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Article

Environmental significance of levoglucosan records in a central Tibetan ice core

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ABSTRACT

The environmental significance of levoglucosan (LEV) records in Tibetan glacier ice layers on sub-annual to annual scales, in particular their suitability as a specific biomarker for indicating past fire changes, is poorly understood at present. In this work, a continuous LEV record was reconstructed in a central Tibetan ice core for the period 1990–2012. The LEV record was classified into two categories based on its LEV concentrations: background levels and extreme events. Annually-resolved LEV records and background levels in the ice core were strongly correlated with satellite observations of fire occurrence frequency over the northern Indian peninsula between 2003 and 2012, especially for strong fire events during the pre-monsoon season (March–May). In addition, peaks in LEV concentrations may represent extreme fire events in regions around the Tibetan Plateau. LEV records in the ice core reflect a long-term increasing trend in fire background and also an increase in fire extreme events, across the Tibetan Plateau and its surroundings. We therefore conclude that LEV records in Tibetan ice cores can be used as a powerful tool for calibrating and reconstructing past fire changes over subtropical Asia.

1. Introduction

The chemical compositions of the individual components of smoke aerosols provide multiple lines of evidence for characterizing fire emissions [1–3]. Levoglucosan (LEV), an organic component, can only be generated from the degradation of cellulose and hemicellulose and accounts for more than 90% of the total dehydration monosaccharides in fire emissions [2]. The lifetime of LEV varies from several hours to more than ten days, under different atmospheric conditions, therefore enabling its long-range transport and global distribution [1–3]. As a specific biomarker, LEV in different matrices (including atmospheric aerosols, precipitation, soil, lake/marine sediment and glacier ice) has been extensively used for indicating fire emission changes [1]. LEV has been widely detected in both polar region ice sheets [4–8] and mid-latitude mountain glaciers [1,9–14], and acts as a major component in glacial geochemistry.

The Tibetan Plateau (TP) is located adjacent to subtropical Asian regions such as south Asia and central Asia [15–17], which are considered amongst the most intense fire emissions sources across the northern hemisphere [11,18]. The TP and its surrounding high-elevation regions, often called "the Third Pole", contain the largest amount of ice outside of the Arctic and the Antarctic [15,17,19]. Fire emissions can be injected into the upper troposphere, occasionally even reaching the lower stratosphere [20,21], and can subsequently be transported and deposited on these high-elevation Tibetan glaciers [11], especially during the major wildfire seasons [13]. Geochemical evidence shows that TP glaciers have been contaminated by LEV at different elevations [11,13,14], and are thought to mainly act as receptors of fire emissions released from surrounding regions [11].

Previous studies have revealed that LEV records in glacier ice layers can provide information about fire emissions changes on time scales ranging from the past several millennia to the last few decades [4,8,16], and can sometimes even capture strong fire event signals [1,6,13]. However, a large concentration of photochemically active groups (e.g., hydroxyl radical and NO_x, etc.) is present in the snow-firn-ice interface on glacier surfaces, which could potentially degrade LEV ([1] and cited references). In addition, levoglucosan is a water soluble component [2]. Strong melting and leaching processes occur on Tibetan glacier surfaces, most notably during summer [22], and may substantially modify the post-depositional LEV distribution in glacier ice layers [13]. These processes must be addressed when assessing the environmental significance of LEV in Tibetan glacier ice and its suitability as a calibration tool for indicating past fire changes, especially on sub-annual to annual time scales [13,16]; however, this issue has received little attention and has not yet been resolved. In this work, we aim to verify whether LEV records in Tibetan glacier ice layers

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can represent regional fire changes, using a continuous LEV record extracted from a central Tibetan ice core.

