; Amundson et al., 2003; Liu and Wang, 2010; Craine et al., 2015). However, other factors such as soil nutrients and bacterial community also play a major role in altering $\delta^{15}N$ signature through various biogeochemical processes such as nitrification and denitrification (Szpak, 2014; Chen et al., 2022; Choi et al., 2023). Additionally, fire is known to significantly impact soil nutrient dynamics and alter the $\delta^{15}N$ signature of plants, as it causes isotopic depletion of ^{15}N in the atmosphere through the release of ash and nutrients, while isotopically enriched ^{15}N persists in charred materials and nutrient-rich soil (Bauhus et al., 1993; Aranibar et al., 2003; Huber et al., 2013). The growth of plants in such soils after fire will have a relatively higher $\delta^{15}N$ (Huber et al., 2013). On the other hand, the spread of atmospheric ash and nutrients into the non-fire region soils through dry and wet

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deposition depletes soil nutrients in ^{15}N (Soto and Diaz-Fierros, 1992; Grogan et al., 2000; Huber et al., 2013). Based on the aforementioned points, a hypothesis can be formulated that the $\delta^{15}N$ value of charred materials (or BC) has the potential to identify the transport pathways of BC through rivers/streams or atmospheric deposition from fire-prone regions to other regions.

In this study, an attempt has been made to deduce the transport pathway of fire produced BC to a Himalayan lake (Wular Lake) during the late Holocene. For this, $\delta^{15}N_{BC}$ was measured in the Wular Lake core covering the time frame from 3744 to 317cal years BP. Verma et al. (2023) utilized the same core sediment samples to reconstruct the historical records of forest fires and precipitation using BC concentrations and C isotopic composition. In a parallel investigation, Shah et al. (2023) conducted a study to explore the changes in lake carbon and nitrogen biogeochemistry that occurred due to shifts in climatic conditions during similar time period. Additional paleoenvironmental studies have been carried out in the Wular Lake using various proxies such as major oxides, grain size, and stable isotopes (Shah et al., 2020, 2021). These studies have provided insights into the changes in lake level, erosion and weathering history as well as lake biogeochemistry during the Holocene. Collectively, these diverse investigations have revealed a period of relatively drier climate conditions between 3744 and 1500 cal years BP with particularly arid condition around 2500 cal years BP. This aridity transitioned into a wetter climate that continues up to the present time.

Presently, the lake exhibits characteristics of both lotic (riverine) and lentic (still water) systems (Ganai and Parveen, 2014). During high precipitation, the lake receives a substantial amount of water through the Jhelum River, effectively becoming part of the river system (Meer et al., 2022; Shah et al., 2020). Conversely, during drier climate conditions, Wular Lake does not receive water from the river and remains isolated (Shah et al., 2020; Meer et al., 2022). This behaviour of the lake makes it an ideal study site to investigate diverse pathways of BC to the lake. Therefore, this study focused on (i) understanding terrestrial N dynamics due to climate change during late Holocene and (ii) identifying the pathways of BC to the lake by measuring $\delta^{15}N_{BC}$ in the lake sediment.

2. Material and methods

2.1. Study area and sampling

Wular Lake (34°23′ N and 74°32′ E) is one of the largest fresh water lakes in India, situated in the Bandipora district of the Union Territory of Jammu and Kashmir, India (Fig. 1). It is located at an attitude of 1580 m above sea level (masl) and has a maximum length of ~16 km and breadth of ~7.6 km, with an area of ~189 km^2. It is a shallow lake, with an average water depth of ~3 m. The majority of water in this lake is received from the Jhelum River (Mushtaq and Pandey, 2014; Shah et al., 2017). This study was conducted on a 2.5 m long sediment core extracted using PVC pipe from the Wular Lake in July 2019 (Fig. 1). The core was subsampled with a resolution of 2 cm and the chronology of the core was established using ^{14}C dating technique. The radiocarbon ages of the core and measurement techniques are already published (Verma et al., 2023).

