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RESEARCH ARTICLE

A seasonal cycle of terrestrial inputs in Lake Van, Turkey

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Abstract Lake Van in Turkey is the world's largest soda lake (607 km^3). The lake's catchment area is estimated to be ~12,500 km^2, and the terrestrial input is carried through eolian, riverine, snowmelt and anthropogenic paths. Extent and seasonality of the terrestrial inputs to the lake have not been studied, but it is essential to evaluate its environmental status and to assess the use of environmental proxies to estimate the lake's response to climate changes. This study aims to measure seasonal changes in terrestrial input of natural and anthropogenic origin as recorded by the fluxes of pollen and biomarkers of soil bacteria and vascular or higher plants, as well as petrogenic biomarkers in monthly resolved sediment traps from August 2006 to July 2007. Fluxes of pollen, soil and higher plant biomarkers seem to be related to precipitation and snowmelt in autumn and spring. In addition, dust storms, which are common during the summer months, may have resulted in long-distance transport. Anthropogenic biomarker fluxes indicate year-round petrogenic contamination although some mature biomarker fluxes are higher in summer and in late winter–spring. The relative changes between petrogenic markers indicate variations in the pollutant sources.

Keywords Seasonal particle cycle · Contamination · Biomarkers · *n*-Alkanes · Branched GDGTs · Hopanes · Steranes

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Introduction

Over the last decades, lake sediments have been shown to be excellent archives of past environmental changes. A record spanning various glacial–interglacial cycles was recovered from Lake Van's subsurface within the frame of the International Continental Scientific Drilling Program project PALEOVAN (Litt et al. 2009, 2011). Evaluating the stored environmental information in the sediments depends on the knowledge about the ecosystem and the understanding of the present day lake response to environmental variability. Knowing the processes driving the terrestrial input is essential to understand for instance carbon deposition and nutrient input, which are linked to climate through weathering and transport processes. On the other hand, understanding the anthropogenic impact is key to assess the environmental status of a lake today and in the recent history.

Lake Van in Eastern Anatolia (Turkey) is the world's largest soda lake, located at 1,648 m above sea level, and has an area of 3,570 km^2, a maximum depth of 460 m, a pH of ~9.5–9.9, ~21–24‰ salinity and 155 $meql^{-1}$ alkalinity (Kaden et al. 2010; Litt et al. 2009; Reimer et al. 2009). Lake Van receives water mainly through precipitation and snowmelt inflow from a catchment area estimated to be about 12,500 km^2. The lake loses water only through evaporation

due to its endorheic nature (Kadioglu et al. 1997; Fig. 1). Our aim was to assess the seasonality of terrestrial input proxies and to establish possible links between them and climatic processes. A mooring with a sequential sediment trap was deployed between August 2006 and June 2007. The sediment trap samples allow us to better understand how terrestrial organic matter (OM), derived from the natural environment as well as from human activity, is deposited in the lake's sedimentary archive.

Natural terrestrial input was assessed using pollen, branched glycerol dialkyl glycerol tetraethers (brGDGTs), branched versus isoprenoid tetraether (BIT) index, and fresh *n*-alkanes, thus covering all terrestrial input paths. Mature *n*-alkanes and petrogenic biomarkers such as hopanes and steranes were used to track the anthropogenic input.

Palynological studies provide information on vegetation changes reflecting past climate variations. Previous palynological studies of Lake Van successfully revealed climatic changes in the area for the last ~20 kyrs (Litt et al. 2009; Wick et al. 2003).

