Science of the Total Environment

# Earthquake, floods and changing land use history: a 200-year overview of environmental changes in southern Siberia as indicated by n-alkanes and related proxies in sediments from shallow lakes

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| Abstract: | The Selenga River basin, located in southern Siberia, is an important component of the Lake Baikal ecosystem, and comprises approximately 80% of the Baikal watershed. The Selenga Delta, is one of the largest inland freshwater floodplains in the world, and plays an important role in the ecosystem functioning of Baikal. The Lake Gusinoye region, is southwest of Lake Baikal and the Selenga Delta, is a more heavily industrialized region within the Selenga River basin. We assessed possible drivers of changes of sedimentary organic matter (OM) composition within two shallow lakes. We focused on individual n-alkanes, one of the most abundant lipids used to provide information on past vegetation. We used multivariate statistics to disentangle changes in the sources of sedimentary OM over time. The depositional OM history of SLNG04B core can be divided in four zones: (i) major influence of non-emergent vascular plants, typically found in transitional environments (ca. 1835 to ca. 1875); (ii) increased influence of grasses/herbs (ca. 1880 to ca. 1910); (iii) transition from non-emergent vascular plants and grasses/herbs to submerged and floating macrophytes and phytoplankton (ca. 1915 to ca. 1945); (iv) maintenance of autochthonous OM from submerged and floating macrophytes and phytoplankton (ca. 1945 to ca. 2014). The depositional OM history of the Black Lake core can be divided in two main zones: (i) major influence of non-emergent vascular plants and submerged and floating macrophytes (ca. 1915 to ca. 1980); (ii) increased influence of grasses/herbs and phytoplankton (ca. 1980 to ca. 2010). Natural events (e.g., an earthquake in 1862 caused flooding and subsidence of much of the land surrounding SLNG04 lake and a further catastrophic flood event in 1897) and anthropogenic activities (e.g., nutrient pollution from expansion of agricultural ) changed the composition of OM resulting in ecological shifts across trophic levels in the Selenga River basin. |
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Pontal do Paraná, December 15, 2022

To: Editor-in-Chief – *Science of the Total Environment*

Dear Editor-in-Chief

We would like to submit the enclosed manuscript, entitled "*Earthquake, floods and changing land use history: a 200-year overview of environmental changes in southern Siberia as indicated by n-alkanes and related proxies in sediments from shallow lakes*" for your consideration for possible publication in *Science of the Total Environment*.

The study assessed the temporal variability of sedimentary organic matter (OM) composition and inputs to two shallow water bodies within the Selenga River basin, an important component of the Lake Baikal ecosystem, classified as a UNESCO World Heritage site as well as a Ramsar site, designated for the conservation and sustainable development of wetlands throughout the world. In this study, sedimentary *n*-alkanes, one of the most common lipids used to provide information on past vegetation, were used to indicate historical changes in the ecological structure of these shallow lakes.

We found that natural events (e.g., an earthquake in 1862 which caused flooding and subsidence around one of the studied lakes and a further catastrophic flood event in 1897) and anthropogenic activities (e.g., nutrient pollution from expansion of agricultural and livestock population) changed the composition of sedimentary OM at the lakes resulting in ecological shifts across trophic levels in the Selenga River basin. These events were well characterized using *n*-alkanes, and confirmed by other sedimentary proxies (e.g., diatoms, macrofossils), emphasising the linkages between these waterbodies, their connectivity to the Selenga River and the changing use of the terrestrial landscape within their catchments.

The shallow lake systems of the Selenga River basin are of critical importance in maintaining the ecosystem health of Lake Baikal and may act as sentinels for recent environmental change. Given the importance of Lake Baikal to the Siberian environment, the protection of its ecosystems is a global concern, especially as recent reports indicate increased accumulation of a variety of contaminants and dramatic changes in the deltaic environment due to global warming. Our study uses organic biomarkers to provide a novel assessment of sedimentary OM dynamics in freshwater floodplain wetlands, as well as a greater understanding of how local environmental changes impact these systems on spatial and temporal scales. As a consequence, we believe it will be of great interest to the broad readership of *Science of the Total Environment*.

Yours sincerely,

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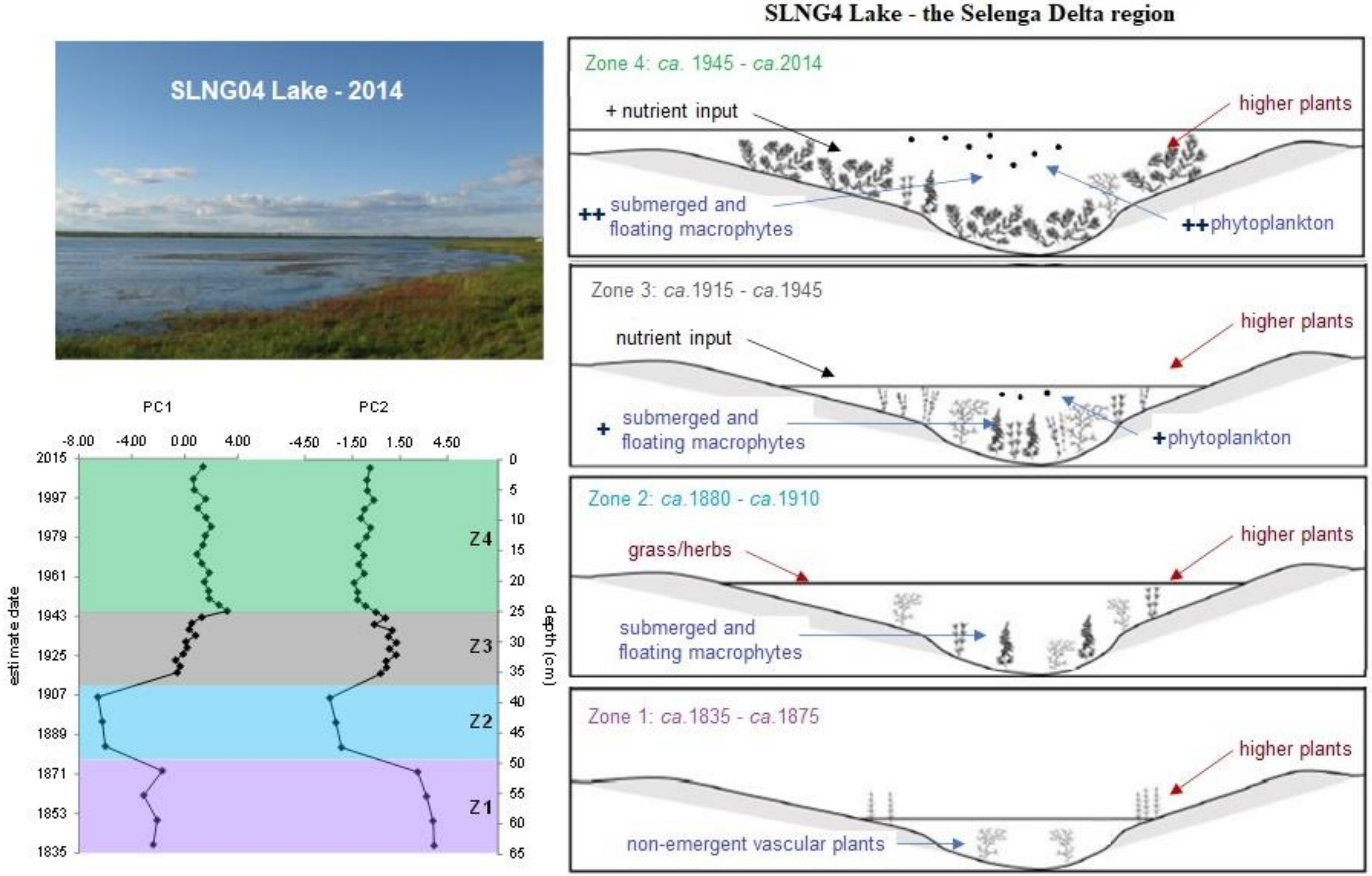
**Graphical Abstract**



## Highlights

> Secular variability on sedimentary organic matter from Siberian lakes is presented

> *n*-alkanes and multivariate data show ecological changes in Siberian lakes

> Changes on sedimentary organic matter are both natural and anthropogenically-induced

> Nutrients from agriculture and livestock changed inputs of sedimentary OM over time

# Earthquake, floods and changing land use history: a 200-year overview of environmental changes in southern Siberia as indicated by *n*-alkanes and related proxies in sediments from shallow lakes

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## Abstract

The Selenga River basin, located in southern Siberia, is an important component of the Lake Baikal ecosystem, and comprises approximately 80% of the Baikal watershed. Within the Selenga River basin, two localized study regions were chosen. The first, the Selenga Delta, is one of the largest inland freshwater floodplains in the world, and plays an important role in the ecosystem functioning of Baikal. It purifies the river waters before they enter the lake, and acts as a refuge for many of Baikal's endemic species. The second location, the Lake Gusinoye region, is southwest of Lake Baikal and the Selenga Delta, and was chosen as a more heavily industrialized region within the Selenga River basin. Anthropogenic activities, including industry, urban settlements, aquaculture and agriculture, have historically increased ecological damage within this area. We assessed possible drivers of changes of sedimentary organic matter (OM) composition within two shallow lakes (SLNG04 and Black Lake), located in the Selenga Delta and the Selenga watershed, respectively. We focused on individual *n*-alkanes, one of the most abundant and common lipids used to provide information on past vegetation. We used multivariate statistics to disentangle changes in the sources of sedimentary OM over time. The depositional OM history of SLNG04B core can be divided in four zones: (i) major influence of non-emergent vascular plants, typically found in transitional environments (*ca*. 1835 to *ca*. 1875); (ii) increased influence of grasses/herbs (*ca*. 1880 to *ca*. 1910); (iii) transition from non-emergent vascular plants and grasses/herbs to submerged and floating macrophytes and phytoplankton (*ca*. 1915 to *ca*. 1945); (iv) maintenance of autochthonous OM from submerged and floating macrophytes and phytoplankton (*ca*. 1945 to *ca*. 2014). The depositional OM history of the Black Lake core can be divided in two main zones: (i) major influence of non-emergent vascular plants and submerged and floating macrophytes (*ca*. 1915 to *ca*. 1980); (ii) increased influence of grasses/herbs and phytoplankton (*ca*. 1980 to *ca*. 2010). Natural events (e.g., an earthquake in 1862 caused flooding and subsidence of much of the land surrounding SLNG04 lake and a further catastrophic flood

event in 1897) and anthropogenic activities (e.g., nutrient pollution from expansion of agricultural and livestock population) changed the composition of sedimentary OM resulting in ecological shifts across trophic levels in the Selenga River basin.