2.2. Extraction of black carbon and isotopic measurements

The protocol for extracting BC from lake sediment in this study was based on the methodologies established by Lim and Cachier (1996). The sediment samples were initially dried in an oven and finely ground into a powder. The powdered samples were decarbonated using HCl and were subsequently centrifuged with ultrapure water (Milli-Q) to neutralize the acid. Next, the samples were treated with a mixture of HF (10 M) and HCl (1 M) at room temperature to dissolve silicate and resolve any superficial carbonate that might be trapped between silicate particles. The samples were once again subjected to centrifugation for neutralization.

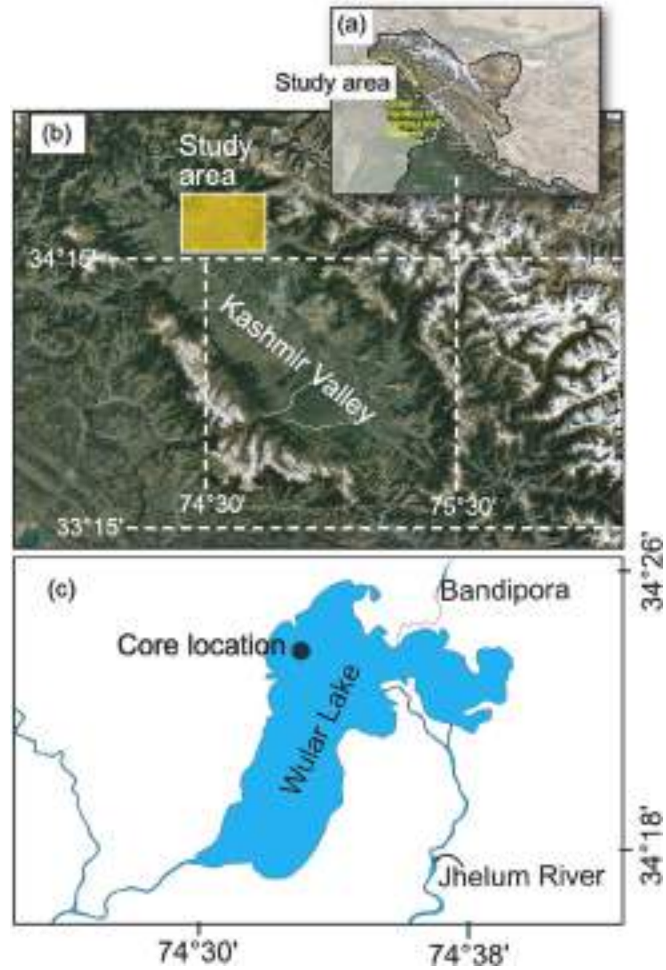

Fig. 1. (a) Map of the Union Territory of Jammu Kashmir, India, (b) map of the Kashmir valley with yellow box showing the location of the study area, i.e., Wular Lake, and (c) location of the core in the Wular Lake. Figures modified after Verma et al. (2023). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

To remove soluble organic matter and kerogen, the samples were treated with a solution of $K_2Cr_2O_7$ (0.1 M) and H_2SO_4 (2 M) at 55 °C for 60 h or until the solution stopped changing color from orange to green. The change in color of the reagent indicated the reduction of dichromate ions, reflecting the completion of the reaction. Finally, centrifugation was performed with ultrapure water to eliminate any traces of the chemicals used and neutralize the samples. Following decantation, the residual samples, considered as BC (Wang et al., 2019), were dried in an oven overnight for mass spectrometric analysis.

The extracted BC samples were analysed for N isotopic composition using an isotope ratio mass spectrometer (Delta V Plus; Thermo Fisher Scientific) connected to an Elemental Analyzer (Flash 2000; Thermo Fisher Scientific) via a Conflo system. Ammonium sulphate (IAEA-N-2; $\delta^{15}N = 20.3 \pm 0.08$‰; N content ~21.2%) was used as standard. Protein (IVA-OAS; $\delta^{15}N = 5.94 \pm 0.08$‰) was also used regularly to confirm the instrument precision with time. The analytical precision for repeated measurements of standard were less than 0.3‰ for N isotopic compositions and less than 10% for N %.

All the samples extracted for BC from the studied core have been analysed earlier for carbon isotopic composition of BC to understand paleofire in the region (Verma et al., 2023). Due to very low N content in some of the extracted BC samples, N isotopic measurements could not be performed with certainty.

