

Sedimentary evolution and lake level fluctuations of Urmia Lake (north-west Iran) over the past 50 000 years; insights from *Artemia* faecal pellet records

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ABSTRACT

A 25 m long sediment core from hypersaline Urmia Lake (north-west Iran) was studied for the Late Quaternary depositional history and palaeoclimate variations using the abundance and compositional characteristics of *Artemia* faecal pellets. Sediment analysis is supported by scanning electron microscopy – energy dispersive X-ray spectroscopy, organic and inorganic carbon content measurements, and stable isotopes ($\delta^{13}\text{C}$ and $\delta^{18}\text{O}$) from faecal pellet carbonates. The imprecise chronology of the core back to 50 kyr BP is supported by ten radiocarbon ages from faecal pellets and bulk sediments. The palaeoenvironmental record is subdivided into four periods: (i) During much of Marine Isotope Stage 3, a period of lake level lowering is characterized by a decreasing amount of faecal pellets, and an increasing amount of coated grains, sulphate minerals and reworked shell fragments. (ii) During late Marine Isotope Stage 3 and early Marine Isotope Stage 2 a lake level lowstand and a lake floor exposure is interpreted based on the relatively low abundance of pellets, which are multicoloured and appear together with volcanic lithics and rounded sulphate minerals. (iii) During late Marine Isotope Stage 2 the record is devoid of pellets but dominated by large sulphate crystals suggesting a prolonged low lake level. (iv) During Marine Isotope Stage 1 a relative lake level highstand is rapidly established with sediments that are highly abundant in fresh pellets. The modern lake level lowstand is represented by a salt crust. The $\delta^{13}\text{C}$ and $\delta^{18}\text{O}$ records measured from faecal pellet carbonates suggest a link with the precipitation versus evaporation balance in the lake over time. From bottom to top the linear trend towards more negative delta values illustrates the increasing amount of precipitation arriving at the lake from the Late Pleistocene to the Holocene. Two prominent isotope minima during the Late Pleistocene and one prominent minimum in the early Holocene mark relative high lake levels, which can also be linked to Lake Van in Turkey.

Keywords *Artemia* faecal pellet, deposition history, lake level fluctuation, radiocarbon age, stable oxygen and carbon isotopes, Urmia Lake.

INTRODUCTION

Hypersaline lakes with salinity higher than >50 g/l are sensitive sedimentary environments, and they allow inferences about past environmental, climate and tectonic events (Hammer, 1986; Anati, 1999; Jones & Deocampo, 2003; Sharifi *et al.*, 2018). Hypersaline ecosystems have semi-arid/arid climates, high dissolved ion concentrations, alkaline water and low nutrient availability, and are thus considered exceptional natural laboratories. These ecosystems have been studied for reconstructing changes in their physicochemical, sedimentological and biological properties (Williams & Liebhold, 2002; Alipour, 2006; Oren, 2009; Lak *et al.*, 2011, 2012, 2022; Gajardo & Redón, 2019; Mohammadi *et al.*, 2021, 2023).

Because of the extreme salinity, hypersaline lakes have low aquatic biodiversity; however, the brine shrimp *Artemia* has adapted well to living in saline and hypersaline waters (e.g. Gajardo & Beardmore, 1989, 2012). As a consequence, *Artemia* sp. faecal pellet abundance, shape and surface morphology, and its mineralogical, geochemical and isotopic composition can provide useful complementary proxy data for inferring lake hydrology change, where other fauna and flora remains in the sediments are very limited (Kelts & Shahrabi, 1986; Djamali *et al.*, 2010; Stevens *et al.*, 2012; Blasi *et al.*, 2020; Kong *et al.*, 2022). For instance, the differences in the mineralogy of evaporitic precipitates associated with the presence/absence of *Artemia persimilis* faecal pellets allowed identifying different periods that suggest changes in the Chasico Lake (Argentina) hydro-chemical environment (Blasi *et al.*, 2020). On the other hand, *Artemia salina* faecal pellets at the tropholytic zone in Techirghiol Lake (Romania) serve as a reliable criterion for the precipitation of high-magnesian calcite inside faecal pellets via bacterial metabolism (Baltres & Medessan, 1978). *Artemia* has effects on the lake water geochemistry by absorbing calcium and magnesium cations and carbonate anions from the lake water and defecates them as faecal pellets, which causes the depletion of lake water from the aforementioned ions and reduces the chemical deposition of carbonate in the lake (Mohammadi *et al.*, 2021). Similarly, *Artemia* controls the mineralogy and sedimentation pattern with dominant biochemical deposits in Urmia Lake (e.g. Kelts & Shahrabi, 1986; Djamali *et al.*, 2010; Stevens *et al.*, 2012; Mohammadi *et al.*, 2021).

Additionally, faecal pellets are important constituents of hypersaline lake sediments because they consist of organic matter, carbonate and variable amounts of different terrigenous and clay minerals. Faecal pellet compositional change can record environmental conditions of the depositional time. They contain organic and inorganic carbon, which allows ^{14}C dating, $\delta^{18}\text{O}$ and $\delta^{13}\text{C}$ isotope analysis, TOC (total organic carbon), ROC (residual oxidizable carbon) and TIC (total inorganic carbon) analysis (e.g. Kelts & Shahrabi, 1986; Stevens *et al.*, 2012). Palynology, limnology, sedimentology and mineralogy studies of Urmia Lake sediments have already provided important data for reconstructing palaeoclimate, palaeohydrology and palaeoenvironment changes of the Turkish–Iranian Plateau (Bottema, 1986; Kelts & Shahrabi, 1986; Mohammadi, 2005; Djamali *et al.*, 2008, 2010; Amini *et al.*, 2010; Manaffar *et al.*, 2011; Stevens *et al.*, 2012; Talebi *et al.*, 2016; Sharifi, 2017; Sharifi *et al.*, 2018; Mohammadi *et al.*, 2019, 2021; Tudryn *et al.*, 2021; Kong *et al.*, 2022). However, faecal pellets as an important lake sediment constituent have received insufficient attention for their potential application in the Urmia Lake palaeoenvironmental reconstruction, and in palaeoclimate and palaeohydrology studies (e.g. Kelts & Shahrabi, 1986; Stevens *et al.*, 2012; Sharifi, 2017; Kong *et al.*, 2022). The study objectives are: (i) to reconstruct the Late Pleistocene–Holocene Urmia Lake palaeoenvironment history using frequency, morphology, isotopic composition, mineralogy and chronology from *Artemia* faecal pellets; (ii) to reconstruct the sedimentary evolution of the lake deposits and relative lake water level fluctuations using controlling factors such as the clastic sediment supply, the formation of chemical–biochemical components, *Artemia* faecal pellet abundance change and diagenesis processes.

STUDY AREA

Urmia Lake in Iranian Azerbaijan (north-west Iran) occupies a part of the eastern segment of the Turkish–Iranian Plateau (Fig. 1). The lake covers an area of approximately 5000 km², it is 140 km long, and is between 15 km and 50 km wide. It is comparable with the Great Salt Lake in the USA in terms of area and water volume (e.g. Kelts & Shahrabi, 1986; Azari Takami, 1987). After the extreme water level fall during the past decades, in 2019 the maximum water depth was