BrGDGTs are usually abundant in peat bogs and soils (Sinninghe-Damsté et al. 2002; Weijers et al. 2006) though in situ production has been suggested for both lake and marine sediments (e.g. Blaga et al. 2010; Peterse et al. 2009). By analogy with the structures of bacterial lipids, the source organisms for brGDGTs are postulated to be soil bacteria, but so far, they remain orphan biomarkers (Weijers et al. 2006). Nonetheless, they are assumed to trace mainly soil-terrestrial input (Walsh et al. 2008; Weijers et al. 2009). Moreover, in situ production of brGDGTs in Lake Van sediment would not be captured by trap samples as those only incorporate biomarkers from terrestrial or water column sources. The BIT index is also used to assess the relative input of terrestrial OM versus aquatic OM production (Hopmans et al. 2004). The BIT ranges from 0, referring to pure marine or lacustrine OM, and 1 indicating pure terrestrial OM (Hopmans et al. 2004). This proxy uses the ratio of brGDGTs and crenarchaeol, a GDGT synthesized by pelagic Thaumarchaeota (mesophile Archaea, formerly Crenarchaeota Group I; see Brochier-Armanet et al. 2008; Sinninghe-Damsté et al. 2002 for details) that stands for the aquatic end member. When interpreting BIT records, it is important to notice that the changes in crenarchaeol can strongly influence the BIT index and that the single aquatic or terrestrial origin of the markers is not as well delimited as previously thought (see Fietz et al. 2011 for details).

The *n*-alkanes are aliphatic lipids that typically range from *n*-C_{15} to *n*-C_{35}. Their presence in aquatic settings can be attributed to in situ production as well as transport by wind or surface run-off from both natural and fossil fuel sources (e.g. Meyers 2003). Microorganisms and multicellular algal sources yield predominantly short-chain *n*-alkanes (<C_{20}) (Cranwell and Volkman 1981; Volkman et al. 1992). Long-chain *n*-alkanes (*n*-C_{21-35}) in contrast are from higher plant waxes. Long-chain *n*-alkanes with a predominance of the odd-numbered ones stand for fresh natural sources (Volkman et al. 1992). In contrast, mature and/or petrogenic *n*-alkanes do not show such odd-over-even predominance. Therefore, the material maturity can be numerically assessed using the carbon preference index (CPI) (Cooper and Bray 1963; Cranwell 1973; Volkman et al. 1992) as follows:

$$\mathrm{CPI} = \frac{1}{2}\left(\frac{C_{25}+C_{27}+C_{29}+C_{31}+C_{33}}{C_{24}+C_{26}+C_{28}+C_{30}+C_{32}} + \frac{C_{25}+C_{27}+C_{29}+C_{31}+C_{33}}{C_{26}+C_{28}+C_{30}+C_{32}+C_{34}}\right)$$

Values of CPI will initially be >1 since odd number *n*-alkanes dominate, but as maturity increases, the odd number predominance is reduced and the index approaches 1.

The presence of mature and/or petrogenic terrestrial input can also be monitored using steranes and hopanes (Volkman et al. 1992). Hopanes are derived from cell membranes of prokaryotes (heterotrophic bacteria and also phototrophic cyanobacteria), and they are characterized by numerous maturity-sensitive stereoisomers (e.g. Ourisson and Albrecht 1992). Steranes are derived from eukaryotic cell membrane sterols, mainly from algae and higher plants. Both hopanes and steranes are commonly used as a fingerprint of petrogenic input to the environment (e.g. Summons and Walter 1990).

Material and methods

Study area

The climate in eastern Anatolia is strongly influenced by changes in the position of the westerly jet stream, the extension of the subtropical low-pressure belt and Siberian high-pressure area (Litt et al. 2009; Reimer et al. 2009; Wick et al. 2003). The natural vegetation is steppe dominated by sub-euxinian oak forest; however, there is hardly any natural vegetation left, and instead, there is a predominance of agricultural land (Wick et al. 2003). The Lake Van area experiences relatively warm summers (above 20°C) and cold winters (below 0°C), but due to the high salinity, no