2. Experimental methods

2.1. Experimental methods and ice core dating

Central Tibetan glaciers have remained relatively stable, in spite of the intensive retreat of the Himalayan glaciers since the late 20th century [15,19]. Furthermore, central Tibetan glaciers are ideal media for studying regional climate and environmental changes owing to the limited human activities [19]. An ice core was extracted from the accumulation area of Zangsegangri Glacier (ZSGR, 34.32 °N, 85.82 °E, 6,070 m above sea level (asl), Figs. 1 and S1 online) in the central TP, during June 2013 [16]. Ice core sections were transported frozen to Lhasa, and stored in a cold storeroom at a temperature of −20 °C. Samples stored under such conditions displayed no apparent degradation of LEV for at least several years [6,10].

Polyethylene terephthalate bottles were washed twice by soaking in ultrapure water for at least 24 h and then rinsing three times with ultrapure water to remove potential contamination. Ice sections were cut at a sub-annual resolution of approximately 3 cm in length. The outer part of each sample was scraped using a pre-cleaned scalpel, and one portion of the inner part was stored in the pre-cleaned polyethylene terephthalate bottles for LEV analysis [10,16].

Ice samples were melted at room temperature (about 15 °C) in a cupboard in a clean room before chemical analysis. For sample preparation, 0.50 mL of each melted sample was extracted and filtered by polyether sulfone filter (0.22 μm mesh size). The filtrate was collected in a 1.50 mL glass vial, and 0.50 mL of acetonitrile was added to each sample before analysis. Sample analysis was performed by a Waters Acquity UPLC system (USA) in the reversed phase mode. For the chromatographic analysis, 5.00 μL of each sample was injected onto a BEH (ethylene bridged hybrid) Amide column (2.1 × 100 mm in length, and 1.7 μm in particle size, Waters, USA). Gradient elution was employed at a flow rate of 0.20 mL min^{-1}. The retention time of LEV was 1.42 ± 0.02 min in ice samples. An Acquity triple quadrupole mass spectrometer equipped with a Z-spray electrospray ionization source was used for determination of LEV. Data were collected in negative ion mode by multiple reaction monitoring. The ion transition *m*/*z* 161/101 was used for quantification of LEV. More detailed information regarding validation of the analytical procedure and performance has been described in our previous study [10].

Seasonal changes in meteorological conditions over the accumulation area of high altitude Tibetan glaciers result in seasonal variations in chemical components (e.g., water isotopes and other proxies), and are often used for counting annual layers in ice core dating [19,23,24]. However, winter precipitation is less than 30 mm at meteorological stations across the central TP, and is lower than the sample resolution in the ZSGR ice core, potentially concealing the seasonal variation of water stable isotope signals [16]. It has been observed that dust storms occur mostly in winter and spring over the central TP regions [20,23], forming dirty layers and insoluble particle peaks [19,23]. In addition, black carbon (BC) aerosols over South Asia are concentrated in winter and the pre-monsoon season, yielding high pre-monsoon BC concentrations in TP glacier ice layers [24]. Ice core dating was thus established by counting the annual variations of dirty layers and dust concentration peaks, while BC variations were used as an additional reference when the dirty layers or particle peaks were unclear. The maxima of β activities and ^{137}Cs caused by the 1963 open-air