3. Results

The content of BC associated N (BC-N) in sediment samples varied between 0.001 and 0.035% with an average of 0.014 ± 0.079% (Fig. 2a and b). The proportion of BC-N within total N (TN) in sediments ranged from 0.4 to 15%. The ratios of BC % and BC-N % (hereafter C/N ratio) in the sediments exhibited a broad range spanning from 3 to 31, with the majority falling between 15 and 25 (Fig. 2c and d). No discernible similarity was observed in variability of C/N ratio with other parameters. No significant correlation was observed between BC-N and TN ($r^2 = 0.05$, $p > 0.05$; Fig. 2e), whose trends also lacked similarity (Fig. 2b).

There was a notable positive correlation between BC-N % and BC % ($r^2 = 0.52$, $p < 0.05$; Fig. 2f). Additionally, BC-N % showed a negative relationship with $\delta^{15}N_{BC}$ ($r^2 = 0.10$, $p < 0.05$; Fig. 2g). The $\delta^{15}N_{BC}$ also showed a poor negative correlation with BC % ($r^2 = 0.06$, $p < 0.05$; Fig. 2g) and no correlation with $\delta^{13}C_{BC}$ (Fig. 2h).

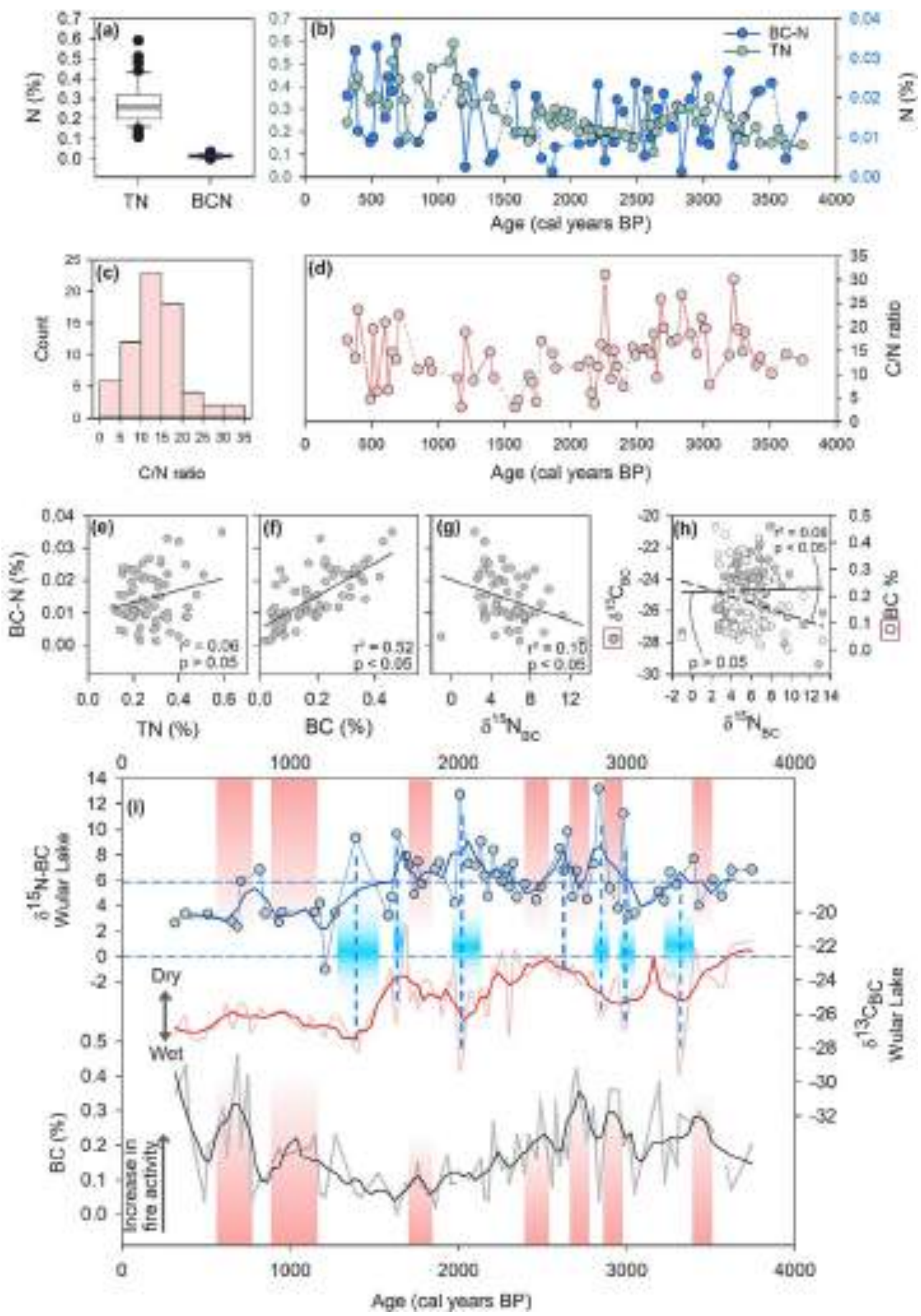

Fig. 2. (a) Box plot of TN (%) and BC-N (%), (b) temporal variability of BC-N (%) and TN (%), (c) frequency (number of samples) plot of C/N ratio of BC, and (d) temporal variability in C/N ratios of BC. Cross plots of (e) BC-N (%) – TN (%), (f) BC-N (%) – BC (%), (g) BC-N (%) – $\delta^{15}N_{BC}$ and (h) $\delta^{15}N_{BC}$ – $\delta^{13}C_{BC}$ (grey dots and black solid line) and $\delta^{15}N_{BC}$ – BC % (white dots and dotted black line). Comparison of (i) $\delta^{15}N_{BC}$, $\delta^{13}C_{BC}$, and BC % of the Wular Lake sediment core. The $\delta^{13}C_{BC}$ and BC % are from Verma et al. (2023). The dark blue, red, and black lines represent a 3-point running average of $\delta^{15}N_{BC}$, $\delta^{13}C_{BC}$, and BC%, respectively. The light red shades represent relatively intense fire periods, while the blue shades indicate wetter climate periods with lower fire incidence in the study region. The dotted blue lines indicate concordant peaks of low $\delta^{13}C_{BC}$ and high $\delta^{15}N_{BC}$. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