**Keywords:** sedimentary organic matter; molecular biomarkers; isoprenoids; Selenga River basin; environmental changes.

## 1. Introduction

Freshwater floodplain wetlands are some of the most dynamic, diverse, productive and often complex ecosystems on Earth, occurring most extensively in regions where precipitation exceeds evapotranspiration (Mitsch and Gosselink, 2007). They are characterized by regularly flooded land, a highly connective landscape, and shallow lakes of varying degrees of connectivity (Junk et al., 2013). More than 80% of known inland wetland systems have been historically lost, especially during the late $20^{th}$ century (Davidson et al., 2018; Wasserman and Dalu, 2022), due to, e.g., nutrient enrichment from urban and agricultural sources, the construction of dams, stream channelization and dredging for flood control (Hobbs et al., 2016; Sievers et al., 2018).

Climate-induced changes in the hydrological regime are often very important drivers of organic matter (OM) production and ecological variability within freshwater wetlands, and consequently, in the type of sedimentary OM accumulated in shallow lakes. They are also often attributed to changes in precipitation levels, which can result in complete shifts in the vegetation and ecological structure within the wetland resulting in different types of sedimentary OM (Cobbaert et al., 2014; Levi et al., 2016). Therefore, the OM content of lake sediments provides a variety of indicators that can be used to infer histories of environmental conditions of lakes and their watersheds (Rullkötter, 2000; Pancost and Boot, 2004). The primary source of OM to lake sediments is from plants in and around the lake. Plants can be divided into two geochemically distinctive groups on the basis of their biochemical compositions: (1) non-vascular plants such as phytoplankton (e.g., cyanobacteria, algae, diatoms, dinoflagellates) that contain little or no carbon-rich cellulose and lignin, and (2) vascular plants, such as grasses, herbs, shrubs, trees on land, and macrophytes in lakes, that contain large proportions of these tissues (Meyers and Teranes, 2001).

Biomarkers are a group of widespread geochemical compounds present in sedimentary OM and applied in the (paleo)reconstruction of regional climate changes (Freeman and Pancost, 2014). Biomarkers can distinguish between allochthonous and autochthonous carbon sources, and also anthropogenic inputs based on differences in molecular types, functional group positions, and carbon chain length (Derrien et al., 2017; Inglis et al., 2022). Biomarkers can also identify specific types of OM sources over time (Volkman et al., 2008). Amongst biomarkers, *n*-alkanes are one of the most common lipids used to provide information on past vegetation. They are biosynthesized by terrestrial and aquatic higher plants and phytoplankton (Eglinton and Hamilton, 1967; Ficken et al., 2000), and have odd-to-even dominance in the number of carbon atoms. Short carbon chain-length *n*-alkanes ($< C_{20}$) are biosynthesized mainly by bacteria and phytoplankton (Derrien et al., 2017; Liu et al., 2020) while submerged and floating macrophytes (classified as non-emergent vascular plants) are generally dominated by medium-carbon chain-length *n*-alkanes (i.e., $C_{20}$-$C_{25}$). Terrestrial sources are dominated by long-carbon chain-length *n*-alkanes ($> C_{25}$) and have a strong odd-to-even carbon preference (Chen et al., 2016). As a component of vascular plant leaf wax, *n*-alkane distribution is generally dominated by *n*-$C_{27}$ to *n*-$C_{35}$, being more abundant in deciduous trees/shrubs and biosynthesized by grasses/herbs (Schefuß et al., 2003).

The aim of this study was to assess the variability of sedimentary OM composition within shallow water bodies of the Selenga Delta, a designated Ramsar site (N° 682), for the conservation and sustainable development of wetlands throughout the world (Ramsar, 2022), and nearby region of the Selenga River basin. Anthropogenic activities within the Selenga River basin, including industry, agriculture, aquaculture, growing urban settlements, mining, and deforestation, pose threats to water quality and ecology in the Delta and other nearby catchment lakes through both chemical pollution (Adams et al., 2018) and increased erosion of sediments into the Selenga river (Bazhenova and Kohylkin, 2013). Two shallow lakes in the region were selected for biomarker analysis (*n*-alkanes and their related ratios) to evaluate the temporal variations in the input and sources of *n*-alkanes through the last decades. We also tried to answer a specific question: can sedimentary *n*-alkanes be used to indicate changes in the ecological structure of Selenga River basin shallow lakes?

## 2. Study area

*2.1. The Selenga River basin*

The Selenga River basin is located in the continental subarctic of southeast Siberia (Fig. 1), and covers an area of almost 450,000 $km^2$ in Siberia and Mongolia, comprising over 80% of the Lake Baikal watershed (Nadmitov et al., 2015). The Selenga River flows approximately 950 km from the head waters in northern Mongolia before it reaches the Selenga Delta and Lake Baikal. The upstream portions of the Selenga River basin, primarily tributaries and headwaters within Mongolia, run through mountainous and forested terrain, while further downstream, the southern Siberian portion, is dominated by steppe vegetation, underlain by permafrost. Part of this steppe has been converted to pasture or crop land (Chalov et al., 2016). Peak river discharge occurs during the summer, when 98% of total sediment loads are transported through the Selenga River basin (Chalov and Romanchenko, 2016). The region is characterized by an ultracontinental climate, with dry, intensely cold winters and short, mild summers, subjected to westerly and northwesterly winds all year-round (Plyusnin et al., 2008).

The Selenga Delta itself (52°14'N; 106°30'E), has formed where the Selenga River enters Lake Baikal, the oldest and deepest lake in the world and classified as a UNESCO World Heritage since 1996. The delta is heavily influenced by tectonic processes with the oldest portions having been formed over two to three million years ago (Scholz and Hutchinson, 2000).

*2.2. Sampled study sites*

Two shallow lakes were selected for study. The first is located within the Selenga Delta itself ('SLNG04'; 52°16'N, 106°40'E), and the second, located outside of the Selenga Delta, but within the Selenga River basin (named 'Black Lake'; 51°24'N, 106°29'E) (Fig. 2). SLNG 04 is an expansive shallow lake, with surface flow connections to both the Selenga River and Lake Baikal. It is partially surrounded by agricultural lands, consisting primarily of livestock rearing.

Black Lake is one in a chain of several shallow freshwater lakes located approximately 200 km upstream of the Selenga Delta along the Selenga River, and 80 km direct distance southeast of Lake Baikal (Pisarsky et al., 2005). Black Lake is situated in the more heavily industrialized area of Gusinoye, and provides therefore a useful comparison to SLNG04.

## 3. Material and Methods

### *3.1. Extraction of sediment cores*

In March 2014, sediment cores were extracted from SLNG04 and Black Lake ('SLNG04B' and 'BRYT02B', respectively), for organic compound analyses. Further sediment cores ('SLNG04C' and 'BRYT02C') were extracted at the same sites for radiometric dating analyses. An Uwitec piston corer (UWITEC Ltd., Austria) fitted with a 6.3 cm internal diameter perspex tube (*n*-hexane-cleaned to eliminate organic contamination) was used. Sediment cores were collected while lakes were ice-covered, through an auger-drilled hole in the ice. Sediment from these cores was vertically extruded and sliced at 0.5 cm intervals in the field, using *n*-hexane-cleaned equipment and placed in *n*-hexane cleaned aluminum foil before being stored in a plastic bag and sealed. Sediments were stored at -20°C and then freeze-dried in preparation for analysis. Dried sediment from each sample depth was ground to a fine powder using an agate mortar and pestle.

### *3.2. Methods for radioisotope dating of sediment cores*

Radiometric dating techniques were used to date the SLNG04C and BRYT02C sediment cores. The detailed methods were published in Adams et al. (2018). Briefly, wet sediment was subsampled from sediment cores at 3.0-cm intervals and freeze-dried. Dried sediment samples were analyzed for $^{210}$Pb, $^{226}$Ra, $^{137}$Cs and $^{241}$Am by direct gamma assay using ORTEC HPGe GWL series well-type coaxial low background intrinsic germanium detectors. $^{210}$Pb was determined via its gamma emissions at 46.5keV, and $^{226}Ra$ by the 295keV and 352keV gamma rays emitted by its daughter isotope $^{214}Pb$. $^{210}Pb$ chronologies for both sediment cores were constructed using the constant rate of supply (CRS) dating model (Appleby and Oldfield, 1978; Appleby, 2001). Artificial radionuclides $^{137}Cs$ and $^{241}Am$ were measured by their emissions at 662keV and 59.5keV, respectively (Appleby et al., 1986). To obtain dates for the undated cores ('B') loss-on-ignition at 550 °C ($LOI_{550}$) was conducted on all collected sediment cores (Heiri et al., 2001). $LOI_{550}$ profiles from both 'B' and 'C' cores were examined for distinct features (i.e., tie-points) present in profiles from both cores. The radiometric dates for the tie-points were then cross-correlated between the dated and undated profiles as described by Adams et al. (2018).

