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Organic matter in sediment layers of an acidic mining lake as assessed by lipid analysis. Part II: Neutral lipids

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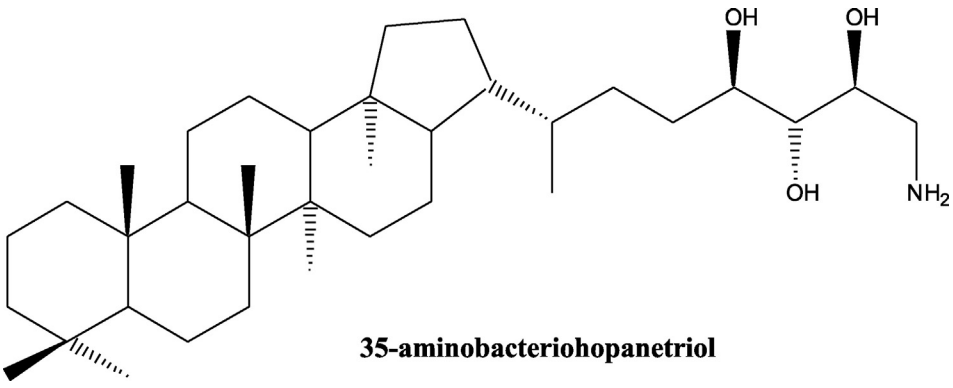
HIGHLIGHTS

- Neutral lipids served as biomarkers to characterize sedimentary organic matter.
- Terrestrial contributions to organic matter dominate over autochthonous sources.
- Pattern of polycyclic aromatic hydrocarbons points to a prevailing pyrolytic origin.
- Petrogenic impact on organic matter proved to be of minor significance.

GRAPHICAL ABSTRACT



35-aminobacteriohopanetriol

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ABSTRACT

Natural neutralization of acidic mining lakes is often limited by organic matter. The knowledge of the sources and degradability of organic matter is crucial for understanding alkalinity generation in these lakes. Sediments collected at different depths (surface sediment layer from 0 to 1 cm and deep sediment layer from 4 to 5 cm) from an acidic mining lake were studied in order to characterize sedimentary organic matter based on neutral signature markers. Samples were exhaustively extracted, subjected to pre-chromatographic derivatizations and analyzed by GC/MS. Herein, molecular distributions of diagnostic alkanes/alkenes, terpenes/terpenoids, polycyclic aromatic hydrocarbons, aliphatic alcohols and ketones, sterols, and hopanes/hopanoids were addressed. Characterization of the contribution of natural vs. anthropogenic sources to the sedimentary organic matter in these extreme environments was then possible based on these distributions.

With the exception of polycyclic aromatic hydrocarbons, combined concentrations across all marker classes proved higher in the surface sediment layer as compared to those in the deep sediment layer. Alkane and aliphatic alcohol distributions pointed to predominantly allochthonous over autochthonous contribution to sedimentary organic matter. Sterol patterns were dominated by phytosterols of terrestrial plants including stigmasterol and β-sitosterol. Hopanoid markers with the $\beta\beta$-biohopanoid "biological" configuration were more abundant in the surface sediment layer, which pointed to higher bacterial activity. The pattern of polycyclic aromatic

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hydrocarbons pointed to prevailing anthropogenic input. Pyrolytic makers were likely to due to atmospheric deposition from a nearby former coal combustion facility. The combined analysis of the array of biomarkers provided new insights into the sources and transformations of organic matter in lake sediments.

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1. Introduction

Lake sediments constitute archives of environmental processes. Sedimentary organic matter (SOM) is derived from both allochthonous and autochthonous sources. The former originate from terrestrial vascular plants, while the latter from aquatic phytoplankton and aquatic macrophytes. In addition to natural sources, anthropogenic inputs to SOM can be significant, especially in areas of recent and/or former industrial activities. Typically, the fraction of SOM decreases exponentially with increasing sediment depth, which is associated with microbial activity. Sediments from lakes related to former lignite mining activities contain elevated concentrations of organic matter (Blodau et al., 2000), yet microbial activity in these sediments is often limited by the lack of organic substrates. This obvious contradiction is due to high content of refractory lignite-derived organic matter, as well as to stabilization of SOM by iron minerals (Laskov et al., 2002).

To study different SOM sources, biomarker approach using lipids as intrinsic, source-specific constituents of prokaryotic and eukaryotic cells has proved very useful (see Pisani et al. (2013) and references cited therein). Such an approach using fatty acids as biomarkers was applied to sediments from an acidic mining lake (AML) (Poerschmann et al., 2012). Fatty acids in this extreme habitat provided information useful for determining the contributions of autochthonous and allochthonous inputs to the SOM. However, fatty acids as the only markers cannot comprehensively distinguish natural from anthropogenic SOM sources. In addition, fatty acids are only partially capable of differentiating between various natural SOM sources. As an example, linoleic acid (18:2ω3,6cc) may originate from both vascular plants and from algae. The association of linoleic acid with typical sterols (see Section 3.6.) would confirm the algal origin. Thus, the significance of the biomarker approach when determining the contribution of different sources to SOM production and its depositional conditions can be enhanced by utilizing an array of different lipid biomarker classes rather than just a single class.