Ranging from −1.0 to ~13.2‰ ($n = 62$ samples), $\delta^{15}N_{BC}$ showed a large fluctuation (Fig. 2i) during the late Holocene with an average of 5.8 ± 2.4‰. Most of the values, however, varied between 2 and 8 ‰. Overall, $\delta^{15}N_{BC}$ showed relatively higher values during 3744 –1500 cal years BP (close to the average) compared to 1500–317 cal years BP (most of the values between 2 and 4‰). Specifically, relatively higher $\delta^{15}N_{BC}$ occurred at 3400, 3000, 2850, 2000, 1600, 1400, and 800 cal years BP, whereas periods close to 3500, 2900, 2500, 1800, 1000, and 600 cal years BP showed lower $\delta^{15}N_{BC}$ (Fig. 2i).

4. Discussion

In this study, BC-N contents were 0.4 to 15% of TN, suggesting significant removal of non-BC N through the oxidation method followed here for BC extraction. The C/N ratios during this study (between 3 and 31) was lower than a previous study (Wang et al., 2019), where it varied between 20 and 59. Although high C/N ratios have been reported for the biochar material used for agriculture purposes (Cayuela et al., 2010; Kirkby et al., 2014), a range of values have been reported in literature. Low C/N ratios for Lolium (~ 7) and Casein (~ 4) char (Knicker et al., 2008), pyrogenic organic matter (~ 10; Soong and Cotrufo, 2015) and burnt forested soils (< 10; Garcia-Oliva et al., 1999; Matosziuk et al., 2020) have been reported. Based on the present and earlier studies, it appears that a range for C/N ratios for BC exists and isotopic composition of N associated with BC can be used as a potential proxy to study terrestrial N dynamics as well as transportation pathways of BC. Additionally, there are very few studies available that elucidate the flow of N associated with BC through various ecosystems during and after a fire, which this study deciphers.