### *3.3. n-alkanes: extraction, fractionation, instrumental analyses and quality assurances*

The analytical method for hydrocarbons was described in detail by Wisnieski et al. (2016). Approximately 10 g of sediments were Soxhlet extracted for 8 hours with 80 mL of a mixture of dichloromethane (DCM) and *n*-hexane (1:1, v:v) and 100 µL of a surrogate standard mixture containing 1-hexadecene and 1-eicosene (50 ng $\mu L^{-1}$) was added to each sample and blanks prior extraction. This organic extract was reduced to *ca.* 2 mL by rotoevaporation, purified and fractionated using 3.2 g of silica (silica gel 60, 0.063 – 0.200 mm; 5% deactivated with Milli-Q® water) and 1.8 g of alumina (aluminium oxide 90 active, 0.063 – 0.200 mm; 5% deactivated with Milli-Q® water) column chromatography. The samples were eluted with 10 mL of *n*-hexane to obtain the aliphatic hydrocarbon fraction, containing *n*-alkanes and the isoprenoids pristane and phytane. Sample extracts were concentrated to 500 µL and 100 µL of the internal standard 1-tetradecene solution (50 ng $\mu L^{-1}$) was added prior to instrumental analysis.

The hydrocarbons were determined by the injection of 2 µL of the extract into a gas chromatograph Agilent GC (model 7890A) equipped with a flame ionization detector and an Agilent 19091J-413 capillary fused silica column coated with 5% diphenyl/dimethylsiloxane (30 m length, 0.32 mm ID, and 0.25 μm film thickness). Hydrogen was used as the carrier gas. The injector and detector temperatures were adjusted to 280 °C and 325 °C, respectively. Splitless injection mode was adopted. The following oven temperature program was employed for analyses: 40–60 ºC at 20 ºC $min^{-1}$, 60 ºC for 2 min, 60–290 ºC at 5 ºC $min^{-1}$, 290 ºC for 5 min, 290–300 ºC at 5 ºC $min^{-1}$, and 300 ºC for 9 min. The individual compounds were identified by matching their retention times with those obtained for external standard mixture (*n*-$C_{10}$ to *n*-$C_{40}$, pristane and phytane) and quantified using a calibration curve ranging from 0.25 to 10.0 ng $\mu L^{-1}$. The detection limits (DL) were 0.01 µg $g^{-1}$ for individual compounds. These data are based on the lowest instrumental sensitivity of *n*-alkanes concentration (0.02 ng $\mu L^{-1}$, respectively) multiplied by the final extracted volume (500 µL) and divided by the sediment weight (10 g) before extraction.

The procedural blanks showed that no peaks interfered with the analyses of the target compounds. Mean recoveries of the surrogate standards, 1-hexadecene and 1-eicosene, ranged from 50 to 99 % (mean = 66 ± 10, N = 61) and from 56 to 106 % (mean = 78 ± 12, N = 63), for at least 90 and 93 % of samples analyzed, respectively. The precision, expressed as the coefficient of variation between triplicates of the standard reference material (SRM) provided by International Atomic Energy Agency (IAEA-408) was lower than 15 % for at least 85 % of compounds analyzed. The measured concentrations of target parameters available in the IAEA-408 reference sheet (e.g., total *n*-alkanes, *n*-$C_{17}$, *n*-$C_{18}$, pristane and phytane) in the SRM were in good agreement with the certified values (lower than 15 %).

*3.4. Data analysis*

To explore temporal trends in the individual odd *n*-alkanes data (*n*-$C_{15}$ to *n*-$C_{35}$), a constrained hierarchical cluster analysis was applied to a Euclidian distance of normalized values, i.e., based on each individual *n*-alkane concentration divided by the sum of odd total *n*-alkanes (*n*-$C_{15}$ to *n*-$C_{35}$). The clustering of the individual odd *n*-alkane (*n*-$C_{15}$ to *n*-$C_{35}$) data were used to visualize stepwise changes in the *n*-alkane contributions along the cores. The clustered core sections appear to represent environmental changes reflected by different *n*-alkane distributions and were therefore used as 'zones' to be evaluated in detail in each sediment core. These zones could be representative of different predominance of *n*-alkanes, and consequently, sources of sedimentary OM. Broken stick analysis was performed to visualize the residual dispersion of the hierarchical classification, and to determine the number of significant zones within the sedimentary *n*-alkanes profile. The constrained hierarchical cluster and broken stick analysis were performed in R using the *rioja* package (Juggins, 2020)

Principal Component Analysis (PCA) was also adopted as a multivariate approach to verify which individual odd *n*-alkanes (*n*-$C_{15}$ to *n*-$C_{35}$) may best explain the core sections through each core, thereby helping to identify the main sources of OM and their possible variations over the period. PCA was performed using these same normalized data sets (individual *n*-alkanes) in order to indicate which variable(s) may explain the zones formed in the cluster analyses. The PCA was applied and plotted using R and the packages factoMineR and factextra, respectively (Husson et al., 2016).

Box- and whisker plots were constructed to display the trends of *n*-alkanes and ratios across depth zones for each sediment core, with median, first and third quartile, outlier and extreme values indicated. Box-plots were created in the Statistica TIBCO® Software (v.14.0.0).

### *3.5. Diagnostic ratios for OM source identification and depositional conditions*

The odd/even (O/E) ratios, carbon preference indices (CPI), terrestrial-to-aquatic ratios (TAR), aquatic proxy ($P_{aq\ wax}$) and the metric average chain length (ACL) ratio were the *n*-alkane proxies calculated in this study. The isoprenoids pristane and phytane were also included based on their ratios with selected *n*-alkanes. Detailed information regarding equations, diagnostic values and source assignment are presented in Table 1 and as Supplementary Information (Sections 1 and 2).

## 4. Results and Discussion

### *4.1. Core chronologies*

The radiometric dating and loss-on-ignition profiles for both SLNG04C and BRYT02C cores are shown in Supplementary Information (Fig. S1 and S2). The $LOI_{550}$ profiles for the sediment cores 'B' and 'C' from each lake show good agreement (Fig. S1 and S2) and were used to apply dates to undated SLNG04B and BRYT02B cores (Adams et al., 2018). The cross-correlation of $LOI_{550}$ records from cores SLNG04C and BRYT02C with those from SLNG04B and BRYT02B allow chronologies to be allocated extending to *ca*. 1835 (179 years) and *ca*. 1915 (99 years), respectively.

### *4.2. The n-alkane levels and distributions in the studied shallow lakes*

Tables S1–S2 display the *n*-alkanes and isoprenoids concentrations. The sum of individual *n*-alkanes from *n*-$C_{14}$ to *n*-$C_{35}$ (Total *n*-Alk ($C_{14}$-$C_{35}$)) concentrations ranged from 2.02 to 61.5 μg $g^{-1}$ (median = 28.2 ± 12.0) in SLNG04B and from 35.9 to 106.9 μg $g^{-1}$ (median = 60.1 ± 13.8) in BRYT02B (Fig. S3). These levels are considered high when compared to sediments from a paleothermokarst lake on the Bykovsky Peninsula, northeastern Siberia (0.6 - 20.8 μg $g^{-1}$; Jongejans et al., 2020) and surficial sediments from Lake Baikal collected in summer 2001 (4 - 55 μg $g^{-1}$; Russell and Rosell-Melé, 2005).

In general, the analyzed samples for both cores presented significant levels of long-chain *n*-alkanes (LC *n*-Alk; *n*-$C_{27}$ to *n*-$C_{32}$) followed by mid-chain (MC *n*-Alk; *n*-$C_{21}$ to *n*-$C_{26}$) and short-chain *n*-alkanes (SC *n*-Alk; *n*-$C_{15}$ to *n*-$C_{20}$) (Tables S1 and S2; Fig. S3). The contribution of the SC *n*-Alk in the Total *n*-Alk is low in the lower core sections, likely due to high lability of the low molecular weight hydrocarbons. A unimodal distribution of *n*-alkanes is observed in all samples analyzed, reaching a maximum between *n*-$C_{27}$ to *n*-$C_{33}$. The *n*-$C_{29}$ was the predominant *n*-alkane in SLNG04B, while *n*-$C_{27}$ was dominant in BRYT02B (Fig. S4). This *n*-alkane distribution is typical for Holocene age sediment in both Lake Baikal (Brincat et al., 2000; Russell and Rosell-Melé, 2005) and in Lake Billyakh, northern Siberia (Tarasov et al., 2013).

*4.3. Changes in the sedimentary OM composition and ecological structure based on n-alkane and isoprenoids proxies in the studied shallow lakes*

*4.3.1. The SLNG04 lake*

The SLNG04B record can be divided in 4 zones from the bottom to the top (Fig. 4) based on cluster and broken stick analyses (Fig. S5 and S6). These four distinct zones correspond to variations in the PCA scores (Fig. S7) and along depth (Fig. S8).

• Zone 1 (*ca.* 1835 to *ca.* 1875; 63 – 50 cm)

Zone 1 is primarily influenced by grasses/herbs (high relative percentages of $n$-$C_{31}$, $n$-$C_{33}$ and $n$-$C_{35}$; Fig. S9) and non-emergent vascular plants ($n$-$C_{25}$ and $n$-$C_{27}$; Fig. S9). Zone 1 presented relatively low concentrations of all groups of *n*-alkanes (SC, MC and LC *n*-Alk), reflective in the low values of Total *n*-Alk (Fig. 3). This is consistent with relatively low levels of $LOI_{550}$ at this time. The combined Total *n*-Alk and $LOI_{550}$ records during the early- to mid-19$^{th}$ century may indicate a time during lake development, progressing from a marginal semi-aquatic or terrestrial site to a lake basin. In fact, the main distinction between this stage and the rest of the core is related to the major influence of $n$-$C_{25}$ and $n$-$C_{27}$, related to non-emergent vascular plants, typically found in a semi-terrestrial or marshy ecosystem transition (Luoto et al., 2017), also corroborated by $P_{aq\ wax}$ values (< 0.35) (Fig. 5) associated with a mixed source. This particular individual *n*-alkanes contribution is reflected in the lower values of ACL found in Zone 1 (Fig. 5). Low concentrations of SC and MC *n*-Alk in Zone 1 indicate a low contribution from autochthonous OM sources (Fig. 3). The absence of submerged aquatic macrophytes and aquatic animal remains, an absence of preserved algal pigments, and very low diatom concentrations were also verified in this part of the core (Adams, 2017), and are consistent with the individual *n*-alkanes distribution.