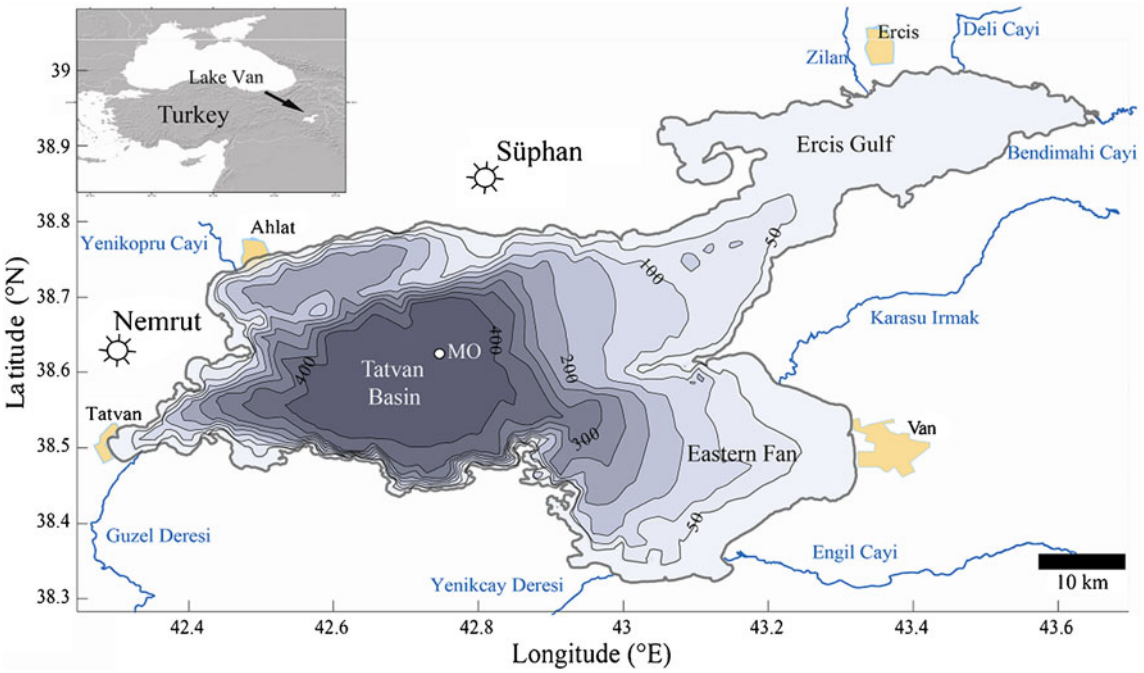

Fig. 1 Bathymetric map of Lake Van with major tributaries, settlements and the mooring site (MO). Two semi-active volcanic systems, Nemrut and Süphan, border the lake

ice forms on the lake surface. Annual precipitation in the city of Van is about 379 mm, with highest precipitation values occurring in April and October. Surface lake water temperatures recorded during the study ranged from 3.4 to 23°C (Stockhecke 2008; Fig. 1). Below 70 m, the water temperature ranges between 3.2 and 3.7°C throughout the year (Stockhecke 2008). Suspended particles are usually deposited as annual varve couplets of light carbonate (spring–summer–autumn) and dark clay-rich laminae (winter) (Stockhecke 2008; Reimer et al. 2009).

Samples

Sediment trap samples were obtained from a mooring deployed in the Tatvan Basin (38.63° N, 42.76° E; Fig. 1) in July 2006 at a water depth of 440 m (10 m above lake bottom). The sequential trap (TECHNICAP-PPS4/3) had 12 cups with individually controlled exposure times (18–35 days) and a funnel-shaped inlet, which ensured that no settled material was resuspended from the bottom of the trap (Stockhecke 2008). All trap samples were recovered in July 2007.

After recovery, the sampling cups were stored in a cool room (4°C) for 48 h to ensure particle settlement. The supernatant water was decanted, and samples were freeze-dried and weighed. The amount of salt present in the samples' standing water was subtracted from the sample mass to subsequently determine the net mass fluxes (see Stockhecke 2008 for details).

Pollen analysis

The preparation of the pollen samples followed the standard procedure (see Faegri and Iversen 1989 for details) followed by ultrasonic sieving (10 μm) to concentrate the palynomorphs. *Lycopodium* spore tablets (Lund University, Sweden) were added to the sediment to calculate the pollen deposition rate. The residue was stained with safranin and mounted on slides in glycerol. The pollen reference collection of Near Eastern plants (Steinmann Institute, Paleontology, Bonn University) as well as descriptions of the circum-Mediterranean pollen flora (Reille 1995, 1998, 1999) were used for pollen taxa identification.