4.1. Role of climate on controlling terrestrial N dynamics in the Kashmir Valley

The $\delta^{15}N$ signature of terrestrial biomass is reflected in the BC generated through the partial combustion of that biomass (Wang et al., 2019). It is known that climate plays a significant role in determining the $\delta^{15}N$ of plants (Amundson et al., 2003; Craine et al., 2015; Garten, 1996). Generally, plants exhibit high $\delta^{15}N$ under a drier climate with relatively lower values occurring during wetter conditions (Handley et al., 1999). In the present study, the overall trend of $\delta^{15}N_{BC}$ aligns with the trend of $\delta^{13}C_{BC}$ (Verma et al., 2023), indicating climate to be the primary factor influencing the N cycle in the Kashmir Valley (Fig. 3). It suggested here that changes in water availability in the Kashmir Himalayan region influenced plants N dynamics. During drier condition, which was determined to have occurred between 3744 and 1500 cal years BP in an earlier study (Verma et al., 2023), the limited availability of N affected N isotopic composition of plants by reducing isotope discrimination during uptake, leading to higher $\delta^{15}N$ (Soto and Diaz-Fierros, 1992; Huber et al., 2013).

In addition, it has also been reported that during drier climate, microbial activity in the soil is often reduced due to limited moisture (Manzoni et al., 2012) affecting processes such as nitrogen fixation, nitrification, and denitrification (Baldwin and Mitchell, 2000). This can result in decreased N transformations and nutrient availability in the soil (Baldwin and Mitchell, 2000). As a result, plants may rely more on organic N sources (Gioseffi et al., 2012), such as amino acids or N compounds with higher $\delta^{15}N$ (3–7‰; Philben et al., 2018), leading to increase in $\delta^{15}N$ of the plant tissue. During the period of relatively increased rainfall between 1500 and 317 cal years BP (Verma et al.,