The low preservation of OM in the Zone 1 is corroborated by the relatively low values of O/E and CPI diagnostic ratios in this stage (outliers to $CPI_1$, Fig. S10 and S11), and the high values to TAR (outliers, Fig. S12) compared to middle and top core sections (Fig. 5). The higher exposure to light and air at SLNG04 lake prior to the mid-19th century increased the degradation of labile components of sedimentary OM (Reuss et al., 2005), and consequently, isoprenoids (pristane and phytane) (Fig. 6) and pigments (Adams, 2017). Low levels of preserved OM content and low *n*-alkanes concentrations may be tied to depositionary oxic conditions, as indicated by the maximum Fe/Mn and minimum Ca/Ti ratios that occurred in this period (Adams, 2017). On the other hand, the LC *n*-Alk that are more resistant to degradation were also present in relatively low concentrations, and may be related to low values of $LOI_{550}$ in the bottom of the core.

• Zone 2 (*ca.* 1880 to *ca.* 1910; 47 – 38 cm)

Zone 2 is influenced by grasses/herbs and higher plants (Fig. S9). Both Zones 1 and 2 are under strong influence of terrestrial sources of organic matter. Low concentrations of SC and MC *n*-Alk concentrations suggests the low contribution of aquatic sources; however, this may also be due to higher degradation rates of these compounds.

Zone 2 presented a slight increase in concentrations of LC *n*-Alk (and consequently in Total *n*-Alk) compared to the Zone 1 (Fig. 3); it was consistent with the increase of $LOI_{550}$ values found in this section of the core, which reached the maximum OM content of the core at 38 cm (*ca.* 1910).

The main distinction between this zone and the rest of the core is related to the major influence of *n*-$C_{31}$, *n*-$C_{33}$ and *n*-$C_{35}$ in the Total *n*-Alk concentration, that are related to grasses and herbs (Fig. S9). This contribution is well marked due the higher ACL values (outliers, Fig. S12) found in Zone 2. The environmental changes in Zone 2, indicated by the *n*-alkanes and related diagnostic ratios (TAR and ACL; Fig. 5), reflected changes in the allochthonous OM input. The deposition of allochthonous OM from vascular plants, grasses and herbs in the Selenga Delta created a deeper open water zone, now hospitable to diverse communities of submerged and floating macrophytes. The low preservation of OM observed during Zone 1 continued into Zone 2, indicated by the relatively low values of O/E and CPI diagnostic ratios in this stage (Fig. 5).

The low levels of *n*-$C_{17}$ and *n*-$C_{19}$, primarily related to phytoplankton, is consistent with the absence of preserved diatoms frustules during this period (Adams, 2017). Shallow depths at this time potentially led to increased turbulence, increased mixing, and increased wave-action, and consequently, the degradation of sedimentary OM. Biological parameters reinforced the environmental changes in Zone 2 due to the slight increase in diatom concentrations, consisting mainly of epiphytic and small benthic diatoms (Adams, 2017). These geochemical and biological features suggest the transition period to the permanent inundation of Selenga Delta (Gell et al., 2007), characterized by a small, shallow-water, high-closure system with mostly littoral and epiphytic habitats (Hay et al., 2000; Summers et al., 2017).

• Zone 3 (*ca.* 1915 to *ca.* 1945; 35 – 25 cm)

Zone 3 marks a transition on the main components of sedimentary OM, from non-emergent vascular plants (*n*-$C_{25}$ and *n*-$C_{27}$) and grasses/herbs (*n*-$C_{31}$, *n*-$C_{33}$) to submerged and floating macrophytes (*n*-$C_{23}$ and *n*-$C_{25}$) and phytoplankton (*n*-$C_{17}$, *n*-$C_{19}$) (Fig. S9). This is primarily shown by the increase of $P_{aq\ wax}$ index and, also by a decrease in TAR values (Fig. 5). In addition, the $CPI_2$ values are typical of terrestrial sources associated with higher plants (*n*-$C_{29}$), that are an important OM source throughout the core (Fig. 5).

Zone 3 showed a clear increase in concentrations of different groups of *n*-alkanes compared to the previous zones and is consistent with the concurrent variation in $LOI_{550}$ (Fig. 3). Similar concentrations of SC and LC *n*-Alk compared to Zone 2 were detected. An increase in MC *n*-Alk was noted in the lower sections of this zone (*ca*. 1915 to *ca*. 1925; 35 – 32 cm), while a fast and prominent increase in *n*-alkane concentrations in the upper sections occurred (*ca*. 1925 to *ca*. 1945; 32 – 25 cm) (Fig. 3). The decrease in ACL in this zone may represent an increase in sedimentary *n*-$C_{29}$ as a consequence of deforestation (Meyers, 2003; Aichner et al., 2010).

There was an increase in the preservation of OM in Zone 3, indicated by increased values of O/E and CPI diagnostic ratios compared to previous zones (Fig. 5). This zone contained the first detectable levels of isoprenoids. Higher values of Pri/Phy, Pri/*n*-$C_{17}$ and Phy/*n*-$C_{18}$ ratios were obtained compared to the rest of the core (Fig. 6 and S13) suggesting an increase in anoxic conditions and corroborated by minimum Fe/Mn ratios *ca.* 1920 (Adams, 2017).

- <u>Zone 4</u> (*ca.* 1945 to 2014; 25 – 0 cm)

Zone 4 showed the highest concentrations of different groups of *n*-alkanes and $LOI_{550}$ (Fig. 3). Fresh aquatic OM from phytoplankton (*n*-$C_{17}$ and *n*-$C_{19}$) and submerged and floating macrophytes (*n*-$C_{21}$ and *n*-$C_{23}$) play an important role in the sedimentary OM in this most recent zone. Notably, we observed an increase followed by a slight decrease in Total *n*-Alk concentration and $LOI_{550}$ between *ca.* 1945 to *ca.* 1980 (25 – 11.5 cm) (Fig. 3), after which levels remained slightly higher through to the top of the core.

Highest levels of Total *n*-Alk, particularly SC and MC *n*-Alk, are expected to be found in the more surficial sections of sediment cores due the recent input of fresh autochthonous OM (Hedges et al., 1997). Also, the individual *n*-alkanes distribution within the PCA confirmed the establishment of current connectivity and sedimentary environmental, with a greater contribution of autochthonous OM from submerged and floating macrophytes (*n*-$C_{23}$ and *n*-$C_{25}$) and phytoplankton (*n*-$C_{17}$, *n*-$C_{19}$). This is also evidenced by the lowest TAR values of the entire core and the constant $P_{aq\ wax}$ values (Fig. 5). However, the main contribution continued to be from higher plants (*n*-$C_{29}$) due to the connection to the Selenga River, and the influx of river-derived terrestrial matter. The slightly higher values of ACL, when compared to Zone 3, confirm this (Fig. 5).

Trends in total *n*-Alk are consistent with biological parameters that indicate maximum lake productivity levels in the late-1950s, notably pigment concentrations in the SLNG04 lake (Adams, 2017). Increasing SC *n*-Alk up to *ca.* 1965, are in agreement with greater diatom concentrations at *ca.* 1960 (Adams, 2017) as phytoplankton are an important source of *n*-$C_{17}$ and *n*-$C_{19.}$ Changes in macrophyte assemblages can influence changes in other biological communities, including diatoms (Sokal et al., 2010), and may be reflected in the distribution of individual *n*-alkanes, and related indices such as O/E (< $C_{24}$), $CPI_{1}$ and $P_{aq\ wax}$ (Fig. 5).

Changes in delta morphology in the late-20$^{th}$ and early-21$^{st}$ centuries caused a shift in discharge from the southwestern to the northeastern branches of the Selenga River Delta (Chalov et al., 2016), with severe implications for sediment and chemical transport and river water flow. Connectivity to the river dictated the abundance and diversity of the diatoms present (as indicated by the high contribution of SC *n*-Alk to the sediment) with a higher diversity reflecting higher connectivity sites and the presence of planktonic species in lakes connected to the Selenga River. Therefeore, it is also possible that decreased macrophyte biomass (indicated by low contribution of MC *n*-Alk) at high connectivity sites is related to increased connectivity with the Selenga River.

The regular and relatively high values of O/E and CPI found in this zone are related to fresh and/or preserved OM, and despite the recent increase in human activities in the Selenga River basin, the hydrocarbons found may be primarily related to biogenic sources. The isoprenoids ratios (Pri/Phy, Pri/*n*-$C_{17}$ and Phy/*n*-$C_{18}$) showed decreasing values in this zone reaching a minimum at the top core (Fig. 6) and reflecting the evolution of anoxic conditions in this zone due to the large amount of deposited OM.

*4.3.2. The Black Lake*

The BRYT02B record can be divided in 2 main zones (Fig. 4) based on cluster and broken stick analyses (Fig. S14 and S15). The top core section (0 – 2 cm) was removed from these analyses as it represented a single sample composed of high amounts of fresh OM and high SC *n*-Alk concentrations. The two identified zones are distinguished by variations in PC1 scores (Fig. S7) with depth (Fig. S16).

• <u>Zone 1</u> (*ca.* 1915 to *ca.* 1980, 67.5 – 23 cm)

Zone 1 is primarily influenced by submerged and floating macrophytes (high/moderate percentages of *n*-$C_{21}$ and, *n*-$C_{23}$, respectively), non-emergent vascular plants (high/moderate percentages of *n*-$C_{25}$) and higher plants (high/moderate percentages of *n*-$C_{29}$, respectively) (Fig. S17).

Vascular plants (*n*-$C_{29}$) and submerged and floating macrophytes (*n*-$C_{23}$ and *n*-$C_{25}$) were the major components of sedimentary OM in mid- (*ca*. 1945 to *ca*. 1955; 50 – 40 cm) and lower (*ca*. 1915 to *ca*. 1945; 67.5 – 50 cm) sections of this zone (Fig. S7 and S17), as confirmed by the lower ACL, increasing $P_{aq\ wax}$ index and decreasing TAR values in this part of the core (Fig. 5). The upper sections of this zone (*ca*. 1955 to *ca*. 1980; 40 – 23 cm) may represent the transition from an environment typically influenced by allochthonous sources (higher plants), with grasses/herbs appearing as an important fraction of terrestrial OM input, to an increase in sedimentary allochthonous OM from phytoplankton (*n*-$C_{15}$, *n*-$C_{17}$ and *n*-$C_{19}$) (Fig. S17), as verified by inverse trends of $P_{aq\ wax}$ index and TAR when compared with lower sections of this zone (Fig. 5).