Extraction for lipid biomarker analysis

Freeze-dried sediment trap samples were extracted by sonication for 5 min, three times each, first with methanol (MeOH), then a mixture (1:1, *v*/*v*) of MeOH and dichloromethane (DCM) and lastly with DCM. After sonication, samples were centrifuged at 1,500 rpm (455 G) to remove particles. The supernatants were collected, and after evaporating the solvents, the extracts were put through a small pipette filled with anhydrous sodium sulphate (>99 %, Merck) to remove remaining water and particles. An internal standard for *n*-alkanes (C_{36} hexatriacontane; Sigma-Aldrich) was added to the samples before extraction. The total lipid extract was divided into apolar, intermediate and

polar fractions with a small column filled with activated silica and using hexane/DCM (9:1, *v*/*v*), hexane/DCM (1:1, *v*/*v*) and DCM/MeOH (1:1, *v*/*v*) as eluents, respectively. All solvents used were from Merck, Darmstadt, Germany.

n-Alkane analysis

The apolar fraction containing the *n*-alkanes was dried with N_2 and redissolved in isooctane. Samples were analysed using a Thermo Trace gas chromatograph equipped with a flame ionization detector, in split/splitless injection mode and with helium as carrier gas at 2 mL min^{-1} with constant flux. After injection of 2 μL of sample, compounds were separated through an Agilent HP-1 capillary column (60 m× 0.25 mm internal diameter, 0.25 μm film thickness) with a 5-m precolumn. Oven temperature was held at 80°C during 1 min, increased to 120°C at a rate of 20°C min^{-1}, then to 320°C at 6°C min^{-1} and held at this temperature for 20 min. Quantification of the target compounds was determined using the hexatriacontane (C_{36}) as internal standard.

Sterane and hopane analysis

To determine sterane and hopane relative abundances, the apolar fraction was analysed using an Agilent 7890A gas chromatograph coupled with an Agilent 5975C mass spectrometer (GC-MS). After injection of 1 μL of sample, in split/splitless mode, compounds were separated through an Agilent DB-5MS capillary column (30 m×0.25 mm internal diameter×0.25 μm film thickness) with a 5-m precolumn. The oven temperature was 60°C during 0.50 min, was then increased to 150°C at a rate of 50°C min^{-1}, then to 300°C at 6°C min^{-1}, and was afterwards held at this temperature for 22 min. Helium was used as carrier gas at a constant flow of 1 mL min^{-1}. The mass spectrometer was operated in electron impact mode at 70 eV. Data were acquired in selective ion monitoring mode.

Branched GDGT analysis

To quantify the brGDGTs, an internal standard (GR; Rethore et al. 2007) was added to the polar fraction after extraction (cf. Huguet et al. 2006). Samples were hydrolyzed with hydrochloric acid/MeOH (5 %) for 4 h at 70°C. Then, DCM and ultrapure and deionized water (Milli-Q®, Millipore, USA) were added, and the DCM-lipid fraction was collected. This was repeated four times. Then, the DCM fraction was further rinsed with Milli-Q® water in order to eliminate possible leftover acid. This was repeated six times until the fraction was clear (see Huguet et al. 2010 for details).

These hydrolyzed fractions were evaporated, redissolved in hexane/n-propanol (99:1, *v*/*v*) and filtered through 0.45-μm PVDF filters (Phenomenex, Torrance, USA) prior to the injection into a Dionex P680 high-performance liquid chromatograph system coupled to a Thermo Finnigan TSQ Quantum Discovery Max MS with an atmospheric pressure chemical ionization (APCI) interface set in positive mode. Extracts were eluted using a Prevail cyano column (2.1× 150 mm, 3 μm; Alltech, Deerfield, USA) fitted with a precolumn filter and a guard column. The solvent programme is derived and modified from Schouten et al. (2007) and Escala et al. (2009). The flow rate was set at 0.6 mL min^{-1}, and the eluting programme was as follows: 98.5 % hexane and 1.5 % n-propanol for 4 min, increased gradually to 5 % n-propanol in 11 min. To clean the column, the proportion of n-isopropanol was increased to 10 % over a minute and held for 4 min. Finally, the n-propanol was lowered to 1.5 % in 1 min and held for 9 min in order to condition the column. The parameters of the APCI were set as follows to generate positive ion spectra: corona discharge, 3 μA; vaporizer temperature, 400°C; sheath gas pressure, 49 mTorr; auxiliary gas (N_2) pressure, 5 mTorr, and capillary temperature, 200°C. brGDGTs were detected in selected ion monitoring mode of $[M+H]^+ \pm 0.5\, m/z$ units.