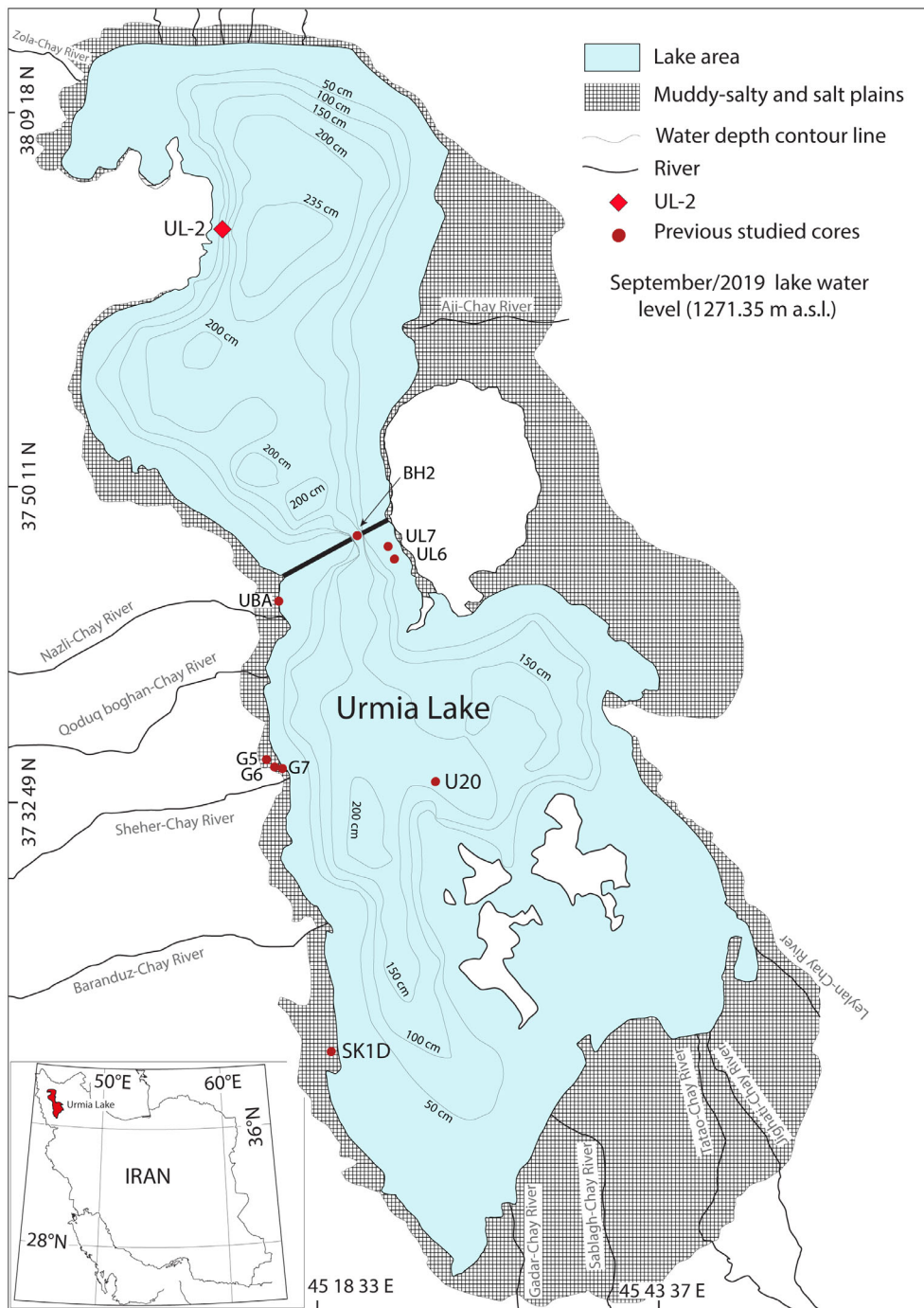


Fig. 1. Location of Urmia Lake in north-west Iran and the position of the studied core UL-2. Positions of previously dated sediment cores are also shown; Urmia 20 (Kelts & Shahrabi, 1986), BH2 (Djamali *et al.*, 2008; Stevens *et al.*, 2012), UBA (Manaffar *et al.*, 2011), SK1D (Talebi *et al.*, 2016), UL6, UL7 (Sharifi, 2017), G5, G6, G7 (Tudryn *et al.*, 2021), G6, G7 (Kong *et al.*, 2022). The lake map was modified from Mohammadi *et al.* (2021).

only 2.35 m in the northern part of the lake (Mohammadi *et al.*, 2023). The endorheic lake is located at an altitude of 1270 m above sea level (m.a.s.l.) in a semi-arid area with 330 mm of

mean annual precipitation and a mean annual temperature of 9.4°C (Darvishi Khatuoni *et al.*, 2011, 2015). The lake water is supplied by 28 perennial and ephemeral rivers, rain and

snowfall, and underground waters and springs (e.g. Amini *et al.*, 2010; Mohammadi *et al.*, 2021; Lak *et al.*, 2022). Urmia Lake can be characterized as a thalassohaline lake in terms of the predominance of Na⁺ and Cl⁻ ions; also, the lake water salinity varies between 120 g/l and 380 g/l depending on location, water depth and season (e.g. Sorgeloos, 1997; Alipour, 2006; Karbassi *et al.*, 2010; Sharifi *et al.*, 2018; Lak *et al.*, 2022; Mohammadi *et al.*, 2023). Particularly, Aji-Chay River, which drains the Miocene evaporite-rich sedimentary rocks in the north-eastern part of the catchment, is an important source of the lake water dissolved ions (Amini *et al.*, 2010; Mohammadi *et al.*, 2019, 2021, 2023; Lak *et al.*, 2022). In the middle part of the lake, a 15 km long stone causeway has limited the water exchange between the northern and southern parts of the lake since 1989, and it affects the lake hydrodynamic system by diverging currents, changing the sedimentation pattern and the water physico-chemical parameters (e.g. Zeinoddini *et al.*, 2009; Mohammadi *et al.*, 2021, 2023; Lak *et al.*, 2022).

Geological setting

The intra-continental Urmia Lake formed in a tectonic depression related to the Tabriz and Zarrineh–Rood strike-slip faults in the eastern part of the Turkish–Iranian Plateau (Berberian & King, 1981; Eftekhari-Nezhad *et al.*, 1989). In the lake catchment, rocks range in age from Precambrian to Quaternary, with variable lithologies including felsic to mafic volcanic and plutonic rocks, ultramafic ophiolitic rocks, low-grade to high-grade metamorphic rocks, terrigenous, carbonate and evaporite sedimentary rocks, and unconsolidated alluvial, fluvial and lacustrine deposits (e.g. Shahrabi *et al.*, 1985; Eftekhari-Nezhad *et al.*, 1989; Sharifi *et al.*, 2018; Delavari *et al.*, 2019; Mohammadi *et al.*, 2020, 2022). The lithology of exposed rocks in the different parts of the catchment is important because it controls the ionic budget of the draining rivers (Amini *et al.*, 2010; Sharifi *et al.*, 2018; Mohammadi *et al.*, 2021; Lak *et al.*, 2022). For example, the exposure of poorly consolidated and erodible Neogene evaporite-rich continental sediments in the north-eastern part of the lake catchment causes an extreme increase in the ionic budget of the Aji-Chay River, and the maximum amount of the lake water ions is supplied from this river (e.g. Amini *et al.*, 2010; Mohammadi *et al.*, 2021).

Artemia urmiana Günther (Crustacea: Anostraca)

The *Artemia* genus of brine shrimps lives in natural saline and hypersaline lakes and man-made salterns along seashores and in inland waters throughout the world. *Artemia* species prefer to live in chloride, carbonate and sulphate-rich water bodies (e.g. Van Stappen *et al.*, 2001; Wurtsbaugh & Gliwicz, 2001). They are non-selective filter feeders, feeding on detritus delivered by rivers and scraped up from the bottom, as well as microalgae and plankton of the water column (e.g. Wurtsbaugh & Gliwicz, 2001). *Artemia* species are adapted to tolerating high salinities ranging from 45 to 340 g/l (e.g. Post & Youssef, 1977; Persoone & Sorgeloos, 1980).

Hypersaline Urmia Lake is known to be one of the largest natural *Artemia* habitats around the world, containing both bisexual and parthenogenetic *Artemia* populations (e.g. Azari Takami, 1987). Bisexual *Artemia urmiana* was thought to be unique to Urmia Lake until it was discovered in 2008 in Lake Koyashskoe, a hypersaline lake on Ukraine's Crimean peninsula (Shadrin *et al.*, 2008). Thus, *Artemia urmiana* is no longer classified as endemic.

MATERIALS AND METHODS

In 2017, the Iran Water and Power Resources Development Company recovered a 25 m long sediment core (UL-2) from the north-west near-shore area of Urmia Lake (Fig. 1) applying the Shelby method. At the Istanbul Technical University (ITU) – Eastern Mediterranean Center of Oceanography and Limnology Applied Research Center (EMCOL), the core was opened, cleaned, described, photographed and sampled. In total, 140 sediment sub-samples were collected based on facies variations along the core. Each sample weighed 2 g, and each was collected from a single layer and subdivided for further analysis. Following that, samples were washed with distilled water, sieved into 63 to 125 µm and >125 µm fractions, and oven-dried at 60°C for one day. This aided an improved visual core description using the binocular microscope.

Faecal pellet and bulk sediment ¹⁴C dating

Ten under-binocular microscope picked faecal pellet samples (10 mg) and seven bulk sediment samples (30 g) were collected for accelerator mass spectrometry (AMS) ¹⁴C dating (Table 1).

Table 1. Conventional ^{14}C radiocarbon age, and calibrated ages of *Artemia* faecal pellets and bulk sediment from different depths of UL-2 sediment core. Calibrations were performed using Calib 8.2 (<http://calib.org/calib/calib.html>). The ages marked by stars were used for the studied core chronology.