Zone 1 showed a general decrease and slight fluctuation in the $LOI_{550}$ values and Total *n*-Alk concentrations (Fig. 3), and contained mainly MC and LC *n*-Alk until *ca.* 1955, while SC *n*-Alk concentration increased until *ca.* 1960. Between *ca.* 1955 and *ca.* 1980 the concentrations of all classes of *n*-alkanes and $LOI_{550}$ values oscillated between their recorded maximum and minimum values (Fig. 3). Magnetic susceptibility values were low and decreased through Zone 1, with low fluctuations throughout this section of the core (Adams, 2017), suggesting decreased input of terrestrial materials.

The values of O/E and CPI in this zone indicate preserved OM and the absence of anthropogenic hydrocarbons, with a marked influence from terrestrial higher plants in the mid- and bottom sections. A decrease in the O/E (< $C_{24}$) and $CPI_1$ ratios (both related to SC and MC *n*-Alk) was observed in the upper sections (Fig. 5), which may indicate the presence of oil hydrocarbons sources. Indeed, the input of hydrocarbons related to crude oil and by-products has been previously characterized in Black Lake (Adams et al., 2018), by the increased concentrations of low molecular weight polycyclic aromatic hydrocarbons (PAHs with 2-3 rings) after *ca.* 1970 and low values (< 0.10) of the anthracene / anthracene + phenanthrene (Ant/178) ratio after *ca.* 1960, that are typically associated with petroleum sources and its by-products (Yunker et al., 2002).

The individual *n*-alkanes distribution and related ratios corroborate the pigment and diatom records from the early-20$^{th}$ century, indicating low productivity, characterized primarily by low algal concentrations and biomass (i.e., low relative contribution of SC *n*-Alk) (Adams, 2017). Macrophyte communities in Black Lake in the early-1900 (Eftesum, 2017), showed the predominance of *Chara* spp, a common species in lower productivity, clear-water shallow lakes, with stable and diverse macrophyte communities (Bazarova and Itigilova, 2006; Sayer et al., 2010). This autochthonous source of sedimentary OM is recorded by relatively high MC *n*-Alk (i.e., *n*-$C_{23}$ and *n*-$C_{25}$) levels in this zone. Changes found in the biomarker analyses in the mid-20$^{th}$ century (*ca.* 1960) also verified a shift in both pigment concentration and diatom assemblage records at Black Lake (Adams, 2017).

The Pri/Phy ratio presented decreasing values ratios in this zone reaching the minimum around *ca.* 1970, remains constant until the top core (Fig. 6), reflecting changes in the oxic (> 2) to redox (< 1) environmental conditions. On the other hand, the Pri/*n*-$C_{17}$ and Phy/*n*-$C_{18}$ presented low and relatively constant values in mid- and lower sections (until *ca.* 1955) (Fig. 6), related to high water column productivity and preserved OM in the sediments. A slight increase and relative steady values until top core on Pri/*n*-$C_{17}$ and Phy/*n*-$C_{18}$ suggest different relative biodegradation rates of *n*-alkanes and the corresponding isoprenoids under anoxic conditions (as indicated by Pri/Phy ratio) (Commendatore and Esteves, 2004). Total algal pigments (chlorophyll-*a* and pheophytin-*a*) are precursors of isoprenoids and high levels were presented in the Black Lake pigment record from *ca.* 1965 until the top core (Adams, 2017), explaining a marked contribution of pristane and phytane in upper sections until recent years.

- Zone 2 (*ca.* 1980 to *ca.* 2010; 23 – 2 cm)

Zone 2 represented a stable period for the lake with an important contribution of fresh aquatic OM from phytoplankton (high/moderate relative percentages of *n*-$C_{15}$ and *n*-$C_{19}$ and, *n*-$C_{17}$) and terrestrial components associated with grasses/herbs (high/moderate relative percentages of *n*-$C_{31}$, *n*-$C_{33}$ and *n*-$C_{35}$) (Fig. S17), especially between *ca.* 1985 and *ca.* 1995.

Zone 2 showed lower concentrations of MC and LC *n*-Alk compared to the previous zone, while SC *n*-Alk concentrations remained constant to the top of the core (Fig. 3). A slight decrease in Total *n*-Alk concentrations was observed while $LOI_{550}$ values were relatively stable (Fig. 3). The individual *n*-alkanes distribution, based on the PCA, confirm the establishment of a new sedimentary environment with a significant autochthonous OM contribution as indicated by *n*-alkanes from phytoplankton (*n*-$C_{15}$, *n*-$C_{17}$, and *n*-$C_{19}$) and more input from grasses/herbs (*n*-$C_{31}$ and *n*-$C_{33}$) together with vascular plants (*n*-$C_{29}$) from allochthonous sources. Lower TAR and steady $P_{aq\ wax}$ values suggest 'fresher' OM from autochthonous sources from *ca*. 1980 (Fig. 5).

The values of O/E and CPI ratio oscillated in Zone 2, and are typical of fresh OM with a marked influence from terrestrial sources and higher plants (as suggested by O/E (> $C_{23}$) and $CPI_2$ ratios that are related to more refractory compounds). However, decreasing O/E (< $C_{24}$) and $CPI_1$ ratios from *ca*.1980 to *ca*. 2004 may be associated with oil hydrocarbon sources, towards the end of Zone 1. Relatively anoxic conditions (as indicated by Pri/Phy < 0.5) may prevent extensive degradation of fresh OM; so, the lower values of these ratios may not be associated to the degradation of *n*-alkanes.

The increases in abundances of several planktonic diatom species from *ca.* 1960, and more intensively *ca.* 1980 (represented by SC *n*-Alk in the PCA, Fig. S13) may be attributed to increases in nutrient-associated levels of turbidity, declines in light penetration, and declines in submerged macrophyte abundances (represented by low significance of *n*-$C_{23}$ and *n*-$C_{25}$) in Zone 2 (Fig. S17) (Adams, 2017). Increases in the proportion of planktonic species, also indicated by increasing pigment and isoprenoids concentrations, suggest increasing eutrophication and a shift towards pelagic-dominated primary production (Battarbee et al., 2001; Sayer et al., 2010).

*4.4. Depositional history at the Selenga River basin*

SLNG04 and Black Lake have undergone several major shifts in state, structure, and likely functioning since the early-19$^{th}$ century reflecting environmental changes in the Selenga River basin. Biomarkers concur with palaeolimnological records and revealed that changes in sedimentary OM at the mid/end-19$^{th}$ century in the Selenga Delta coincide with the Tsagan earthquake just off-shore of the Delta in 1862. The earthquake was the strongest ever recorded in east Siberia (with a magnitude of 7.5) and was integral in forming the contemporary structure of the right bank of the Selenga Delta (Vologina et al., 2010). This seismic event resulted in substantial subsidence and flooding of a large area of the northeast corner of the Delta, with a total area of approximately 200 $km^2$ (Orlov, 1872). Furthermore, a catastrophic flood event in the late-

19th century in Transbaikalia, flooding many towns and villages along the Selenga River, caused an increase in the river height by 4 metres within one day (Kadetova and Radziminovich 2014), was likely responsible for increased input of allochthonous OM, trace and major elements in the Selenga Delta at this time (Yang et al., 2016). The apparent predominance of allochthonous sources from vascular plants, grasses and herbs buried in the SLNG04 lake seems be related to the Tsagan earthquake and the transport of these materials from the drainage basin to the Delta due extreme flooding events. Also, the Tsagan earthquake of 1862 and the flood event in the late-19th century contributed to high sediment exposure to light and air increasing the degradation of labile components of sedimentary OM and increasing the oxic potential of the sediments at this time, due to high levels of sediment influx/erosion to the site (Gell et al., 2007) during this period (Adams, 2017).

Land-use conversion for agricultural expansion and intensification began in the Selenga River basin in the early-19th century as a response to increased populations in the region due the development of the Trans-Siberian Railroad and mining operations in different portions of this area (Robinson and Anisova, 2004). Agricultural expansion was the primary cause of massive deforestation events and land conversion from boreal forest-steppe (higher plants, with predominance of $n$-$C_{29}$) to croplands in the 19th century in the Selenga Delta and Lake Gusinoye regions. These land conversions resulted in an increase in the erosion and transport of sediment and allochthonous OM directly into the Selenga River affecting large areas of the basin (Eimers et al., 2008; Bazhenova and Kobylkin, 2013). Also, increased agriculture within the Selenga River basin during this period resulted in increasing eutrophic conditions that was reflected in changes in the main components of sedimentary OM, from non-emergent vascular plants and grasses/herbs to submerged and floating macrophytes and phytoplankton in Selenga Delta (Fig. S9).

Further agricultural expansion in the Selenga River basin occurred intensively from the mid-1950s, with a peak around the 1980s (Robinson and Anisova, 2004), and contributed to an increase to hypereutrophic conditions in the Selenga Delta, and increasing the contribution of autochthonous OM to the sediments (Fig. 3). Increased connectivity to the Selenga River, increased agricultural activity and anthropogenic disturbances within the Selenga River basin in the early-20th century likely resulted in increased nutrient flux to the Selenga Delta. A possible mechanism relates to stimulated plant production (submerged and floating macrophytes), leading to increased photosynthetic removal of $CO_2$ from the water column, resulting in increasing pH (Potasznik and Szymczyk, 2015). A higher pH would lead to increased phosphorus release from sediments (Koski-Vahala et al., 2001; Wu et al., 2014), thereby stimulating plant productivity during the early- to mid-20th century, as well as slight reducing conditions in the Selenga Delta.