In contrast to "natural" markers, the pattern of polycyclic aromatic hydrocarbons (PAHs) has been shown to appropriately reflect the anthropogenic impact on SOM (see Lohmann et al. (2005) and references cited therein). However, PAHs lack source specificity when considering autochthonous vs. allochthonous inputs.

In the framework of this contribution, a wide array of neutral signature families other than fatty acids was dealt with:

(i) Alkanes, long-chain alcohols, n-alkan-2-ones, terpenes/terpenoids and sterols were used to discriminate autochthonous aquatic sources of SOM versus allochthonous terrestrial sources. Autochthonous aquatic sources in the lake under study were limited to algae and bacteria, as no submerged or floating macrophytes were present there. Consequently, throughout the remainder of the text, autochthonous/aquatic will be synonymous with algal/bacterial. On the other hand, allochthonous/terrestrial will include material derived from vascular plants, anthropogenic sources and atmospheric deposition. In the framework of the contribution we considered reeds and bulbous rush, which are formally emergent macrophytes, to be allochthonous, because they were growing mostly above the waterline and in nearby wetlands.
(ii) PAHs were used to characterize anthropogenic input.
(iii) Hopanes/hopanoids as membrane stabilizers of prokaryotes were used to assess the bacterial community composition.

To release lipidic biomarkers, sediment samples were subjected to exhaustive solvent extraction using pressurized liquid extraction (PLE). GC/MS-identification of functionalized target analytes (alkanols, sterols, hopanols) was facilitated by pre-chromatographic silylation to produce non-polar TMS (trimethylsilyl) derivatives. Covalently bound markers such as phytol were released by alkaline saponification of dried sediments. Herein, the biomarker content of two sediment layers from an AML was analyzed to obtain information on the sources and diagenetic transformation of organic matter in the sediments.

2. Materials and methods

2.1. Study site

Sediment samples whose fatty acid patterns have been characterized earlier (Poerschmann et al., 2012) were taken from Mining Lake 111 (AML 111) in the Lusatian mining district. The lake is one of the most studied pit lakes worldwide (Koschorreck, 2013). Briefly, the lake had a pH = 2.6 with the water body containing high concentrations of sulfate (12.5 mM) and (buffering) iron (Fe^{3+}, 2.5 mM). Sediment samples were collected at the deepest point in the northern basin at a water depth of 6.5 m using a gravity corer. Sediment cores were subsampled for the surface sediment layer (0–1 cm) and deep sediment layer (4–5 cm) and preserved at 4 °C under anoxic conditions. The total inorganic carbon (TIC) in either sediment amounted to ~2 mmol L^{-1}. Vertical profiles of oxygen concentrations revealed a decline in the hypolimnion, but always remained above 2 mg L^{-1} (Kamjunke et al., 2004). The underwater light intensity proved sufficient for net primary production for most of the benthic algae (Kamjunke et al., 2004). The lake is fed by groundwater (pH 4, up to 1 g L^{-1} iron concentration). There is no surface outflow, but a groundwater outflow. The water residence time is about 5 years (Koschorreck, 2013).

2.2. Chemicals

Solvents and internal standards were pesticide residue grade from Sigma-Aldrich (Munich, Germany); the same holds true for the silylation agent bis(trimethylsilyl)trifluoracetamide (BSTFA). Solvents were degassed prior to use. Deuterated internal standards (IST) [$^2H_{10}$]phenanthrene (phenanthrene-d_{10}) to calibrate PAHs, [$^2H_{34}$]n-hexadecane to calibrate n-alkanes, n-alkanols, ketones, and squalene, and [$^2H_6$]cholesterol (2,2,3,4,4,6) to calibrate sterols, were purchased from Promochem (Wesel, Germany). Authentic standards included a PAH Mix (Cat. 861291-Sigma-Aldrich), sterols (cholesterol, ergosterol, campesterol, stigmasterol, β-sitosterol) and fatty alcohol mixture nC_{16}, nC_{18}, nC_{20} (Cat. 59020, Sigma-Aldrich). Authentic standards available from previous activities included phenols (phenol, cresols, xylenols), phytol, n-alkanes (nC_{10}–nC_{30}), C_1–C_3 alkylated naphthalenes, an array of mono- and sesquiterpenes including limonene (serving to calibrate terpenes/terpenoids), as well as 12 individual hopane hydrocarbon standards (listed in the Supplementary Information section). The latter were a gift from Dr. H. Richnow (UFZ). Among them was 17α(H)22,29,30-tris-norhopane, which served to calibrate hopanes.

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