Depth (m)	Material	Conventional ^{14}C age (yr BP) $\pm 1\sigma$	Cal ^{14}C ages (cal yr BP) $\pm 2\sigma$
3.67	Faecal Pellet	Not enough carbon	–
3.75	Bulk sediment	9949 $\pm$ 36	11 350 $\pm$ 80
4.72	Bulk sediment	10 800 $\pm$ 53	12 750 $\pm$ 60
*5.03	Bulk sediment	7543 $\pm$ 35	8360 $\pm$ 50
*6.01	Bulk sediment	10 631 $\pm$ 64	12 650 $\pm$ 70
6.04	Faecal Pellet	Not enough carbon	–
6.23	Faecal Pellet	23 000 $\pm$ 220	27 300 $\pm$ 420
*9.98	Faecal Pellet	20 341 $\pm$ 172	24 460 $\pm$ 500
*10.10	Bulk sediment	22 389 $\pm$ 71	26 730 $\pm$ 280
14.14	Faecal Pellet	37 267 $\pm$ 730	41 830 $\pm$ 830
*16.90	Bulk sediment	33 411 $\pm$ 167	38 230 $\pm$ 750
*17.46	Faecal Pellet	33 000 $\pm$ 487	37 660 $\pm$ 1350
*19.90	Bulk sediment	37 154 $\pm$ 224	41 920 $\pm$ 330
19.95	Faecal Pellet	Not enough carbon	–
*21.75	Faecal Pellet	43 982 $\pm$ 1492	46 500 $\pm$ 2860
*22.84	Faecal Pellet	42 268 $\pm$ 1246	44 980 $\pm$ 2090
*24.76	Faecal Pellet	45 144 $\pm$ 1725	47 690 $\pm$ 3750

The dried samples were ground in a pestle to make them into powder. Before proceeding with the analysis, the carbon percentage (%) was measured using elemental analysis (EA) to confirm enough carbon for AMS measurement. The sample was about 50 mg for EA. After cleaning and combustion in closed quartz tubes, the generated CO_2 is graphitized using H_2 (Hajdas *et al.*, 2004). The faecal pellets were dated using a MICADAS radiocarbon facility at ETH Zürich, Switzerland. Bulk sediment dating was conducted in the AMS laboratory at the Korea Institute of Science and Technology, South Korea. The dated carbons from *Artemia* faecal pellets and bulk sediments are inorganic and organic carbon, respectively. Sample preparation and dating steps are described in detail by Wacker *et al.* (2010) and Kim *et al.* (2017), respectively. Due to the lack of materials that absorb their carbon from the atmosphere for dating, and comparing those ages with radiocarbon ages of contemporaneously deposited *Artemia* faecal pellets and bulk sediment (received

their carbon from the lake water), the old carbon reservoir effect for Urmia Lake was not calculated.

The measured ages (yr BP) were calibrated to calendar ages (cal yr BP) based on the IntCal20 radiocarbon calibration, which is implemented in the web-based Calib 8.20 software (calib.org).

Total organic carbon, residual oxidizable carbon and total inorganic carbon of faecal pellets

Three carbon fractions from faecal pellet samples linked to the thermal destabilization of organic and inorganic phases were analysed at the Alfred Wegener Institute (AWI, Potsdam, Germany). Natali *et al.* (2020) demonstrated that thermochemical diagrams reveal three carbon fractions associated with thermal destabilization of organic and inorganic phases; these fractions are measured at 400°C (TOC), 600°C (ROC) and 900°C (TIC). In total, 20 dried and powdered faecal pellet samples (each sample 10 mg;

Table S1) were measured using a Soli TOC Cube elemental analyser (Elementar, Stockport, UK). The measurements were performed in accordance with the DIN19539 standard protocol (DIN Standards Committee Water Practice, 2016). In addition, five analysed control standards show a device-specific accuracy of ± 0.1 wt% (weight percent): 0.1 wt% is also used as the detection limit. For the assessment of faecal pellet composition, the organic matter and the carbonate contents were estimated using the following equations (see Kwiecien *et al.*, 2008; Pribyl, 2010):

$$\begin{aligned} \text{Organic matter content(\%)} : \text{TOC} + \text{ROC} \\ = (\text{TOC} + \text{ROC}) \times 100/50 \end{aligned} \quad (1)$$

$$\text{Carbonate content (\%)} : (\text{TIC} \times 100)/12 \quad (2)$$

Faecal pellet stable inorganic oxygen ($\delta^{18}\text{O}$) and carbon isotopes ($\delta^{13}\text{C}$)

Twenty faecal pellet samples (each sample 10 mg; Table S2) were measured for retrieving inorganic $\delta^{18}\text{O}$ and $\delta^{13}\text{C}$ isotope ratios at AWI (Bremerhaven) using a Thermo Scientific MAT 253 mass spectrometer coupled to a Kiel IV Carbonate Device (Thermo Fisher Scientific, Waltham, MA, USA). Beforehand, samples have been heated up to 500°C over 4 h to remove organic carbon. Measurements are reported in δ notation in comparison to the VPDB (Vienna Pee Dee Belemnite). The repeatability precision was better than ± 0.07 for $\delta^{18}\text{O}$ and ± 0.03 for $\delta^{13}\text{C}$ based on an internal laboratory standard (SHK-2020). During the analysis, the laboratory standard was repeatedly measured for verification of instrumental stability (14 times in total).

Thin section study

At the ITU Eurasia Institute of Earth Sciences Thin Section Laboratory, 21 samples of faecal pellets, coated grains and shell fragments (Table S3) were carefully placed onto Kapton® double-sided tape and hardened with Struers® epoxy resin. The epoxy was attached to a glass slide and manually ground and polished, resulting in a sample thickness of 30 μm . Eventually, various sediment component internal structures were studied and photographed from the thin sections using a polarized light microscope.

Scanning electron microscopy with energy dispersive spectrometer

Faecal pellet surface micromorphology and external and internal structure information was obtained under SEM for 26 samples taken from different core depths (Table S4). The gold-coated sputter technique was applied using the SEM ZEISS IGMA VP with EDS detector (Carl Zeiss, Oberkochen, Germany) at the Research Institute for Earth Science, Geological Survey of Iran. The EDS detector delivered point information on element composition (O, Ca, C, Si, Al, Mg, Fe, Na, K, S, Mo, Ti and N). Detailed elemental distribution maps were produced and internal structures of faecal pellets were examined at Hacettepe University Advanced Technologies Application and Research Center (HUNITEK) on a Carl Zeiss EVO 50 SEM, which is combined with a Bruker AXS X Flash 3001 EDS (Bruker, Billerica, MA, USA).

X-ray diffraction analysis

Mineralogical analysis of five powdered faecal pellet samples and one sample of coated grains (with faecal pellet core; Table S5) was performed at the Istanbul Technical University X-Ray Laboratory of the Geological Engineering Department using a Bruker D8 Advance XRD. Diffractions were revealed using a dedicated Lynxeye detector (Bruker) for 0 to 70° 2 θ with 0.1°/sn step speed at 40 kV and 40 mA using a Cu K α radiation source at 25°C. The MDI Jade software program was used to evaluate the diffractograms, and characteristic peak positions allowed the authors to infer the mineral composition.

RESULTS

Chronology

The radiocarbon chronology analysis is determined from 10 faecal pellets and seven bulk sediment samples (Table 1). However, three of the faecal pellet samples had insufficient carbon for dating, also two faecal pellet ages and two bulk sediment ages produced age reversals. That is why the chronology and a depth-to-age relation of the studied sediment core was eventually based on 10 radiocarbon datings. The maximum radiocarbon age (47.6 cal kyr) of the core bottom points to MIS 3 (Marine Isotope Stage 3). When the age distribution is grouped according to MIS

the core is classified into three parts: the lowermost core age and five more ages from overlying sediments fall into MIS 3 (25 to 12 m); two ages cover MIS 2 (12 to 6 m); and two ages document MIS 1 (6 m to recent; Table 1; Fig. 8). The average sedimentation rate of the core between 24.76 m and 9.98 m depth is about 0.62 mm/year (MIS 3 to early MIS 2), between 9.98 m and 6.01 m core depth the sedimentation rate decreases to about 0.34 mm/year (MIS 2), and the sedimentation rate further decreases to about 0.25 mm/year between 6.01 m and 5.03 m core depth (MIS 1).

Facies description

According to the lithological observations together with mineralogical analysis through the studied core, various macroscopic and microscopic sediment features were documented that allow the authors to define different sedimentary facies. In addition to sediment layer thickness and physical appearances, abundance, shape and colour of *Artemia* faecal pellets were also noted. Detrital and biogenic components are mainly represented by sulphate minerals, coated grains, volcanic lithics, volcanic glass, mica, feldspar, pyroxene, shell fragments and grapestones.