Population growth after the end of World War II, as well as intensive increases in livestock populations until *ca.* 1990, contributed to the increase in nutrient flux to soils, and increased sediment erosion within the Selenga River basin (Bazhenova and Kobylkin, 2013). The enrichment from increased populations and intensification of agricultural practices drove the observed changes found in the sedimentary OM in the mid-20th century (*ca.* 1960) at Black Lake. Differing controls for OM and mineral supply has been assumed in Lake Gusinoye region post-1950 (Adams, 2017), indicating a higher influx of mineral sources and coinciding with increased development and industrialization near Lake Gusinoye in the 1950s (Pisarsky et al., 2005).

The construction of the Irkutsk Hydroelectric Dam along the Angara River (the only outflow from Lake Baikal) was completed in the mid-1950s. The filling of the associated reservoir was completed in 1963 and the environments surrounding of the Lake Baikal, and were submerged (Pinegin et al., 1976), increasing the water depth, surficial area and the connectivity of Selenga Delta with Selenga River, promoting changes in submerged macrophyte and diatom communities (Brock et al., 2011). These features may have contributed to increased preservation of sedimentary OM and substantial changes in sedimentary redox conditions since *ca.* 1940 and, as well as to the development of diatom assemblages in the early-1960s (Adams, 2017). Indeed, the sharp, though temporary, increase in sedimentation rate in the early-1960s (1963 ± 4 yrs) may correspond with the timing of the flood associated with the construction of the Irkutsk Dam and may contribute to the evolution of anoxic conditions and preservation of a large amount of deposited OM.

The decades after the end of World War II were characterized by intense economic growth and industrial expansion in the USSR, and industrial and mining growth in the Lake Gusinoye region (Pisarsky et al., 2005). This may have increased petroleum product demand (e.g., diesel fuel, motor oil), increasing usage and consequently, indirect input to the environment and the occurrence of oil hydrocarbons in aquatic systems as indicated by *n*-alkanes and polycyclic aromatic hydrocarbons in sediments of the region (Adams et al., 2018).

## 5. Conclusions

The drivers of sedimentary OM composition and sources since the 19th century in the Selenga River basin have been both natural and anthropogenically-induced. Local natural disturbances, regional natural variability in climate, and anthropogenic stressors have resulted in ecological responses within the Selenga River basin during the last two centuries, including the composition of lacustrine sedimentary OM.

The greatest period of agricultural expansion (resulting in wide-scale deforestation) and population growth in the Selenga River basin occurred in the mid-20th century, and was closely linked with periods of economic development in Russia. Changes in hydrological regimes (flood events), and nutrient pollution, changed the composition of sedimentary OM in the region.

The SLNG04B record extends back to the early-19th century, while the record from Black Lake extends only to the late-19th century. Within the period covered by these records, the primary finding is the biomarker response to the elevated nutrient levels related to increasing regional anthropogenic development in southeast Siberia, with agricultural intensification in the early-20th century, and economic and industrial intensification following World War II in the early to mid-20th century.

The individual *n*-alkanes and their related proxies provide valuable information on the origin, degradation level and deposition conditions of the deposited OM over the last two centuries in southern Siberia and can be used to corroborate ecological changes in freshwater floodplain wetlands.

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Table 1. *n*-Alkane ratios used in the literature to distinguish the different sources of organic matter (OM).

| Name | | *n*-Alkane equations | Diagnostic values | Source assignment | References |
|---|---|---|---|---|---|
| Odd/Even ratios | O/E (< $C_{24}$) | $\frac{\sum \text{odd } C_{11}–C_{23}}{\sum \text{even } C_{10}–C_{22}}$ | ~ 1.0 | petroleum and their by-products | Tolosa et al. (2004) |
| | | | >> 1.0 | biogenic origin | |
| | O/E (> $C_{23}$) | $\frac{\sum \text{odd } C_{25}–C_{37}}{\sum \text{even } C_{24}–C_{36}}$ | ≤ 1.0 | progressive OM degradation | Bray and Evans (1961)<br>Marzi et al. (1993) |
| | | | >> 1.0 | fresh OM | |
| Carbon Preference Index | CPI 1 | $0.5^* \frac{(n\text{-}C_{15}+n\text{-}C_{17}+n\text{-}C_{19}+n\text{-}C_{21}+n\text{-}C_{23})}{(n\text{-}C_{14}+n\text{-}C_{16}+n\text{-}C_{18}+n\text{-}C_{20}+n\text{-}C_{22})} + \frac{(n\text{-}C_{15}+n\text{-}C_{17}+n\text{-}C_{19}+n\text{-}C_{21}+n\text{-}C_{23})}{(n\text{-}C_{16}+n\text{-}C_{18}+n\text{-}C_{20}+n\text{-}C_{22}+n\text{-}C_{24})}$ | ≤ 1.0 | anthropogenic and/or reworked material | Bray and Evans (1961)<br>Aboul-Kassim and Simoneit (1996) |
| | | | 1.0 – 3.0 | aquatic OM contribution | |
| | CPI 2 | $0.5^* \frac{(n\text{-}C_{25}+n\text{-}C_{27}+n\text{-}C_{29}+n\text{-}C_{31}+n\text{-}C_{33})}{(n\text{-}C_{24}+n\text{-}C_{26}+n\text{-}C_{28}+n\text{-}C_{30}+n\text{-}C_{32})} + \frac{(n\text{-}C_{25}+n\text{-}C_{27}+n\text{-}C_{29}+n\text{-}C_{31}+n\text{-}C_{33})}{(n\text{-}C_{26}+n\text{-}C_{28}+n\text{-}C_{30}+n\text{-}C_{32}+n\text{-}C_{34})}$ | > 4.0 | terrestrial OM contribution | |
| Terrestrial-to-Aquatic Ratios | TAR | $\frac{n\text{-}C_{25}+n\text{-}C_{27}+n\text{-}C_{29}+n\text{-}C_{31}+n\text{-}C_{33}}{n\text{-}C_{15}+n\text{-}C_{17}+n\text{-}C_{19}+ n\text{-}C_{21}+n\text{-}C_{23}}$ | < 1.0 | autochthonous contribution | Bourbonniere and Meyers (1996)<br>Silliman et al. (1996)<br>Chevalier et al. (2015) |
| | | | > 4.0 | terrigenous source predominance | |
| Aquatic Proxies | $P_{aq\ wax}$ | $\frac{n\text{-}C_{23}+n\text{-}C_{25}}{n\text{-}C_{23}+n\text{-}C_{25}+n\text{-}C_{27}+n\text{-}C_{29}}$ | < 0.1 | allochthonous OM | Ficken et al. (2000)<br>Li et al. (2020) |
| | | | 0.1 - 0.4 | mixed sources | |
| | | | > 0.4 | autochthonous OM, typically submerged and floating macrophytes | |
| Average Chain Length ratios | ACL | $\frac{27 * (n\text{-}C_{27}) + 29 * (n\text{-}C_{29}) + 31 * (n\text{-}C_{31}) + 33 * (n\text{-}C_{33}) + 35 * (n\text{-}C_{35})}{n\text{-}C_{27}+n\text{-}C_{29}+n\text{-}C_{31}+n\text{-}C_{33}+n\text{-}C_{35}}$ | 27 – 28 | predominance of vascular plant | Poynter and Eglinton (1990)<br>Freeman and Pancost (2014)<br>Sachse et al. (2006)<br>Vogts et al. (2012) |
| | | | 28 – 29 | contribution of trees/shrubs | |
| | | | < 29 | contribution of grasses/herbs | |
| Isoprenoids and short *n*-alkanes ratios | Pri/Phy | $\frac{\text{Pristane}}{\text{Phytane}}$ | < 1 | anoxic conditions | Tissot and Welte (1978)<br>Didyk et al., 1978<br>Peters et al. (2005) |
| | | | > 3 | oxic conditions | |
| | Pri/*n*-$C_{17}$ | $\frac{\text{Pristane}}{n\text{-}C_{17}}$ | < 1 | increase on biodegradation of *n*-alkanes | Peters and Moldowan (1993)<br>Commendatore and Esteves (2004) |
| | Phy/*n*-$C_{18}$ | $\frac{\text{Phytane}}{n\text{-}C_{18}}$ | > 3 | increase in the delivery of phototrophic OM | |