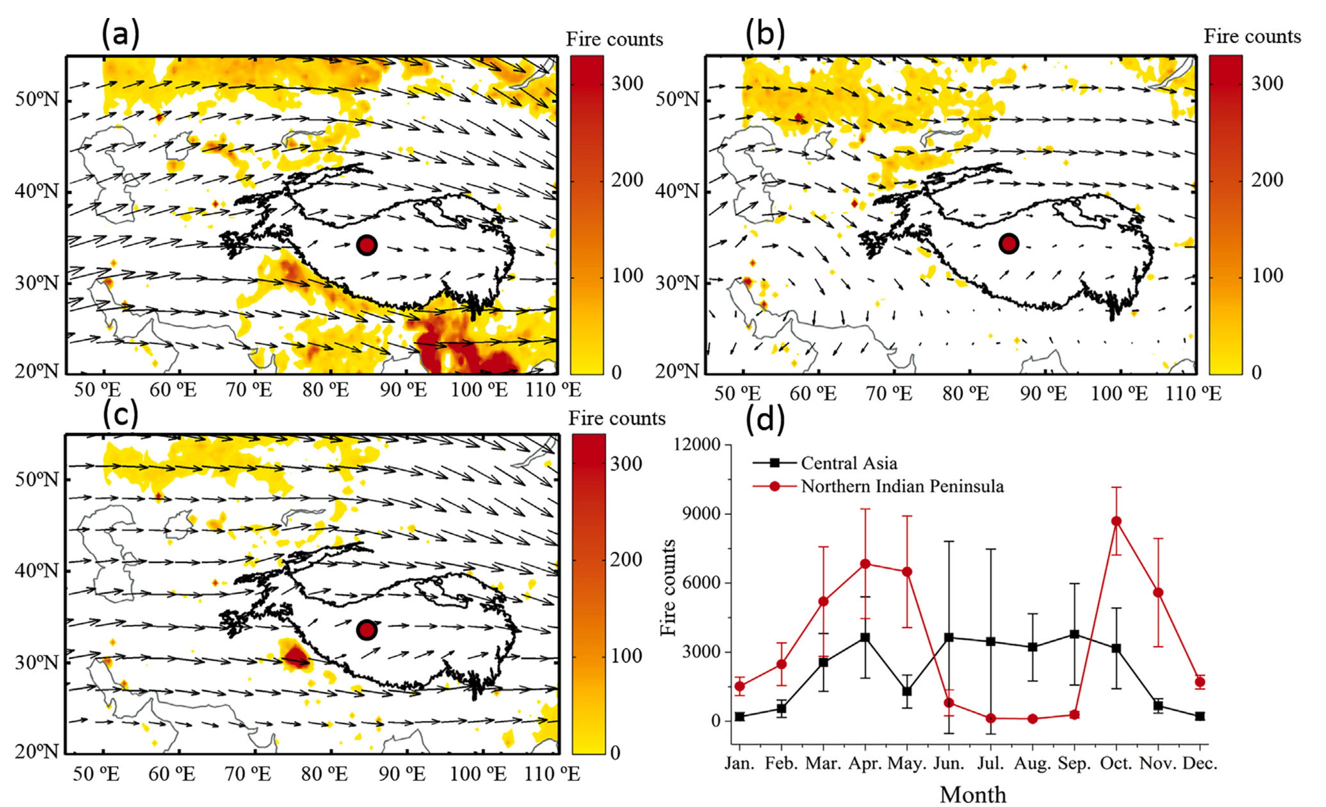

Fig. 1. Averages of wind fields at 500 hPa and fire counts over the TP and its surrounding regions from 2003 to 2012, (a) March to May, (b) June to September, (c) October to November, and (d) monthly variations of mean fire counts over central Asia and northern Indian peninsula, and the error bar indicates ± 1 stdev. The black line indicates the area of TP, and the red circle indicates the location of the ZSGR ice core.

nuclear weapon tests were used as an absolute layer for confirming the dating results [16,19,23]. More details about the ZSGR ice core dating results can be found in our previous work by You et al. [16].

2.2. Modis fire emissions data

Moderate Resolution Imaging Spectroradiometer fire products (MODIS V6 data retrieved from http://modis-fire.umd.edu/, onboard NASA's Terra and Aqua satellites) [18,25] were used for calibration with LEV records from 2003 to 2012. Although MODIS fire counts differ between Terra and Aqua, detected fire variations are broadly consistent [25]. Fire spots with confidences lower than 50% were eliminated (about 13% of Terra and 14% of Aqua) in order to improve reliability. Fire spots with a fire radiative power (FRP) higher than 100 MW were classified as strong fire events [16].