Based on sediment and faecal pellet description, the core is subdivided into five main facies from bottom to top:

Facies 1 (25.00 to 14.72 m depth; Fig. S1) is primarily composed of greyish brown sandy mud, it holds coated grains, anhedral and cement type sulphate minerals (dominantly gypsum), a minor amount of brownish broken pellets, and shell fragments are admixed (Fig. 2A). Grey sandy mud with diagenetic sulphate minerals and faecal pellets are visible at depths of 22.24 to 21.68 m, 19.39 to 18.20 m, 17.09 to 16.67 m, 16.10 to 15.80 m and 15.20 to 14.72 m. Less commonly, greyish brown medium-grained sandy mud with sulphate minerals, coated grains and faecal pellets is common at depths of 22.61 to 22.25 m, 21.67 to 19.40 m and 17.49 to 17.10 m. Very dark grey coarse-grained sand with coated grains, shell fragments and some faecal pellets are found between 25.00 to 22.62 m and 18.19 to 17.50 m depths. Occasionally, distinct sediment layering with colour change from light to dark is observed: i.e. at 22.84 m, 22.17 m, 21.68 m, 19.95 m, 19.76 m and 17.43 m depths. In other cases, sediment change is gradual from one layer to another. At depths of 18.75 to 18.66 m and 18.30 to 18.24 m, light

brownish to grey-coloured clay is intercalated with white thin layers of coarse-grained sulphate minerals. This is the most notable change in the facies (Fig. S1).

Facies 2 (14.71 to 8.15 m depth; Fig. S2) is represented by a uniform dark greyish-brown mud with predominately rounded and fine-grained sulphate minerals. Coated grains and fresh faecal pellets are scattered throughout. Faecal pellets are multi-coloured black, orange, brown and dotted. They are admixed with black and brown coloured volcanic lithics (up to 2.5 mm in size) and volcanic glasses are scattered at depths of 14.72 to 12.30 m and 9.60 to 9.00 m (Fig. 2B). There are light grey-coloured layers containing fine-grained anhedral gypsum minerals at depths of 10.13 to 10.19 m, 11.00 to 11.07 m and 13.83 to 13.86 m. Black-coloured laminae occur sporadically at depth intervals of 14.60 to 14.00 m and 12.80 to 11.87 m, with volcanic lithics and glass visible under a binocular microscope (Fig. S2).

Facies 3 (8.14 to 6.11 m depth; Fig. S3) is defined as grey coloured coarse-grained sand size euhedral sulphate minerals (Fig. 2C). The sand-sized sediments are interrupted by brownish mud at depths of 8.05 to 7.90 m, 7.62 to 7.51 m, 7.40 to 6.83 m and 6.35 to 6.27 m, while white-coloured thin laminae occur at depths of 6.55 m and 6.13 m. Sulphate minerals are abundant in Facies 3 at two depth intervals: 7.90 to 7.40 m and 6.80 to 6.10 m. The sediment in the upper part of this facies, between 6.28 m and 6.23 m depth, turns to grey-brown and is composed of fine silt and clay rich in dotted and multi-coloured faecal pellets. Volcanic glasses are observed at depths of 8.14 to 7.40 m and 6.30 to 6.20 m (Fig. S3).

Facies 4 (6.10 to 3.20 m depth; Fig. S4) is composed of greenish-grey fine sand, silt and micrite, and is dominated by fresh faecal pellets (Fig. 2D). It begins with an unconformity at the base and continues with greenish-grey silty clay and intercalated fine sand layers. Above 5.60 m depth, a 0.13 m thick layer of oxidized light olive grey clay appears. A second unconformity is observed at a depth of 5.00 to 4.90 m. Faecal pellets are mostly fresh, soft and cream-coloured. In addition, a small amount of coated grains and grapestones are scattered in this facies (Fig. S5).

Facies 5 (3.19 to 0 m depth; Fig. S4) is composed of recent (last 25 years) evaporite deposits; i.e. halite and gypsum.

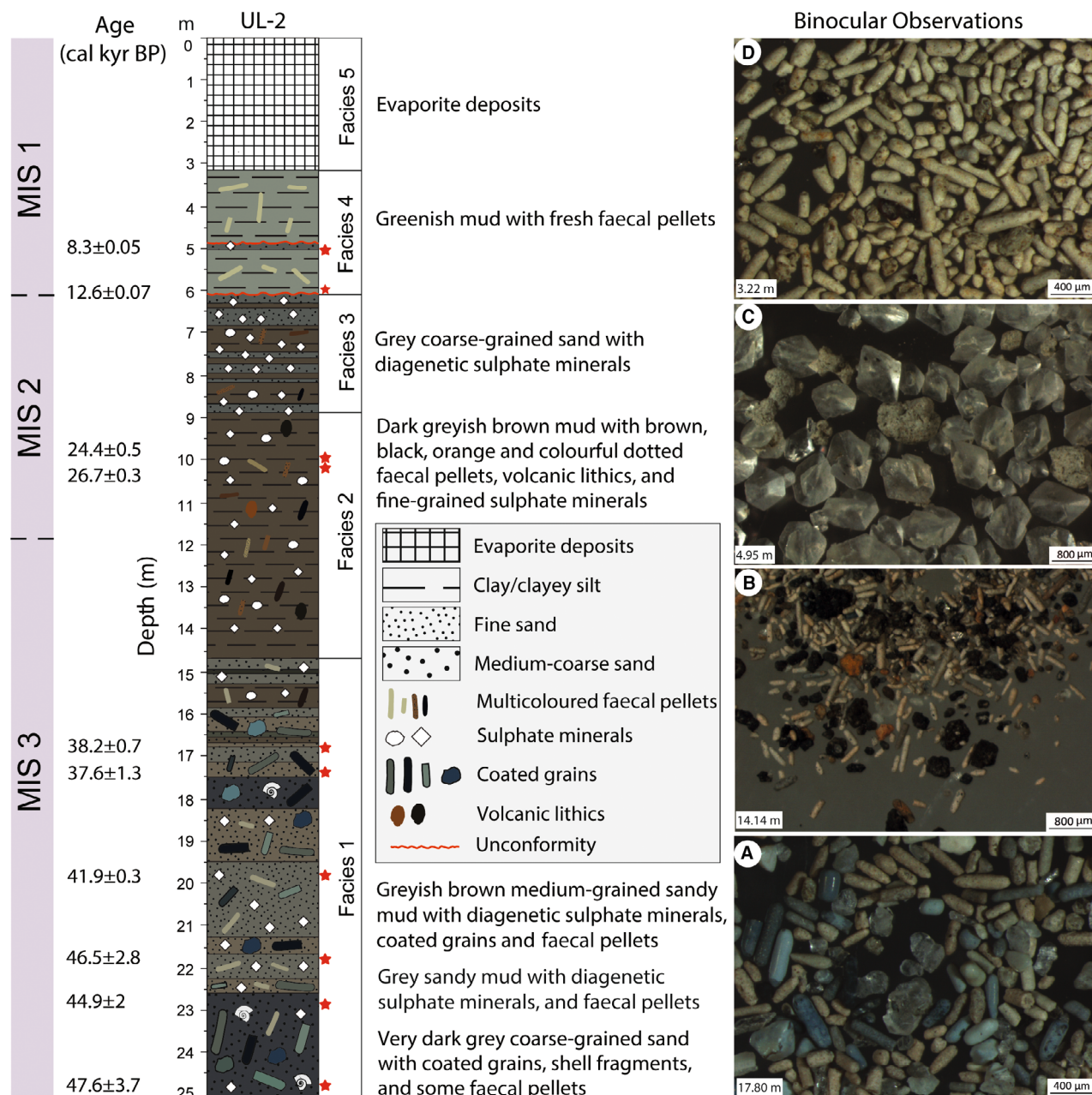


Fig. 2. Stratigraphy of the UL-2 core with numbered sediment facies. Examples of dominant facies components are shown: (A) a mixture of coated grains, sulphate minerals and some faecal pellets as typical of Facies 1; (B) heterogeneous and broken faecal pellets with volcanic lithics and rounded sulphate minerals as typical of Facies 2; (C) euhedral twinned sulphate minerals as typical of Facies 3; (D) fresh faecal pellets as typical of Facies 4. Colour-coding of sediment layers is based on the Munsell colour chart. Calibrated AMS ^{14}C ages are given with red stars.