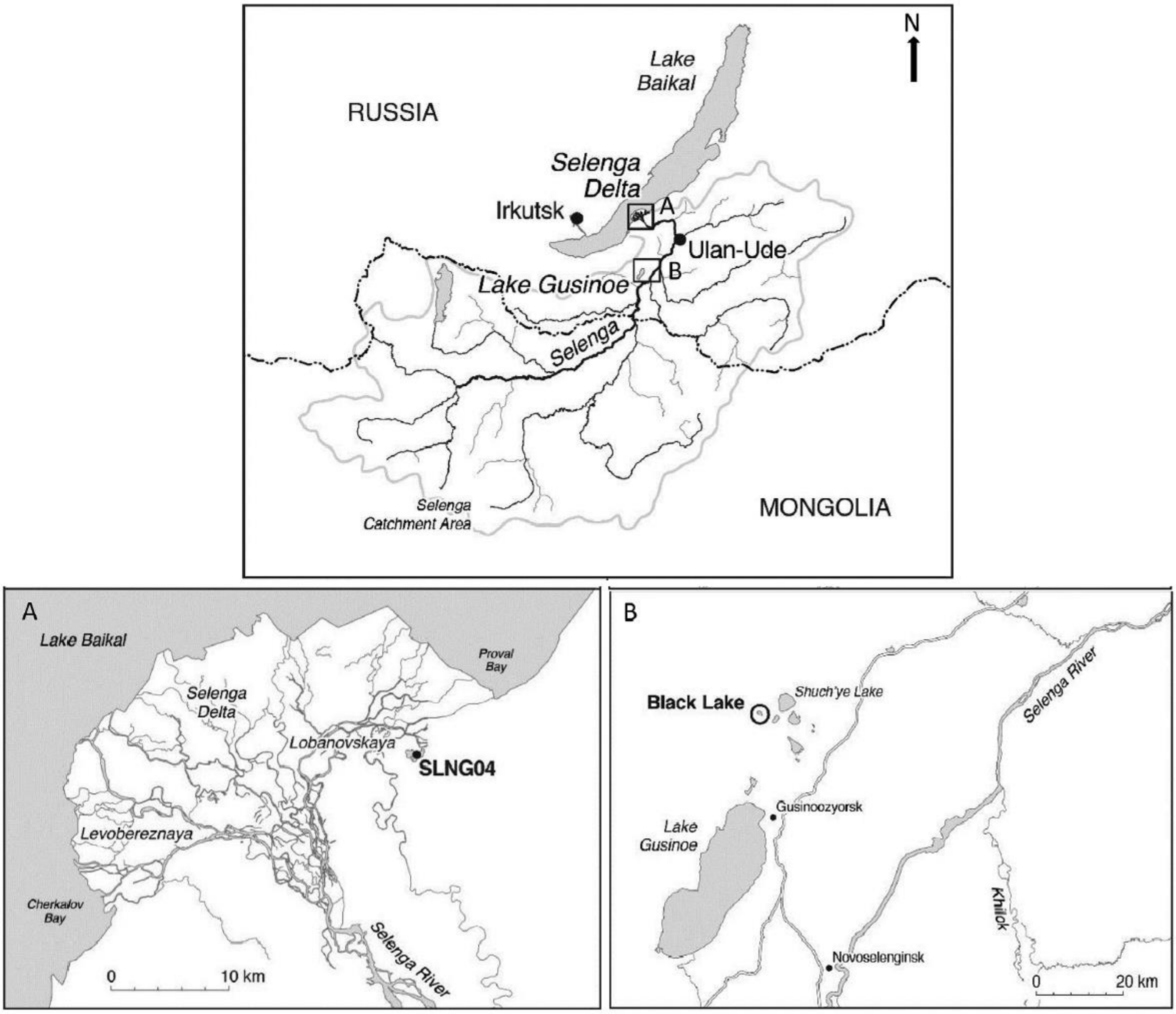



Fig. 1. Map of the Selenga River basin in southeast Siberia and northern Mongolia, with major river tributaries and cities labelled. Subset A. Map of the Selenga Delta with SLNG04 location indicated. Subset B. Location of Black Lake (BRYT02 site) in the Gusinoozersk region (adapted from Adams et al. 2018).


Fig. 2. Shallow lakes studied in Selenga River basin. Subset A: SLNG04 lake, view from open water, facing southeast; Subset B: SLNG04 view from shore, facing northwest; Subset C: BRYT02 view from the eastern shore, facing northwest; Subset D: Black Lake (BRYT02 site) view from the southeastern shore, facing west.

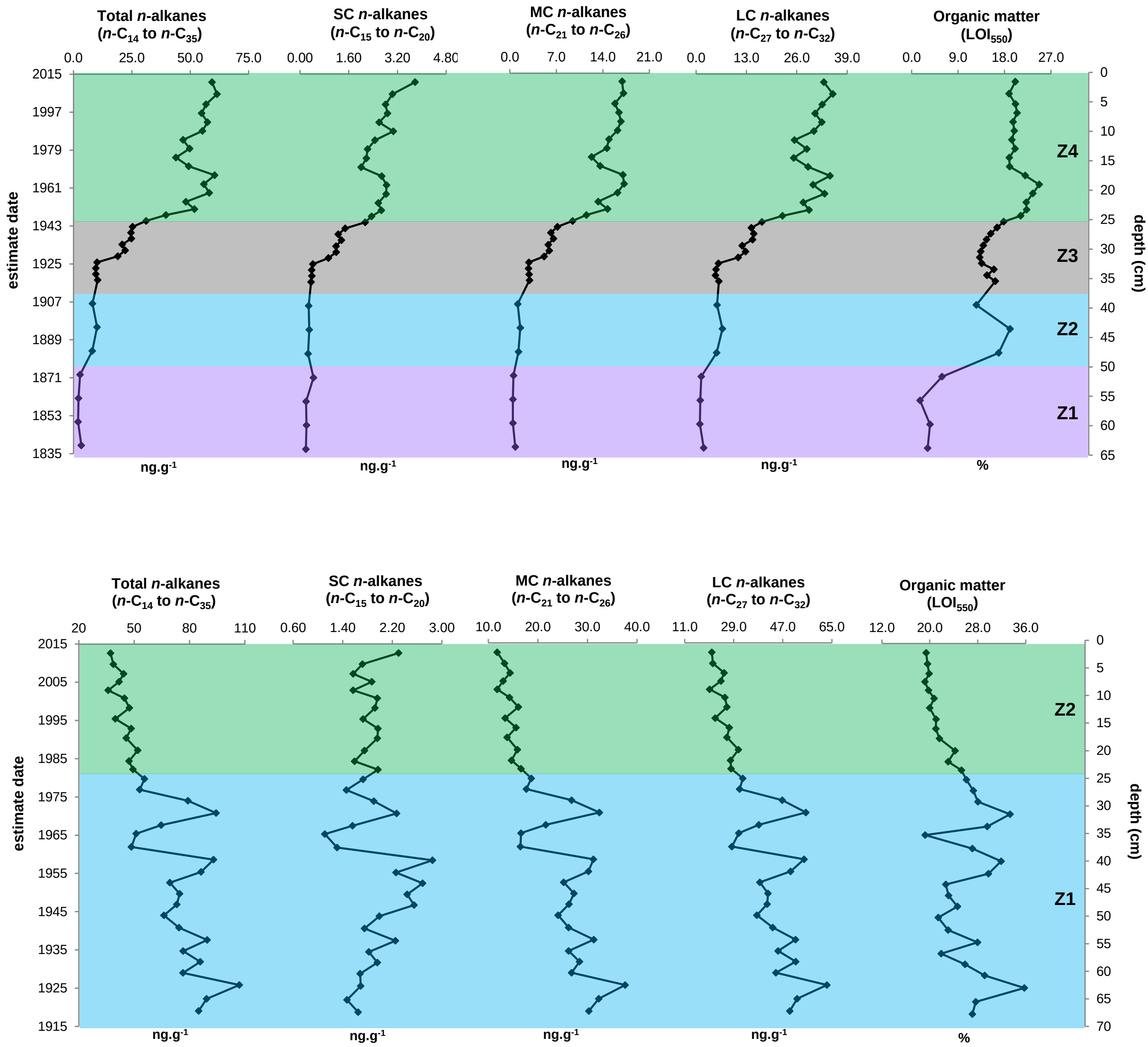



Fig. 3. Profiles of total, short-chain (SC), mid-chain (MC) and long-chain *n*-alkanes (LC) (in μg $g^{-1}$) and organic matter content ($LOI_{550}$, in %) for the sediments from SLNG04B and BRYT02B cores.

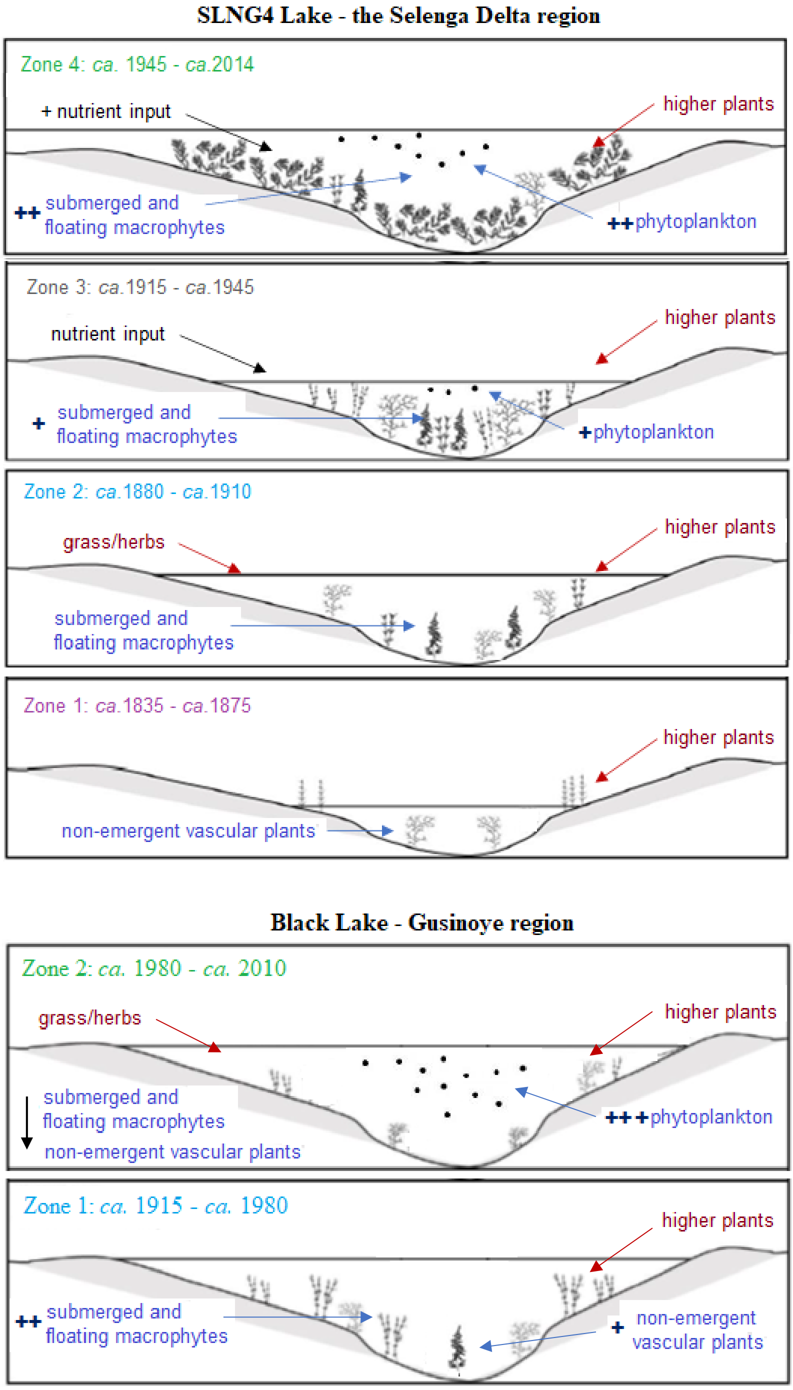



Fig. 4. Conceptual diagram of ecological community change at SLNG04 and Black Lakes since the late-19th and early-20th, respectively.