Results and discussion

Terrestrial input was assessed using pollen and molecular markers. It was compared to mass fluxes and physical parameters in order to understand source, extent and seasonality of both natural and anthropogenic input of OM into Lake Van. The highest total mass and organic carbon fluxes were found in spring and summer when the water column was stratified, while the lowest mass fluxes occurred in winter when the water column became mixed and the catchment area was snow covered (Fig. 2).

Natural terrestrial input

Natural terrestrial input is generally coupled to the organic carbon and mass fluxes (Fig. 2). All terrestrial input proxies show high fluxes in August 2006 that then decrease towards winter followed by a spring peak. All natural markers show a further peak during summer 2007 (Fig. 2). Highest pollen concentrations were found during summer while almost no pollen was observed in winter (Fig. 2b). The deposited pollen indicates dominance of herbaceous steppe vegetation (grasses and herbs pollen flux, Fig. 2b) in Lake Van's surroundings, which phytogeographically belongs to the so-called Irano-Turanian zone (Zohary 1973). This steppic vegetation type, located on the northern and eastern part of Lake Van, is mainly characterized by grasses, chenopodes, *Artemisia* and other composite plant species (Litt et al. 2009; Wick et al. 2003). In addition, some oak forest remnants can be observed mainly in the valleys of the Bitlis