Sediment components with particular emphasis on *Artemia* faecal pellets

Artemia faecal pellets are capsule-shaped, and they vary in size from silt to very coarse sand. Most pellets have lengths between 250 μm and

500 μm (medium to coarse sand), but they can reach a maximum length of over 1400 μm . The faecal pellets are predominantly cream (Fig. 3A), grey, light brown and orange coloured (Fig. 3B). Occasionally they form the core of coated grains

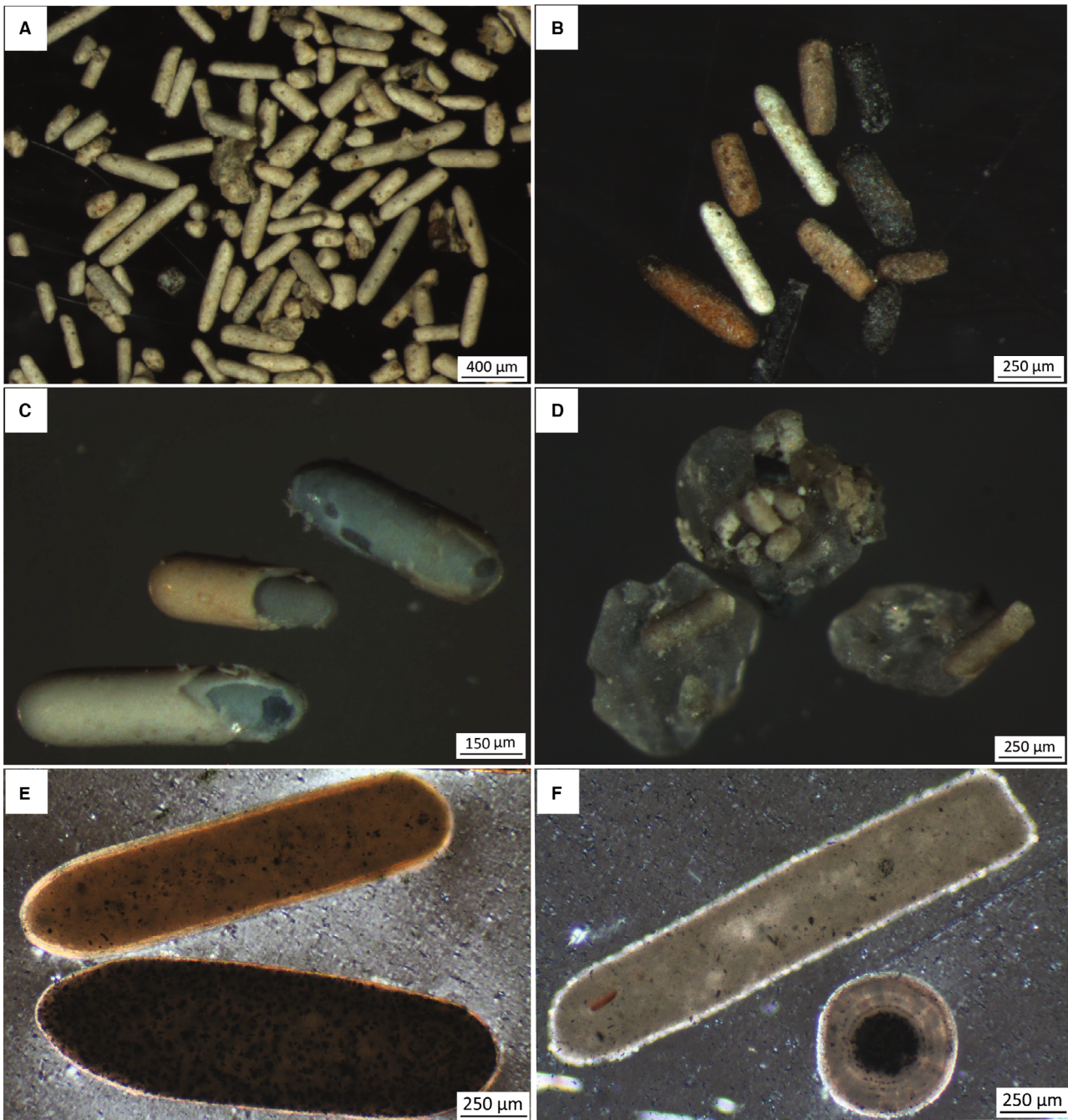


Fig. 3. Binocular microscope images of different types of faecal pellets: (A) cream-coloured faecal pellets (from 3.22 m depth); (B) multi-coloured broken faecal pellets with spotted surfaces (from 6.31 m depth); (C) faecal pellets in the core of coated grains (from 18.94 m depth); (D) faecal pellets enclosed by diagenetic sulphate mineral cement (from 22.27 m depth). Thin section images of faecal pellets: (E) spotted faecal pellets (from 16.73 m depth); and (F) faecal pellet with scattered spots as well as a little coated grain with a black faecal core and grey rims through the cross-cut (from 5.38 m depth).

(Fig. 3C), in some cases, they are randomly encased in sulphate mineral cement (Fig. 3D). Some faecal pellets show a uniform distribution

of the tiny spots (Fig. 3E), while others have larger spots, which are randomly scattered over the pellet (Fig. 3F). The SEM images of faecal

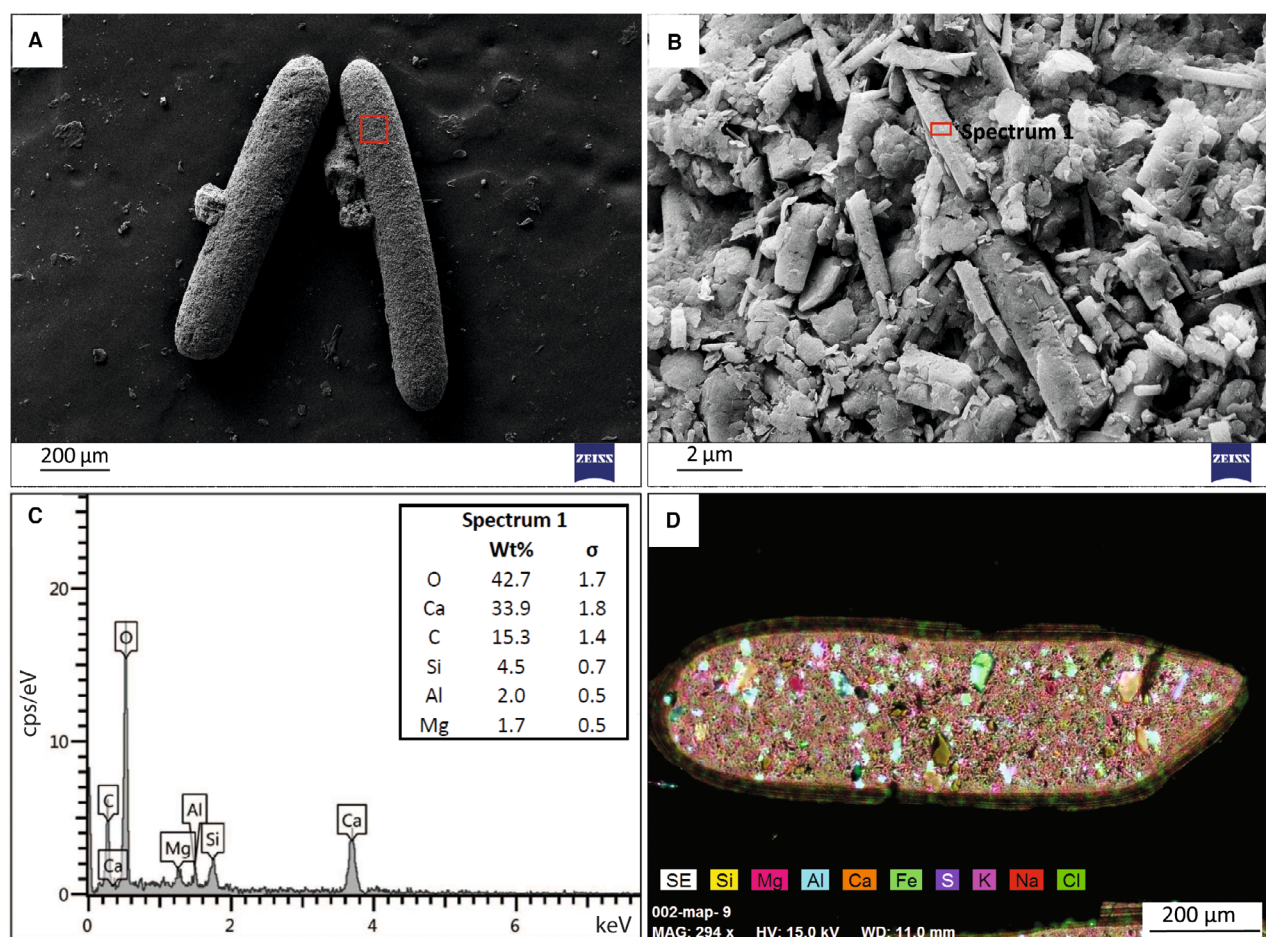


Fig. 4. Scanning Electron Microscopy (SEM) and Energy Dispersive Spectrometer (EDS) result for a faecal pellet and coated grain: (A) micrograph with a red square indicating the EDS analysis spot (from 3.67 m depth); (B) zoomed SEM micrograph of the same faecal pellet illustrating the multi-crystalline micritic surface of a faecal pellet; (C) EDS elemental analysis and proportional (wt%) of faecal pellet; and (D) SEM-EDS micrograph showing the elemental distribution pattern over another pellet sample in the core of a coated grain (from 14.04 m depth).

pellets demonstrate that their internal texture and surface morphology is not homogenous, in most cases they contain prismatic aragonite crystals and a minor amount of other minerals (Fig. 4A and B). The elemental composition of the faecal pellets as determined by SEM-EDS generally includes major amounts of O, Ca and C, and a trace amount of Si, Al and Mg (Fig. 4C). A longitudinal section through a pellet specimen in the core of a coated grain (Fig. 4D) shows an elemental distribution pattern where various elements (i.e. Si, Mg, Al, Ca, Fe, S, K, Na and Cl) are randomly arranged within the pellet. Expressed in the mineralogical composition, as determined by means of XRD, the faecal pellets are dominantly composed of aragonite, calcite and quartz, while in the coated grains with faecal pellet cores kaolinite and gypsum

are observed, too (Table S5). Coated grains with different shapes are made up of a core surrounded by a concentric lamina or laminae (Fig. 3C, E and F). Along the core (longest axis), sizes of coated grains vary between 350 μm and 1300 μm . The inner particles are mostly made of faecal pellets (Fig. 3C), clastic minerals, shell fragments and diagenetic sulphate minerals. Concentric rims are orange, dark blue, cream, white and beige in colour (Fig. 3C).