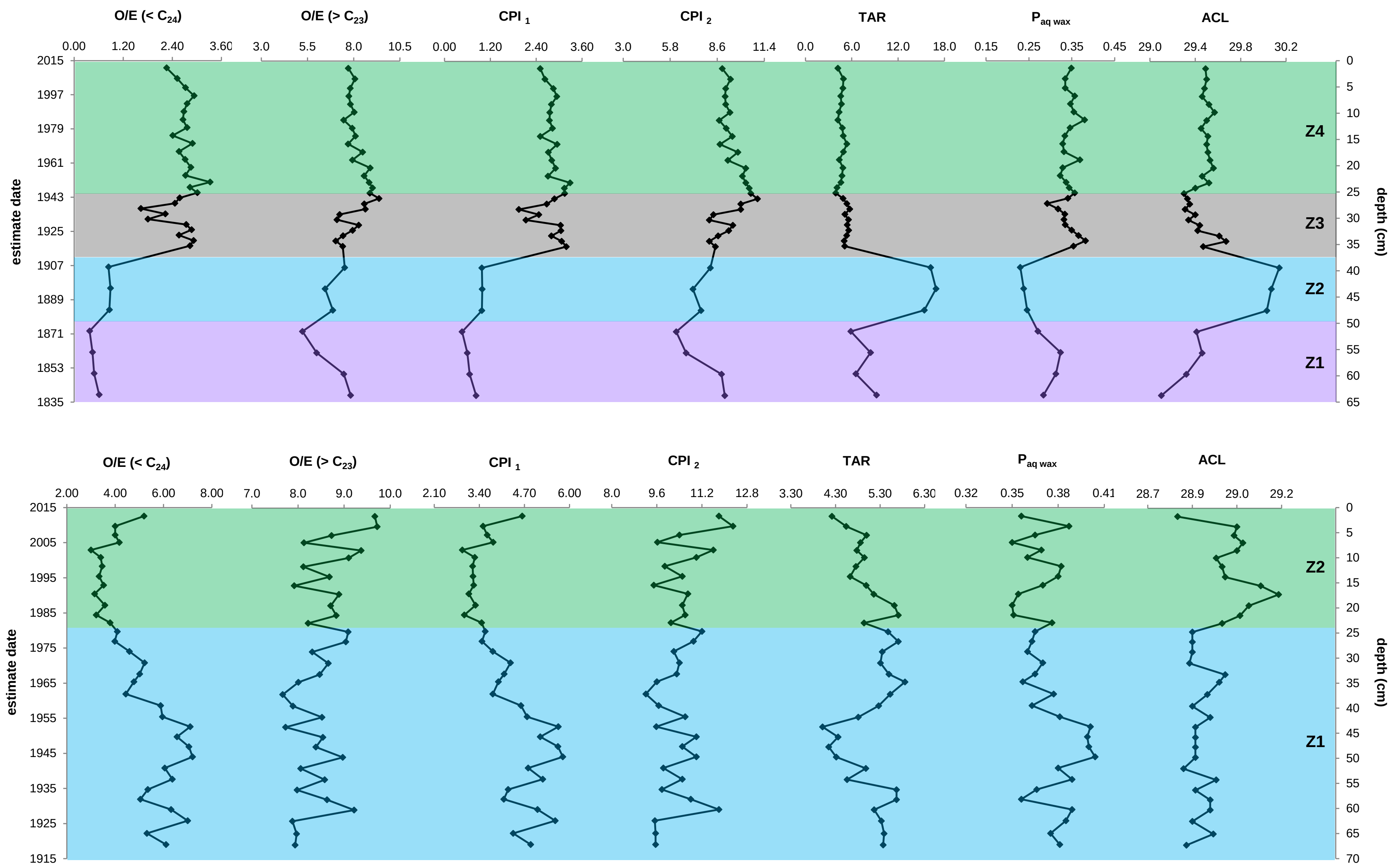
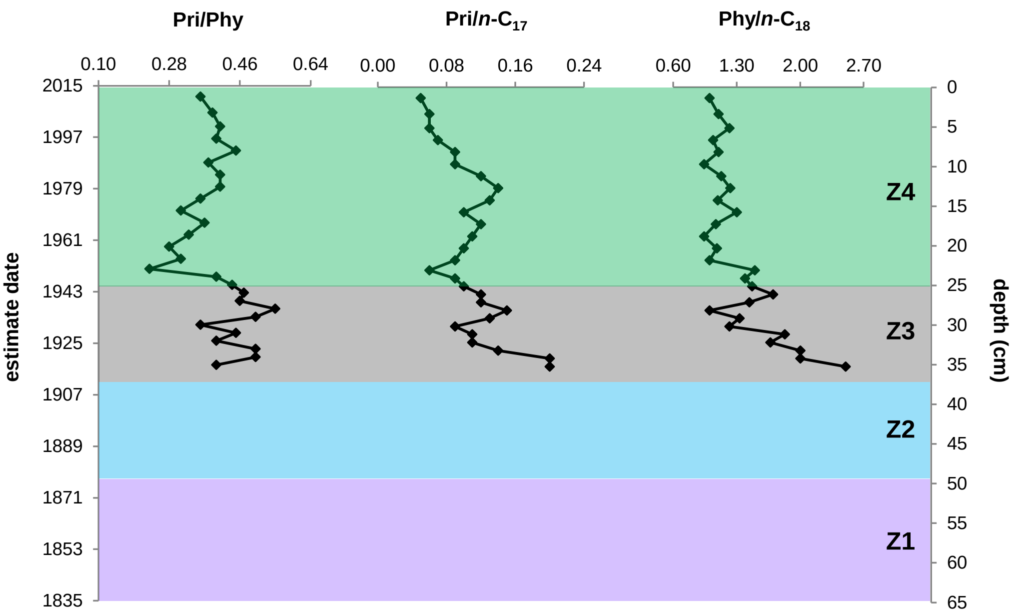



Fig. 5. Profiles of selected *n*-alkane indices for the sediments from SLNG04B and BRYT02B cores: odd/even (O/E < $C_{24}$ and O/E > $C_{23}$), carbon preference index considering short-chain *n*-alkanes (CPI $_1$), and long-chain *n*-alkanes (CPI $_2$), terrestrial over aquatic (TAR), the $P_{aq\ wax}$, and average chain length (ACL) ratios.



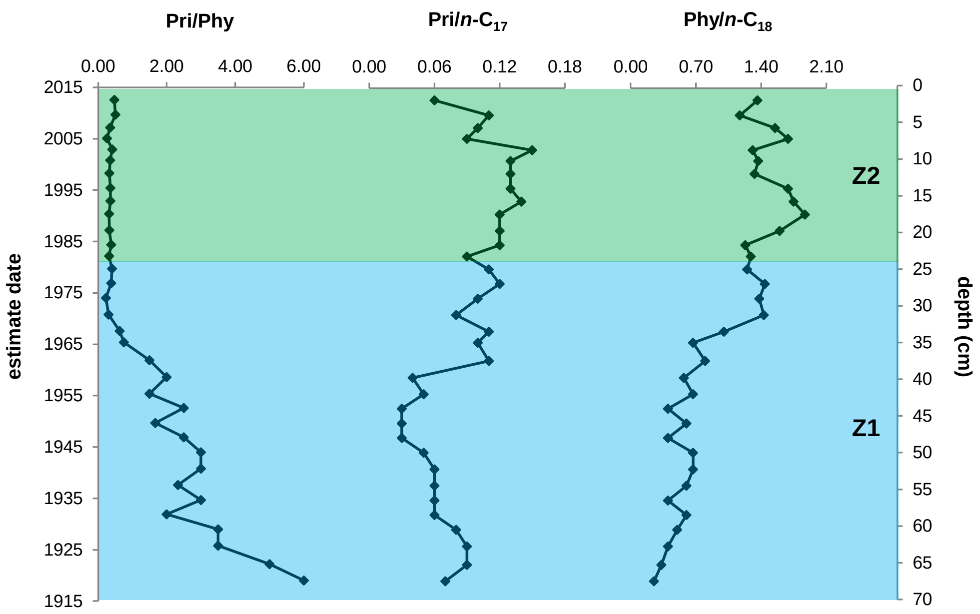




Fig. 6. Profiles of isoprenoid indices for the sediments from SLNG04B and BRYT02B cores: pristane/phytane (Pri/Phy), pristane/$n$-$C_{17}$ (Pri/ $n$-$C_{17}$), and phytane/$n$-$C_{18}$ (Phy/ $n$-$C_{18}$) ratios.

**Declaration of interests**

_Title_: *Earthquake, floods and changing land use history: a 200-year overview of environmental changes in southern Siberia as indicated by n-alkanes and related proxies in sediments from shallow lakes*

_Authors_: *César C. Martins, Jennifer K. Adams, Handong Yang, Alexander A. Shchetnikov, Maikon Di Domenico, Neil L. Rose, Anson W. Mackay*

☒ The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

☐The authors declare the following financial interests/personal relationships which may be considered as potential competing interests:

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**CRediT author statement**

**César C. Martins**: Conceptualization; Data curation; Formal analysis; Visualization; Writing - original draft; Writing - review & editing. **Jennifer Adams**: Conceptualization; Data curation; Writing - review & editing. **Handong Yang**: Formal analysis; Writing – review & editing. **Alexander A. Shchetnikov**: Conceptualization; Project administration; Resources. **Maikon Di Domenico**: Formal analysis; Writing – review & editing. **Neil Rose**: Conceptualization; Supervision; Writing – review & editing. **Anson W. Mackay**: Conceptualization; Supervision; Project administration; Resources; Writing – review & editing.