3. Results and discussion

The preliminary LEV concentration results showed an increasing trend ($r^2 = 0.25$, $n = 157$, $P < 0.001$) over the study period (Fig. 2). Thirty-two samples had LEV concentrations lower than the LOD, accounting for about 20% of the samples analyzed in this work. Some samples had extremely high LEV concentrations, and the highest LEV concentration was 8.49 ng mL^{-1} at a depth of 1.70 m, which is more than 9 times the arithmetic mean (0.88 ng mL^{-1}). Fourteen samples had LEV concentrations higher than the mean + 1stdev (1.96 ng mL^{-1}), and were considered to represent strong events; seven samples had LEV concentrations higher than the mean + 2stdev (3.04 ng mL^{-1}) and were considered extreme events (Fig. 2). All of these strong LEV events are concentrated in the upper 2.0 m of the ice core, corresponding to the period from 2006 to 2012, indicating strong LEV events have occurred more frequently in recent years. The annually-based LEV arithmetic mean was 0.92 ng mL^{-1} and displayed an increasing trend from 1990 to 2012 [16] (Fig. 2).

Our previous studies have indicated that central Asia and the northern Indian peninsula are the main LEV sources in the ZSGR ice core [16]. Furthermore, MODIS observations revealed that fires mainly occurred from March to May and October to November over the northern Indian peninsula (20°–35 °N, 65°–90 °E, Fig. 1a, c, d) [11,21], and from June to September over central Asia (35°–50°N, 60°–90°E, Fig. 1b and d) [11]. The wind field analysis at 500 hPa (about 5500 m asl) was used to reveal the possible

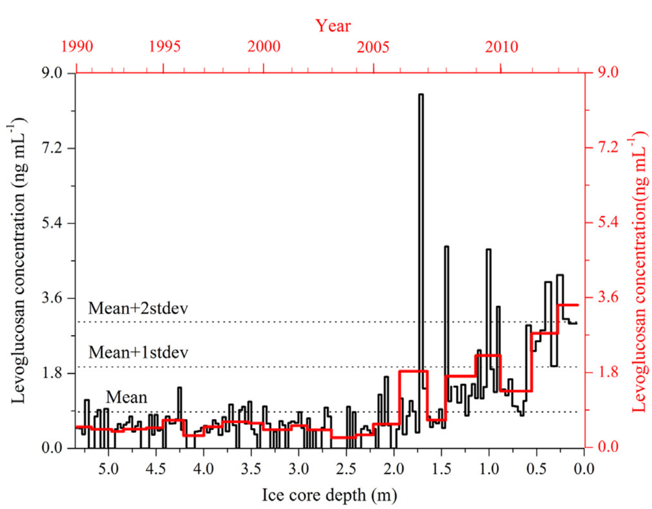

Fig. 2. Preliminary LEV concentration variations with depth (black line, left and bottom axis), and annually-resolved LEV concentration changes (red line, right and top axis) in the ZSGR ice core.

sources of LEV deposited onto the ZSGR glacier. Results showed that air masses that could transport LEV to the central TP and ZSGR Glacier were mostly from the northern Indian peninsula throughout the year, carried by the southern branch of the westerlies (Fig. 1). Air masses from central Asia, especially those from latitudes higher than 40 °N, were mostly deflected by the mid-latitude westerlies above 500 hPa (Fig. 1); however, previous work based on water stable isotopes and geochemical analysis has provided evidence that air masses from central Asia could have an important influence on central Tibetan glaciers [19,23,26].