The diagenetic sulphate minerals (dominantly gypsum) along the core vary in shape and size, in brightness, and in impurity, partially they show twinning growth (Fig. 2C). Generally, their size is between 50 μm and 1.5 mm, but the largest crystal size has been found to measure 46 mm in length at depth of 7.15 m. Sulphate minerals are seen with euhedral cruciform

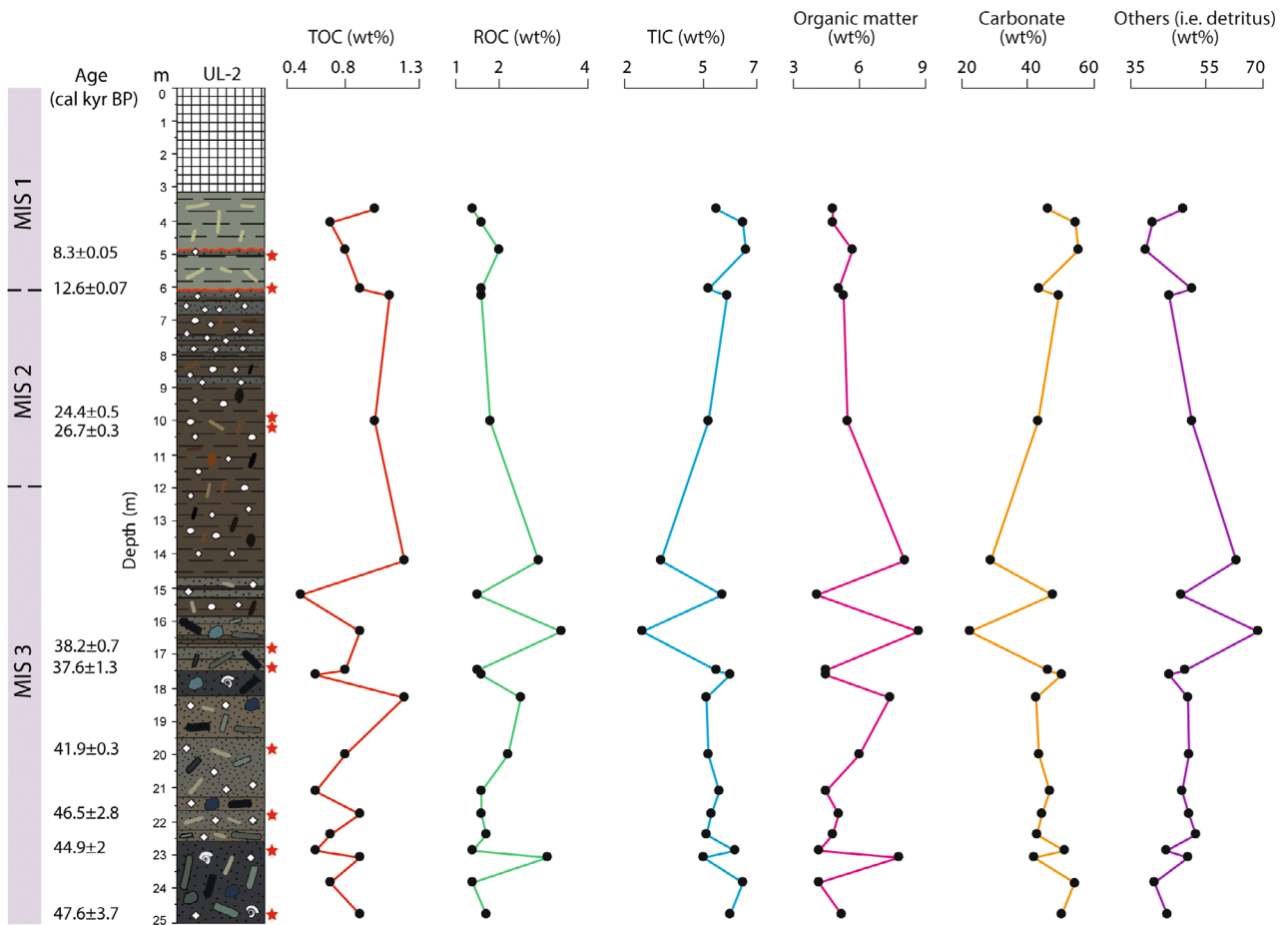


Fig. 5. UL-2 downcore faecal pellet TOC (total organic carbon), ROC (residual oxidizable carbon) and TIC (residual oxidizable carbon) elemental contents in weight-percent (wt%), and calculated faecal pellet composition (organic matter, carbonate and non-carbonate content); closed-sum portions of organic matter, carbonate and detritus.

twinning crystals, polycrystalline, subhedral and anhedral shapes, and they partially have a rounded shape or are of a cement type (Figs 2C and 3D).

Detrital minerals of different types and amounts are present along the core. Especially in the core bottom (Facies 1) they form the cores of coated grains. However, they are most abundant in the middle part of the core (Facies 2) as sand-sized black and brownish volcanic lithics and glass particles (Fig. 2B).

Total organic carbon, residual oxidizable carbon and total inorganic carbon of faecal pellets

Organic (TOC and ROC) and inorganic carbon (TIC) contents of faecal pellets narrowly range along the core, values are given in Table S1 and

Fig. 5. The highest TOC values of 1.2 wt% are recorded at 18.25 m and 14.14 m depths, while the lowest TOC value of 0.5 wt% is measured at 15.18 m core depth. The highest ROC content (3.4 wt%) is measured at a depth of 16.31 m, while the lowest ROC content (1.4 wt%) is measured at depths of 23.83 m, 22.84 m and 3.67 m. The lowest TIC values are found at a depth of 16.31 m (2.7 wt%) and at 14.14 m core depth (3.4 wt%), while all other TIC values range from 5.0 to 6.6 wt%. The TOC contents of faecal pellets are lower than ROC contents, but TOC and ROC contents generally parallel one another for much of the core. With the exception of the upper core part (6.00 to 3.00 m) TIC generally anticorrelates with the contents of TOC and ROC; i.e. TIC maxima are associated with minima of TOC + ROC and vice versa (Table S1; Fig. 5).

The portions of organic matter (wt%) and calcium carbonate (wt%) are estimated using Eqs 1 and 2, respectively. As determined from twenty measurements of TOC, ROC and TIC, the average composition of Urmia Lake *Artemia* faecal pellets has 5.5 wt% of organic matter (minimum 4.1 wt%, maximum 8.7 wt%), 45.2 wt% of carbonate (minimum 22.6 wt%, maximum 55.3 wt%) and 49.2 wt% of other components (minimum 39.0 wt%, maximum 68.7 wt%; Table S1). Other detrital components include clay minerals, quartz and feldspar grains. The highest values of the detrital portion are measured in the mid-core part (i.e. at 16.31 m and 14.14 m depth; Fig. 2B).

Oxygen isotopes ($\delta^{18}\text{O}$) and carbon isotopes ($\delta^{13}\text{C}$) of faecal pellets

Values of $\delta^{18}\text{O}$ and $\delta^{13}\text{C}$ from faecal pellets show a narrow range of variation along the core. The $\delta^{18}\text{O}$ values range between -3.52‰ and 1.14‰ , and $\delta^{13}\text{C}$ values vary between -5.68‰ and -0.88‰ . Generally, for $\delta^{13}\text{C}$, higher values are recorded in the bottom core part, and trend towards lower values farther upcore (Fig. 6). For $\delta^{18}\text{O}$ this trend is less distinct. The $\delta^{18}\text{O}$ maximum value is observed at 6.23 m depth, whereas the maximum value of $\delta^{13}\text{C}$ is recorded at 22.84 m core depth. The $\delta^{18}\text{O}$ linear trend (orange line) is almost stationary over the record, whereas the $\delta^{13}\text{C}$ values (blue line) have a slight trend towards more negative values from bottom to top (Fig. 6). Generally, the two sets of delta values go parallel over the record; a relative maximum of one isotope is accompanied by the relative maximum of the other one, the same is true for relative minimum values.