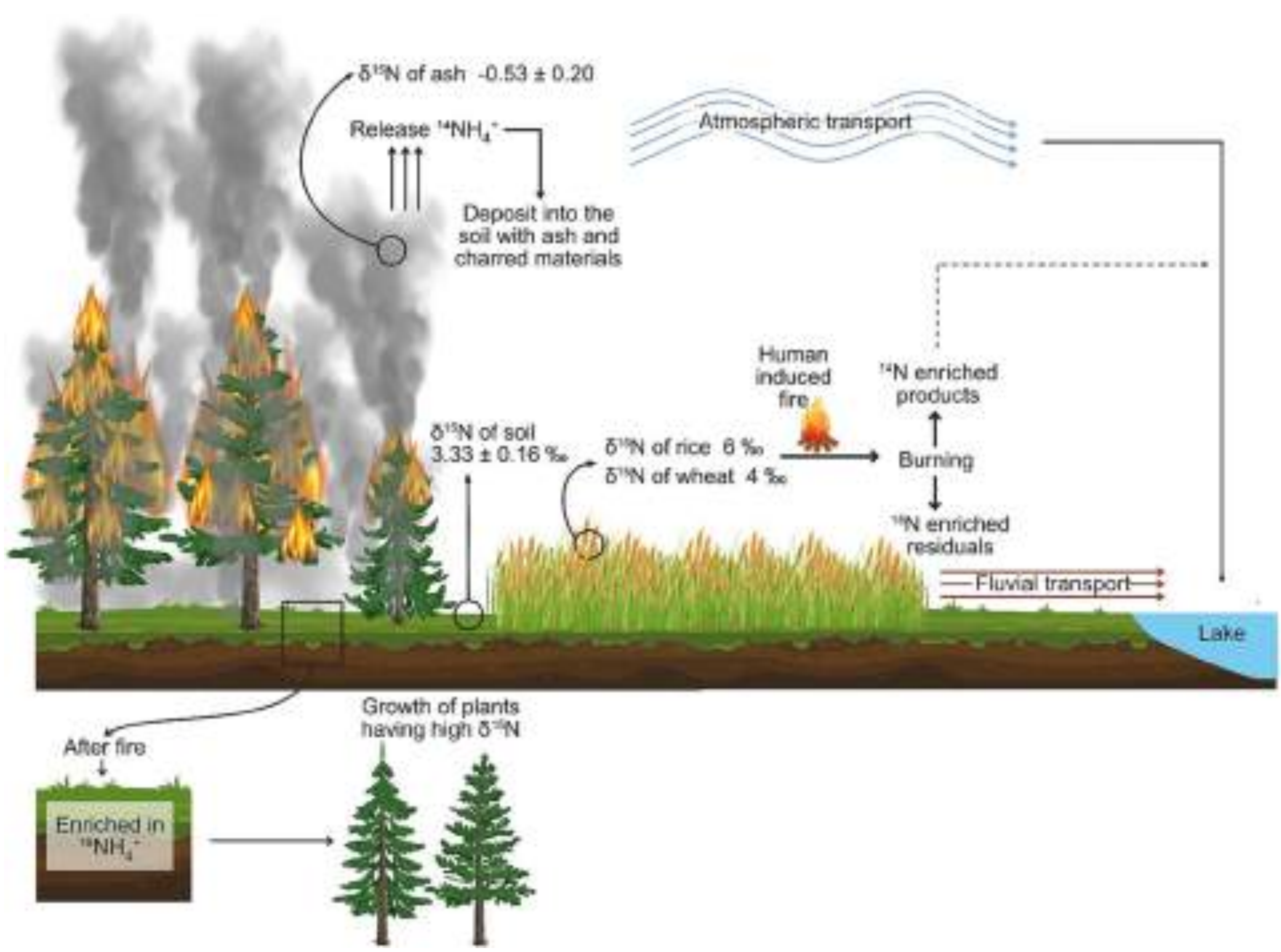

Fig. 3. Simplified **c**onceptual diagram of N dynamics in fire prone region.

2023), there was likely a higher availability of nutrients. This increased availability might have led to an increase in isotopic fractionation, resulting in a lowering of the $\delta^{15}N$ in plants (Handley et al., 1999). As a result, the produced BC would have relatively lower $\delta^{15}N$ during wetter climate compared to values during the drier climate conditions (Fig. 2). However, no significant statistical correlation was observed between $\delta^{13}C_{BC}$ and $\delta^{15}N_{BC}$, likely due to inputs from various BC sources (each with different $\delta^{15}N$) into the lake. This suggests that forest fires in the region, along with the associated BC production, played a pivotal role in determining the $\delta^{15}N$ of burnt materials. These materials are transported to the lake via atmospheric and terrestrial pathways, both of which are influenced by the region's climatic conditions.

4.2. Pathways of black carbon into the lake

Apart from the impact of climate on overall N dynamics in the Kashmir Himalaya, the fluctuations observed in the $\delta^{15}N_{BC}$ values strongly imply a difference in transport pathway of pyrogenic material from one location to another (Fig. 2). Previous studies have provided strong evidence that fire plays a significant role in disturbing nutrient dynamics (Grogan et al., 2000; Aranibar et al., 2003; Huber et al., 2013). Generally, it has been observed that fire increases the $\delta^{15}N$ values of charred material due to the loss of N-containing ash material enriched in ^{14}N (Saito et al., 2007; Huber et al., 2013). It has also been suggested that during high-intensity fires, the rapid and complete combustion of plant material into ash results in almost complete loss of N, causing minimal isotopic fractionation of ^{15}N (Huber et al., 2013). On the other hand, during low-intensity fires, the combustion process is slower and incomplete, leading to a partial loss of N with potentially higher isotopic fractionation of ^{15}N (Huber et al., 2013). Some of studies have shown export of ^{15}N depleted NO_3^- from burnt soil to cause enrichment of residual N (Herman and Rundel, 1989; Grogan et al., 2000; Huber et al., 2013). Additionally, fire stimulated nitrification was also suggested for enrichment of ^{15}N in NH_4^+, which probably also contribute to the enrichment of N in plants (Johnson et al., 2011). Eventually, there are two components of burnt plants: the first component gets emitted into the atmosphere and subsequently deposited in adjacent areas through both wet and dry deposition exhibiting low $\delta^{15}N$ (Huber et al., 2013). On the other hand, the second component, characterized by high $\delta^{15}N$ remains in the soil and is transported by water during periods of heavy precipitation (Huber et al., 2013; Mukherjee and Kumar, 2021) (Fig. 3).