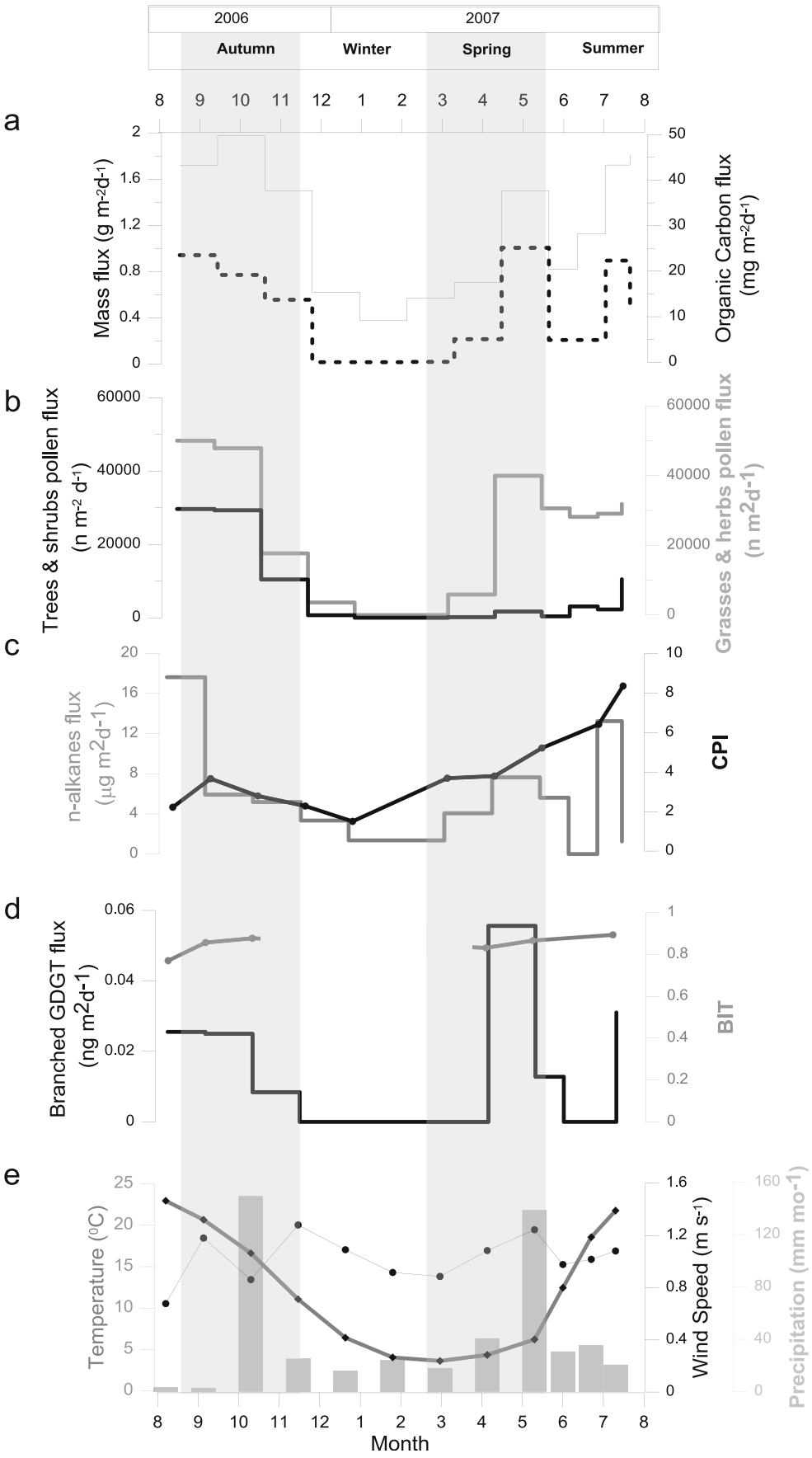

Fig. 2 Natural and anthropogenic terrestrial input compared to physical parameters. **a** Mass flux (*line*) and organic carbon flux (*dotted line*). **b** Pollen fluxes from trees and shrubs (*black line*) and grasses and herbs (*grey line*). **c** Total *n*-alkane flux (*grey line*) and CPI (*black line*). **d** Branched GDGT flux (*black line*) and BIT index (*grey line*). **e** Temperature (*circles*), wind speed (*diamonds*) and precipitation (*grey bars*)