In order to verify the major source for recent LEV records in the ZSGR ice core, MODIS observed fire count changes over central Asia and the northern Indian peninsula were calculated (Fig. 3). Results showed that central Asia experienced decreasing fire activity over the period from 2003 to 2012. The annual total fire counts displayed a decreasing rate of about 6% per year (Fig. 3a) and the fire counts during the major fire season (June to September) displayed a decreasing rate of about 7% per year (Fig. 3b). In comparison, the northern Indian peninsula showed increasing fire activity during the same period [21], and the annual total fire counts showed an increasing trend at about 8% per year from 2003 to 2012 (Fig. 3a). The fire counts from March to May displayed an increasing rate of about 9% per year and from October to November displayed an increasing rate of about 5% per year (Fig. 3b). The increasing trend is more noticeable for strong fire events with FRP > 100 MW, which increased at a rate of about 21% per year from 2003 to 2012 over the northern Indian peninsula (Fig. S2a online). Furthermore, although total fire counts were reported to be comparable between pre-monsoon (March–May) and post-monsoon (October to November) seasons over the northern Indian peninsula (Fig. 1d and Refs. [11,21]), the pre-monsoon season fires accounted for about 76% of the total strong fire events during the study period (Fig. S2b online). Strong fire events during the pre-monsoon season showed an increasing trend of about 24% per year from 2003 to 2012.

The annually averaged LEV concentrations and fluxes in the ZSGR ice core are highly correlated (both with $P < 0.01$) with fire counts over the northern Indian peninsula (Table 1). However, high LEV concentrations in some years are mostly controlled by sporadic LEV extreme events (Fig. 2). For instance, the year 2006 had a mean LEV concentration of 1.83 ng mL^{-1}, about twice the annual average during the study period, mostly due to one sample at 1.70 m with a LEV concentration of 8.49 ng mL^{-1}. This time corresponded with low fire counts over the northern Indian peninsula in 2006 (Figs. 3 and S2 (online)). In order to reduce the impacts of these extreme LEV events, the annually-resolved LEV background level was calculated after removing extreme LEV values (those higher than mean + 2stdev, equal to 3.04 ng mL^{-1}). The annual LEV background level was strongly correlated with fire counts ($P < 0.001$) over the northern Indian peninsula (Table 1). Results indicated that LEV records in the ZSGR ice core were strongly influenced by fire events, and therefore could be used for calibrating regional fire changes at least on sub-annual to annual time scales. LEV records in the ZSGR ice core can be used as powerful tools for reconstructing historical fire changes.

The equilibrium line altitudes of central Tibetan glaciers range from about 5750–5900 m asl [15]. Despite being relatively stable, glacier surfaces were reported to be melting during summer even within the accumulation area in the central TP [22]. Meltwater could remove LEV from the glacier surface, and transport it through spaces between snow/ice particles, such that LEV becomes concentrated in layers close to the superimposed ice [13]. This process could potentially modify the LEV distribution in glacier ice layers after deposition, and indeed this has been reported on some Tibetan glaciers [13,14]. Once the melt water penetrates the superimposed ice layers, LEV records could be strongly modified and will

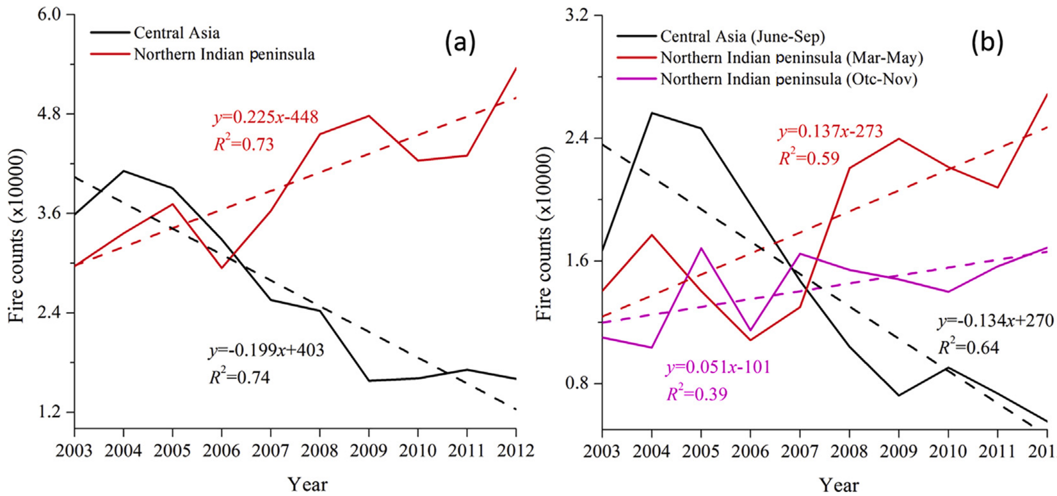

Fig. 3. MODIS-derived (a) annual total fire counts and (b) fire counts during major fire seasons over central Asia and the northern Indian peninsula from 2003 to 2012, and the dash lines indicate the trends.