Apart from the larger picture of relatively drier and wetter periods before and after 1500 cal years BP in the regions (Verma et al., 2023), fluctuations were noticed in $\delta^{15}N_{BC}$ throughout. In general, periods with relatively wetter climate at 3400, 3000, 2850, 2000, 1600, 1400, and 800 cal years BP indicated by relatively lower $\delta^{13}C_{BC}$ and BC% aligned with relatively higher $\delta^{15}N_{BC}$. On the other hand, periods close to 3500, 2900, 2500, 1800, 1000, and 600 cal years BP, indicating relatively drier climate through higher $\delta^{13}C_{BC}$ and BC%, exhibited lower $\delta^{15}N_{BC}$ (Fig. 2i). As most of the relatively lower $\delta^{15}N_{BC}$ are coincident with period of relatively drier climate, it indicates that atmospheric BC having low $\delta^{15}N$, was the dominant source of BC to the lake during drier periods (Fig. 2i). Conversely, alignment of higher $\delta^{15}N_{BC}$ with low $\delta^{13}C_{BC}$ indicates transportation of residual burnt materials from soils into the lake via terrestrial pathways during wetter periods.

There were a few peaks that showed very high $\delta^{15}N_{BC}$ (Fig. 2i). In the same samples, very low $\delta^{13}C_{BC}$ were reported with relatively low BC% (Verma et al., 2023). Such high $\delta^{15}N$ were probably due to the slash-and-burn agriculture used during Neo and Mega-lithic periods (Baum et al., 2016). In many ancient agricultural societies, burning fields or crop residues was employed as a means to clear land for cultivation (Rösch et al., 2002; Baum et al., 2016). Reported $\delta^{15}N$ values of modern and fossil rice and wheat grain were close to 6‰ (Kaushal et al., 2019), and burning can increase the $\delta^{15}N$ of the residual (Huber et al., 2013). Additionally, disturbances such as grazing, clear-felling or land use change also cause enrichment of ^{15}N in bulk soil (Koerner et al., 1999; Cook, 2001; Pardo et al., 2002), mostly as a result of enhanced nitrification that discriminate against the heavier isotope (Szpak, 2014). Therefore, it is plausible to suggest that the high $\delta^{15}N$ values were a result of the burning of agricultural waste. This burning process would have contributed significantly to the $\delta^{15}N_{BC}$ signature, especially considering the relatively low occurrence of forest fires, both natural and human-made, in the region.

Overall, it has been observed that during periods of high forest fires (high BC %), BC levels showed lower $\delta^{15}N_{BC}$, regardless of the general climate conditions in the region. This suggests a higher proportion of atmospheric BC deposition into the lake. On the other hand, during periods of low forest fires (low BC %), which coincided with wetter conditions, the lake received residual pyrogenic carbon via runoff resulting in high $\delta^{15}N_{BC}$.

5. Conclusion

The findings of this study indicate that the terrestrial N cycle in the Kashmir Himalayan region is influenced by shifts in climate conditions. The $\delta^{15}N_{BC}$ measurements provide insights into the potential pathways of BC transportation to the Wular Lake. The results specifically suggest that the transportation of BC in the Kashmir Himalayan region varied depending on climatic conditions. During periods high fire activity, indicated by low $\delta^{15}N_{BC}$, BC was dominantly transported through wet and dry atmospheric deposition. Conversely, during wetter periods with lower fire activity, indicated by high $\delta^{15}N_{BC}$ values, riverine transport is suggested to be the dominant mode of BC transportation into the lake.

Author contribution

Abdur Rahman and Rayees Shah participated in the sample collection. Abdur Rahman and Ajayeta Rathi analysed the samples. Abdur Rahman wrote the draft. M. G. Yadava helped set up the methodology. Abdur Rahman and Sanjeev Kumar conceptualized the project. Sanjeev Kumar supervised the project and arranged funding for the study. All authors helped in drafting the text.

Declaration of Competing Interest

Authors report no conflict of interest.

Data availability

Data will be made available on request.

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