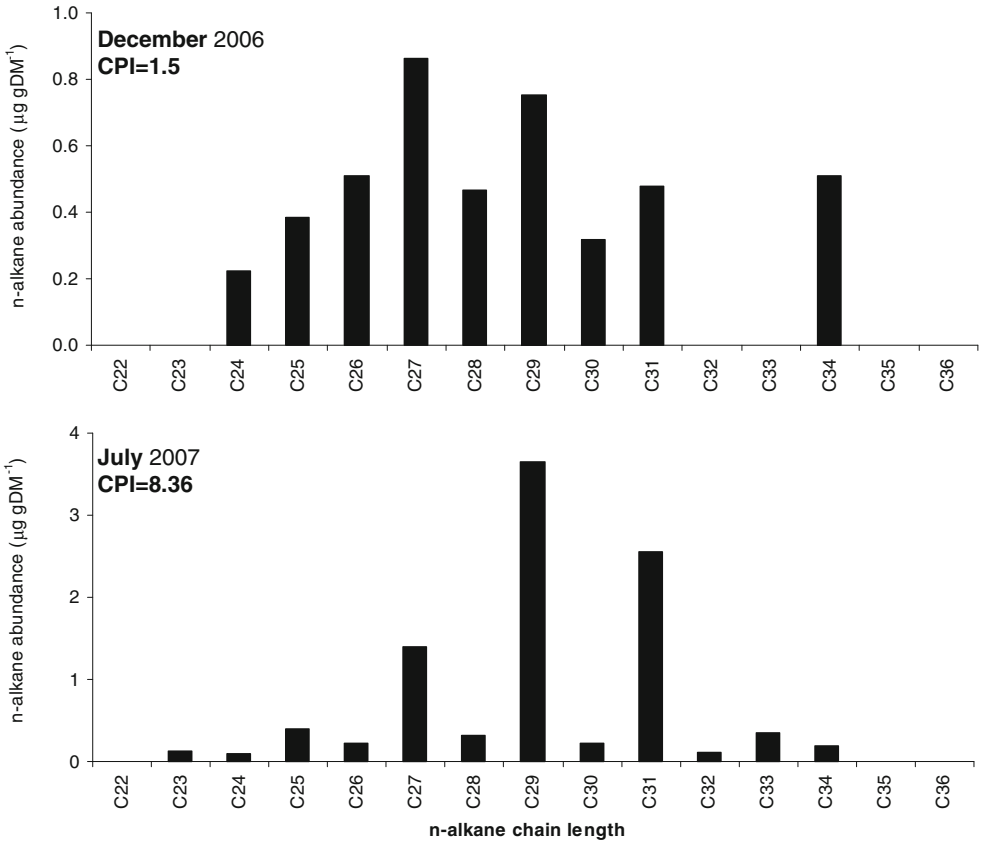

Fig. 3 Distribution of individual *n*-alkanes in trap samples from December 2006 (lowest CPI) and July 2007 (highest CPI)

Massive, southwest of Lake Van, which are also reflected in the pollen spectra (tree and shrub pollen flux, Fig. 2b). Pollen flux trends are in broad agreement with the growth seasons. However, aeropalynological studies in Bitlis have shown increasing pollen concentrations from spring to summer with maxima in May and June (Celenk and Bicakci 2005), whereas the pollen fluxes in our sediment traps reach maxima between July and September. July to September is characterized by the lowest precipitation, the highest temperatures (Fig. 2e) and common maximum wind speeds of around 10 ms^{-1}. Based on data provided by the Turkish State Meteorological Service, dust storms are very common during this period, positively correlated to temperature (Littmann 1991). Thus, maximum pollen fluxes in Lake Van might be related to dust storms (Littmann 1991).

Pollen and lipid biomarkers can both be transported over long distances. In Lake Van, the molecular markers also show higher summer fluxes, which match pollen fluxes (Fig. 2). There is no clear relation between the increased biomarker flux and the precipitation or wind speed changes in summer 2006 (Fig. 2), so that the high summer biomarker flux could also be explained by increased input due to a dust storm. Such a storm would result in increased erosion of leaf waxes and soil particle transport: it could on the one hand

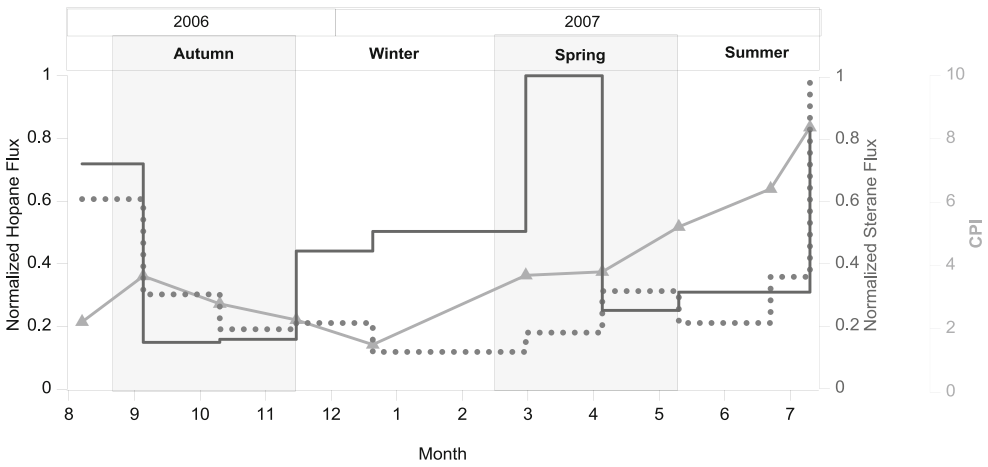

Fig. 4 Changes in petrogenic contribution according to **a** normalized hopane flux (*black line*), **b** normalized sterane flux (*grey dotted line*) and CPI (*light grey triangles*). Normalization of fluxes was accomplished by finding the sample with the highest flux and then normalizing all the fluxes to that value

explain the overall leaf wax-derived *n*-alkane maximum and on the other the soil-derived brGDGT maximum.