Table 1
Correlation confidence between fire counts over the northern Indian peninsula and annually based LEV variations in the ZSGR ice core from 2003 to 2012 [a)]

Correlation coefficient (r)	Annually LEV variations		
	concentration	flux	background
Total fire counts	0.76^{**}	0.67^{*}	0.84^{***}
Fire counts with FRP > 100 MW	0.83^{***}	0.77^{**}	0.86^{***}
FRP > 100 MW during pre-monsoon	0.79^{**}	0.72^{*}	0.85^{***}

a) * $0.01 < P < 0.05$; ** $P < 0.01$; *** $P < 0.001$.

vary gradually between neighboring samples [13]; this would render them unsuitable for representing the natural variability in fire occurrence at least on an annual scale. Results showed that the preliminary LEV records displayed event-based characteristics in the ZSGR ice core (Fig. 2), indicating that post-depositional processes on Tibetan glacier surface did not completely alter the LEV distribution in ice layers. For instance, the LEV concentration in the sample at a depth of 1.70 m was 8.49 ng mL^{-1}, yet samples above and below this level all showed LEV concentrations fluctuating close to the mean value (Fig. 2). This isolated extreme value contributed more than half of the annual LEV concentration for 2006 (Fig. S3 (online)). Some similar LEV extreme values can also be seen in the ZSGR ice core (Fig. 2), and may be used to assess the impacts of extreme or strong fire events.

The extreme fire event identified at 1.70 m (year 2006) may be traced back to the central Asia regions (Fig. 4). There were 8 fire counts with FRP > 3000 MW detected in an area near the lakefront of Balkhash Lake, during Aug 2006 (approximately 47.06 °N, 73.24 °E, Table S1 online). Those fire counts were the most concentrated extreme fire events in central Asia from 2003 to 2012. Although the high-elevation Tianshan Mountains and the Pamir Plateau (Fig. 1), which lie along the air mass trajectories, could possibly block the transport of smoke plumes, the wind field at 700 hPa (close to 3,000 m asl, Fig. 4) shows that some of the air masses from central Asia could still be transported to the TP. This phenomenon can be seen more clearly at lower altitudes, for example 850 hPa (close to 1,500 m asl, Fig. 4). Smoke aerosols from central Asia carried by those air masses can be transported and deposited onto Tibetan glacier surfaces, possibly causing the extreme high LEV events in the ice core records. Nevertheless, the influences of those extreme fires should be evaluated using fire-related climate models [27] in future work, and such results do not imply a one-to-one relationship between fire events in the source region and the detected LEV records, because many factors (e.g., atmospheric deposition during atmospheric transport; ice core sample resolution; and other processes) could impact LEV records in glacier ice [1].