DISCUSSION

Faecal pellets in hypersaline lakes studies

The salinity of hypersaline lakes prevents fauna and flora from living there except for *Artemia* and halophyte algae species (e.g. Jensen, 1918; Sorgeloos *et al.*, 2001; Gajardo & Redón, 2019). Thus, changing *Artemia* faecal pellet properties in the sediments likely can act as a good proxy for inferring palaeoclimate, palaeoenvironment, palaeoecology, palaeosalinity and palaeobiology of hypersaline lakes (e.g. Eardley & Gvosdetsky, 1960; Baltres & Medegšan, 1978; Kelts & Shahrabi, 1986; Djamali *et al.*, 2010; Stevens *et al.*,

2012; Oviatt *et al.*, 2015; Maszczyk & Wurtsbaugh, 2017; Sharifi, 2017; Blasi *et al.*, 2020; Kong *et al.*, 2022). According to Oviatt *et al.* (2015), the post-Bonneville sediment of Great Salt Lake consists of Early Holocene non-faecal-pellet mud with rare faecal pellets and abundant *Artemia* cysts overlain by Holocene faecal-pellet-rich mud with dominant *Artemia* faecal pellets and rare cysts. Those authors suggested a dry climate with low lake levels for the Early Holocene. The abundance of *Artemia* cysts shows that the lake water level was low enough for *Artemia* to thrive in the hypersaline water. Also, according to Maszczyk & Wurtsbaugh (2017), the reduction of algal concentration in the Great Salt Lake water (160 cm high columns) by *Artemia gracilis* grazing is linked with a higher sedimentation rate due to the fast sinking of large faecal pellets. Based on recorded organic-rich muds with faecal pellets and interbedded sodium-sulphate salts Oviatt *et al.* (2015) suggested a shallow lake environment under dry climate conditions in the Holocene for the Great Salt Lake. Moreover, Baltres & Medegšan (1978) studied high-magnesium calcite in *Artemia salina* faecal pellets in the hypersaline Techirghiol Lake (Romania), and assumed that ammonia and sulphide contribute to the precipitation of high-magnesium carbonate in the *Artemia* faecal pellet pore spaces. Additionally, Blasi *et al.* (2020) discovered carbonaceous sludge, organic matter and clay as specific compositional components of *Artemia persimilis* faecal pellets in Chasico Lake (Argentina). Those authors suggested that the absence of *Artemia* species and associated faecal pellets were related to the decrease in lake water salinity and the arrival of predators (such as the pejerrey *Odontesthes bonariensis*) to the lake at about 1220 years cal BP.

In Urmia Lake, Kelts & Shahrabi (1986) found that *Artemia urmiana* faecal pellets are the second most important sedimentary component, and that the pellets were mostly composed of aragonite with minor amounts of clastic minerals such as quartz, calcite, dolomite and clay minerals. Djamali *et al.* (2010) linked the abundance of *Artemia* faecal pellets from different depths of Urmia Lake sediments to previous hydrological conditions of Urmia Lake over the Late Quaternary.

In Urmia Lake sediments, *Artemia* faecal pellets are frequently cream-coloured, multi-coloured spotted, light brown, rarely black and orange in colour (Figs 2 and 3), which indicates variation in their composition. When the organisms consume inorganic and organic particles

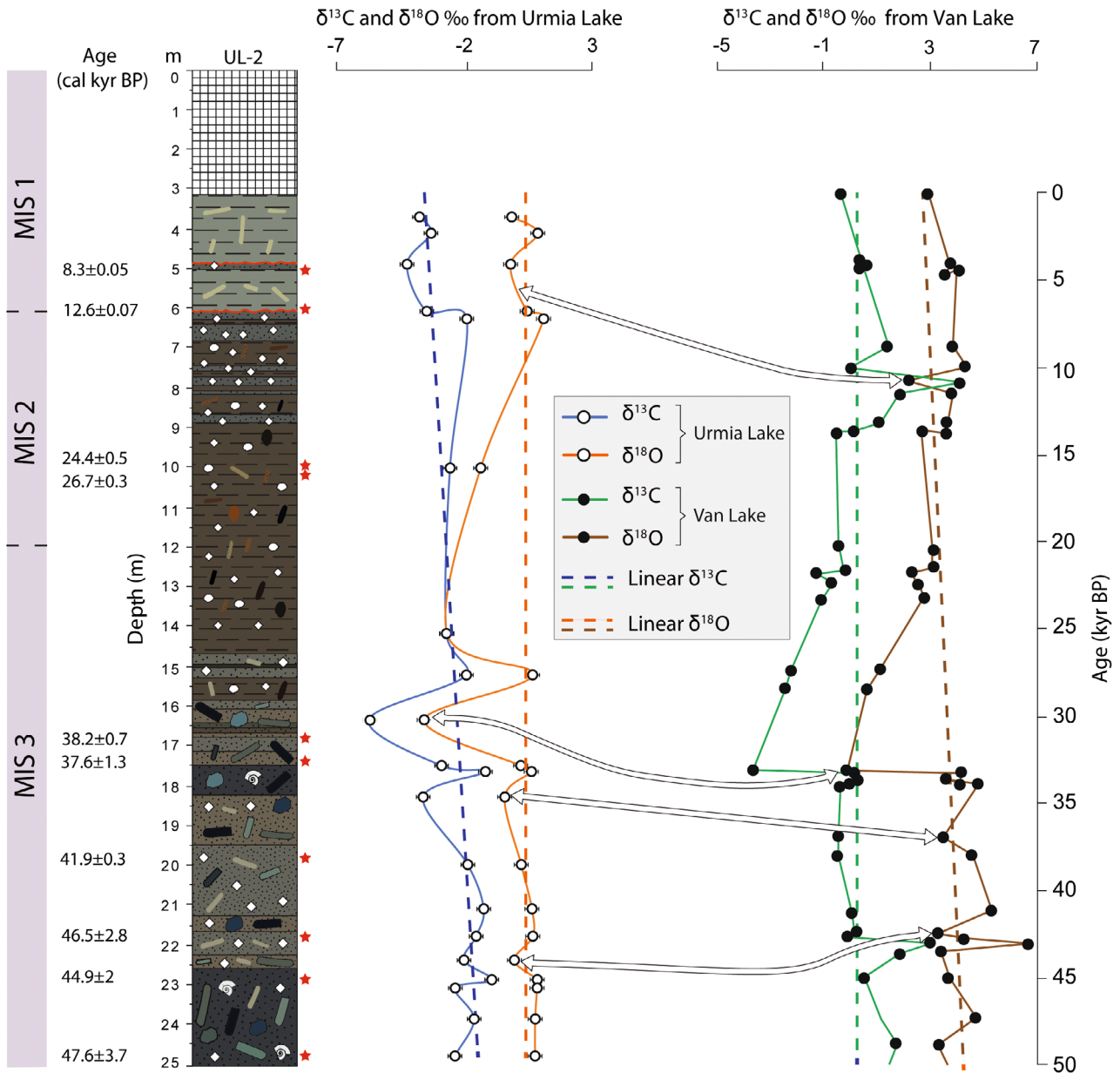


Fig. 6. Oxygen and carbon isotopic composition variation in Urmia Lake sediments (UL-2 core), and comparison with reported data from Van Lake (Çağatay *et al.*, 2014). Both lakes show a very similar trend with about 3700 years of time offset.

suspended in water, the variety of pellet colour and mineralogy might reflect the water chemical and detrital constituents (Reeve, 1963; Stevens *et al.*, 2012). Spotted faecal pellets (Fig. 3B) reveal the proportion of probably detrital material in the *Artemia* diet, whereas cream-coloured pellets (Fig. 3A) show low detrital input and suggest optimal living conditions for *Artemia* (Fig. 8). Thin section images (Fig. 3E and F) showing evenly distributed spots in the faecal

pellet point to carbonate precipitation within the intestinal tract of brine shrimp, which homogenizes the material, as already hypothesized by Eardley (1938). It discards the opposite view that early diagenetic microbial effects (burrowing and boring; Baltres & Medessan, 1978) may be responsible for the Urmia Lake pellets' colour. Faecal pellets can be intact, friable or broken, they can be a core of coated grains, and they also can be enclosed by diagenetic sulphate