While the *n*-alkane flux drops in September, the brGDGT flux remains high till October, possibly due to their different sources and input paths: the observed precipitation increase in autumn (Fig. 2) also increases riverine input of soil-derived brGDGTs, but does not affect the *n*-alkanes which are preferentially wind transported. In early spring, the *n*-alkane input would be coupled not only to increased wind but also to the snowmelt release of those *n*-alkanes deposited in the snow over the winter. brGDGTs seem to only enter the system once snowmelt is coupled with higher precipitation in late spring (Fig. 2). This may be due to their less accessible nature, as brGDGTs have to be leached from the complex soil matrix or enter the system attached to soil particles.

The BIT index ranges between 0.8 and 0.9, which indicates a relatively high terrestrial input of brGDGTs compared to the aquatic-produced crenarchaeol. However, absolute brGDGT abundances were extremely low and below detection limit from late autumn to spring (Fig. 2). Moreover, the BIT index remains almost constant unaffected by either high or low brGDGT fluxes (Fig. 2). This, together with the extremely low crenarchaeol values (see Huguet et al. 2011 for details), indicates that the high calculated BIT index is a reflection of the low crenarchaeol abundance and its changes rather than of changes in the brGDGT terrestrial input, which is in agreement with previous observations by Fietz et al. (2011).

Anthropogenic terrestrial input

Lake Van receives anthropogenic terrestrial input from surrounding cities (Figs. 1 and 2), which enters the lake through sewage discharge and storm water drains (Öğün et al. 2005). The populated eastern shore receives wastewater discharges from a municipal refinery plant (Öğün et al. 2005). Most of the surrounding populations discharge their water untreated or only physically refined (Öğün et al. 2005). 5β-Coprostanol (5β-cholestan-3β-ol) is a 27-carbon stanol formed from the biohydrogenation of cholesterol (cholest-5en-3β-ol) in the gut of most higher animals and birds and has frequently been used as a biomarker for the presence of human faecal matter in the environment (e.g. Bull et al. 2002). Samples were checked for presence of coprostanol, but levels were below detection limit probably due to the distant location of the trap with respect to the wastewater discharge sites.

Chemical evidence of petrogenic contamination in the sediment was reported in the Tatvan Basin, although the source could not be elucidated (Güven et al. 2004). We evaluate seasonal changes in the anthropogenic input with mature molecular markers that are linked to petrogenic sources such as mature *n*-alkanes, hopanes and steranes. Generally, the *n*-alkane input in Lake Van is dominated by C_{27}, C_{29} and C_{31} (e.g. Fig. 3), which indicate a higher plant source. CPI values range from 1.5 in October 2006 to 8.4 in July 2007 (Figs. 2 and 3). Lower CPI values are observed in autumn and winter when natural *n*-alkane flux is diminished (Fig. 2). Thus, in this case, the CPI is not a good indicator of petrogenic contamination as the index is dominated by the strong natural *n*-alkane terrestrial input which masks the presence of mature *n*-alkanes. The presence of mature *n*-alkanes can be observed only when the natural *n*-alkane flux is diminished. But petrogenic contamination is clearly indicated by the hopane and sterane fluxes which are present through the annual cycle.

Petrol and/or wood consumption for house heating during the winter is a likely source for mature *n*-alkanes, hopanes and steranes. This would explain the increase of hopane fluxes in winter and early spring and their sharp decrease in April 2007 (Fig. 4). Hopane and sterane fluxes also increase in summer (Fig. 4). Even though this summer increase in petrogenic contamination biomarkers may be linked to an increase in population and anthropogenic activity during the touristic season, other sources are also possible and no clear link could be established.

Conclusions

This study shows that the intensities of natural and anthropogenic terrestrial input in Lake Van vary seasonally. Natural terrestrial organic matter input is highest in summer and spring. While the summer peak may be linked to dust storms that are common in the area, the spring peak is likely related to increased precipitation and snowmelt for the brGDGTs and stronger wind transport coupled to increased seasonal production for the pollen and higher plant *n*-alkanes. Anthropogenic input on the other hand enters the system through untreated wastewater, wind transport and run-off. Petrogenic contamination is observed through the year and is most prevalent in winter–early spring and late summer. We suggest that the winter peak is linked to house heating; the source of the summer peak remains unclear but may be related to tourism-related increased untreated wastewater input.

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