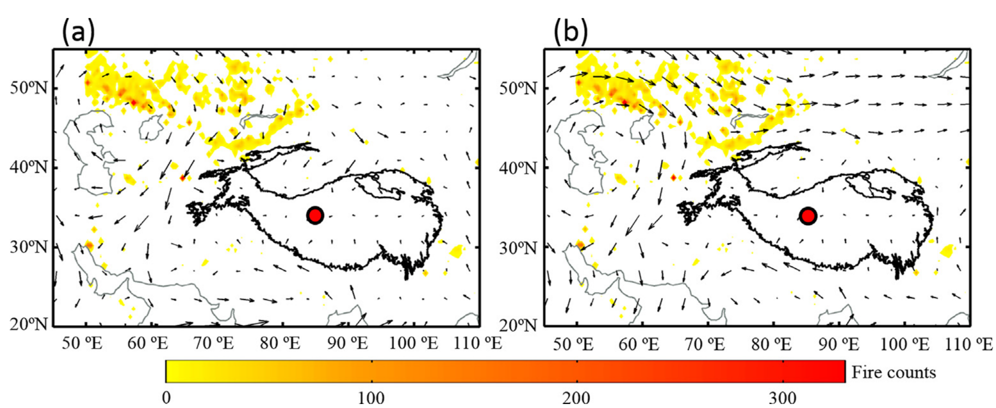

Fig. 4. Wind fields (850 hPa (a) and 700 hPa (b)) and fire counts over the TP and its surrounding regions during August 2006.

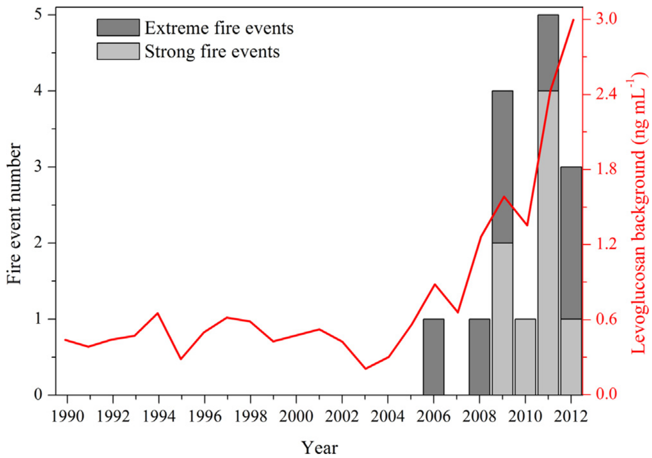

Fig. 5. Variations in fire derived from the LEV record in the ZSGR ice core, from 1990 to 2012: extreme values (columns, left *y*-axis) and background changes (red line, right *y*-axis).

Annually-based LEV concentrations and flux records in the ZSGR ice core indicate a recent increase in fires in TP and its surroundings from 1990 to 2012, in terms of both the long-term trend and fire events. Strong and extreme fire events represented by samples with LEV concentrations higher than the mean + 1stdev ($1.96\ ng\ mL^{-1}$) were all concentrated in the period from 2006 to 2012 (Figs. 2 and 5), indicating that strong fire events have occurred more frequently in recent years. In addition, the annually-resolved LEV background level displayed a significantly increasing trend ($r = 0.69$, $n = 22$, $P < 0.001$, Figs. 5 and S3 (online)), indicating that the increasing frequency of fire events has also strongly affected background levels.

4. Conclusion

The annually-based LEV record extracted from a central Tibetan ice core was found to be highly correlated with satellite-observed fire count variations from 2003 to 2012. Strong fire events over the northern Indian peninsula were revealed to be a major factor regulating annual LEV records in the ZSGR ice core during the study period. Background fire levels showed an overall increasing trend and were accompanied by a greater frequency of fire events in recent years. Extreme fires during August 2006 in central Asia could be responsible for the highest LEV concentration, at a depth of 1.70 m, in the 1990–2012 ice core record. High occurrences of strong and extreme fire events may be responsible for the rapid increase in LEV concentration since 2006.

Results demonstrate that continuous LEV records in an ice core can capture the signals of discrete fire events as well as the long-term trend, and can therefore provide additional fire event information for reconstructing past fire changes. This work provides crucial evidence and new insights into the ability of LEV in Tibetan glacier ice to be used as a tool for extending fire change records over subtropical Asia regions, at least on annual time scales. Future work should pay more attention to the annually-revolved levoglucosan records in ice cores from multi-decadal to millennial scales, which are crucial for understanding ancient fire changes. However, there is still a lack of high resolution evidences up to date.

Conflict of interest

The authors declare that they have no conflict of interests.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.scib.2018.12.016.

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