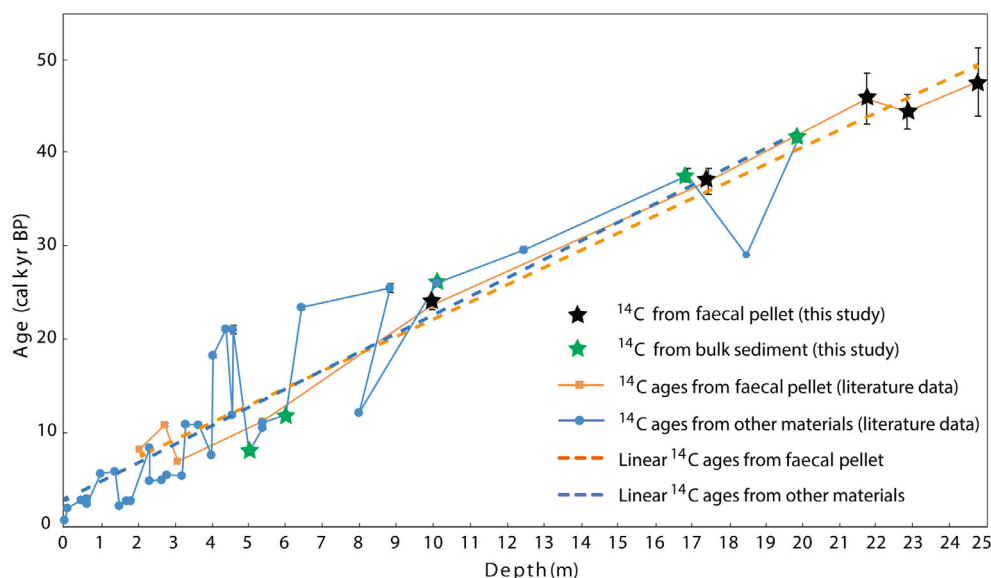


Fig. 7. Comparison of ages (cal kyr BP) and their linear trends obtained from faecal pellets and bulk sediments from the studied core and other material from previous studies (Kelts & Shahrabi, 1986; Djamali *et al.*, 2008; Manaffar *et al.*, 2011; Talebi *et al.*, 2016; Sharifi, 2017; Tudryn *et al.*, 2021; Kong *et al.*, 2022).

mineral cement (Fig. 3D). Reworked and broken faecal pellets (Figs 2 and 3) are linked to agitated near-shore processes as well as river eroding forces on the exposed lake margins during lake regression periods. In Urmia Lake, coated grains dominantly form in the near-shore environment during times of lake regression (Fig. 8) as a result of the interactions of wave action and chemical precipitation of carbonate when moving the particles. The repeated grain rotations in supersaturated water (i.e. rich in dissolved ions such as calcium, carbonate, bicarbonate and sulphate) cause the formation of concentric laminae around the faecal pellets and forms capsule-shaped coated grains (Fig. 3C, E and F; Eardley, 1938; Lowenstam & Epstein, 1957; Morse & Mackenzie, 1990; Sumner & Grotzinger, 1993; Duguid *et al.*, 2010).

Chronology

Faecal pellets, as a biochemical sediment component, previously have been used for dating (applying both ^{14}C and U-Th methods) in hypersaline Urmia Lake due to scarcity of fauna and flora remains (e.g. Kelts & Shahrabi, 1986; Stevens *et al.*, 2012; Sharifi, 2017; Kong *et al.*, 2022; this study). The ages of faecal pellets and bulk sediments from one sediment layer overlap; although bulk sediment ages have lower error bars than

faecal pellet ages (Table S6). This difference most probably is related to a reservoir effect on faecal pellet ages, where *Artemia* absorbs suspended and dissolved carbonates from lake water without filtering (e.g. Eardley, 1938; Illing, 1954; Reeve, 1963). The chronological framework for the studied core using ^{14}C ages from faecal pellets and bulk sediment is compared with previously published radiocarbon ages obtained from Urmia Lake (Table S6; Fig. 7). Previous studies on Urmia Lake obtained ages from various sediment depths and locations in the lake (Table S6; Fig. 1), and they were performed using a variety of materials, including bulk sediment, *Artemia* faecal pellets, *Artemia* cysts, charcoal, organic matter, plant remains and the total carbonate fraction (Djamali *et al.*, 2008; Manaffar *et al.*, 2011; Talebi *et al.*, 2016; Sharifi, 2017; Tudryn *et al.*, 2021; Kong *et al.*, 2022; Table S6). When comparing the depth-to-age distribution of faecal pellets and bulk sediment ages they show a fairly good agreement with one another; the difference amounts to a few hundred years with regard to their respective error bars and their linear trends over the radiocarbon time range. When compared with published ages of organic matter from other studies, those other data have a much higher scatter, especially with ages between 10 and 25 cal kyr BP obtained from sediments between 4 cm and 9 m depth (Fig. 7).

Another point worthy of mention is the location of four reversed ages in the upper part of the core (Table 1; Fig. 8), where the lake presumably experienced sediment exposure (Facies 2) and where the sediment record has two noticeable unconformities (Facies 3 and 4; Fig. 2). Presumably, these four samples have material that can be explained with sediment reworking on the exposed parts of the lake during times of lower lake levels (Fig. 8). The variable radiocarbon reservoir effect could be another explanation for these four reversed ages.

Sedimentation history and lake level fluctuations

According to lithological, mineralogical and biogenic contents of the studied core, the sedimentation history of the lake is evaluated in response to lake level fluctuations that are examined using four different periods during three Marine Isotope Stages (MIS 3, MIS 2 and MIS 1).

Marine Isotope Stage 3 (48 to 29 kyr BP)

At the core site, deposition during most of MIS 3 is represented by period 1 that is interpreted as a shallow lake, implying a relative lake-level lowering (Fig. 8). This is reflected in the relatively high abundance of coarse-grained sand, including coated grain fractions and reworked shell fragments (Fig. S5A). It is noteworthy that faecal pellets can be found scattered throughout the period 1 sequence (Facies 1), and they frequently form cores of elongated capsule-shaped coated grains (Fig. 2A). The limited amount of individual faecal pellets tend to be cream in colour, while the broken and older reworked pellets tend to have hard and shiny surfaces, which are grey, black and dark blue in colour (Fig. 2A). The relatively low lake water level is linked with the transport of dominantly broken shell fragments from marginal water swamps, lake terraces, and even surrounding foraminifera-bearing formations to the lake. Typical shell fragments seen in this facies include foraminifera, ostracods (Fig. S5A and B), gastropods and bivalves that have been identified from exposed lake terraces by Salehipour Milani *et al.* (2020).

Period 1 shows a relatively low amount of TOC and ROC values, and a slightly higher TIC amount (Table S1) pointing to the dominance of chemical sedimentary processes during MIS 3, which is supported by the abundance of coated

grain components. During this period, the TOC values are lower than the ROC values and, consequently, the TOC/ROC ratio is low (Table S1). Natali *et al.* (2020) suggested that low TOC/ROC ratios associated with less negative $\delta^{13}\text{C}_{\text{TOC}}$ values characterize sediment where its organic matter was affected by a transformation mediated by biological activity. However, from UL-2 only $\delta^{13}\text{C}_{\text{TIC}}$ values are available, which are indeed among the least negative in the record. According to Stevens *et al.* (2012) the amount of organic and inorganic carbon is controlled by variations in sediment and water input to the lake, lake level fluctuations and bio-productivity changes through time. In Urmia Lake, the organic and inorganic carbon amount is strongly linked to the lake water fluctuation (Table S1; Figs 5 and 8). During times of higher lake levels and with optimum ecological conditions for *Artemia*, the produced organic carbon by algae and the supplied dissolved organic and inorganic carbon by rivers are absorbed by *Artemia* and accumulated in faecal pellets. With the water level falling and an increasing lake water salinity, the ecological conditions become unfavourable for *Artemia*, while the halophyte algae experience maximum bloom. During this period, organic and inorganic carbon contribute to the formation of coated grains. With continuous water level fall, the amount of transported allochthonous organic matter (for example, charcoal, wood and leaves) by rivers becomes dominant and the autochthonous organic matter becomes minimal due to the extreme lake water salinity.

The relatively low variations of $\delta^{18}\text{O}$ and $\delta^{13}\text{C}$ isotope values (except for a sample with dominant coated grain from depth 16.31 m) in MIS 3 suggest an almost stable low lake water level during the MIS 3 period (Figs 6 and 8). A similar sharp change is recorded in the Lake Van isotope record in MIS 3 around 33 655 cal yr BP (Çağatay *et al.*, 2014; Fig. 6). Those authors assumed that the change was related to a rapid transgression and was most likely caused by meltwater supply or moisture-bearing storm tracks from the northern Atlantic with low $\delta^{18}\text{O}$ and $\delta^{13}\text{C}$ values. Similarly, in Urmia Lake the recorded abrupt change a little before 38.2 cal kyr BP (depth of 16.31 m) was most probably affected by the high amount of water and sediment supply (Fig. 5) input to the lake in a short period. At this depth, the faecal pellets show the highest amount of ROC and detrital component (Fig. 5).

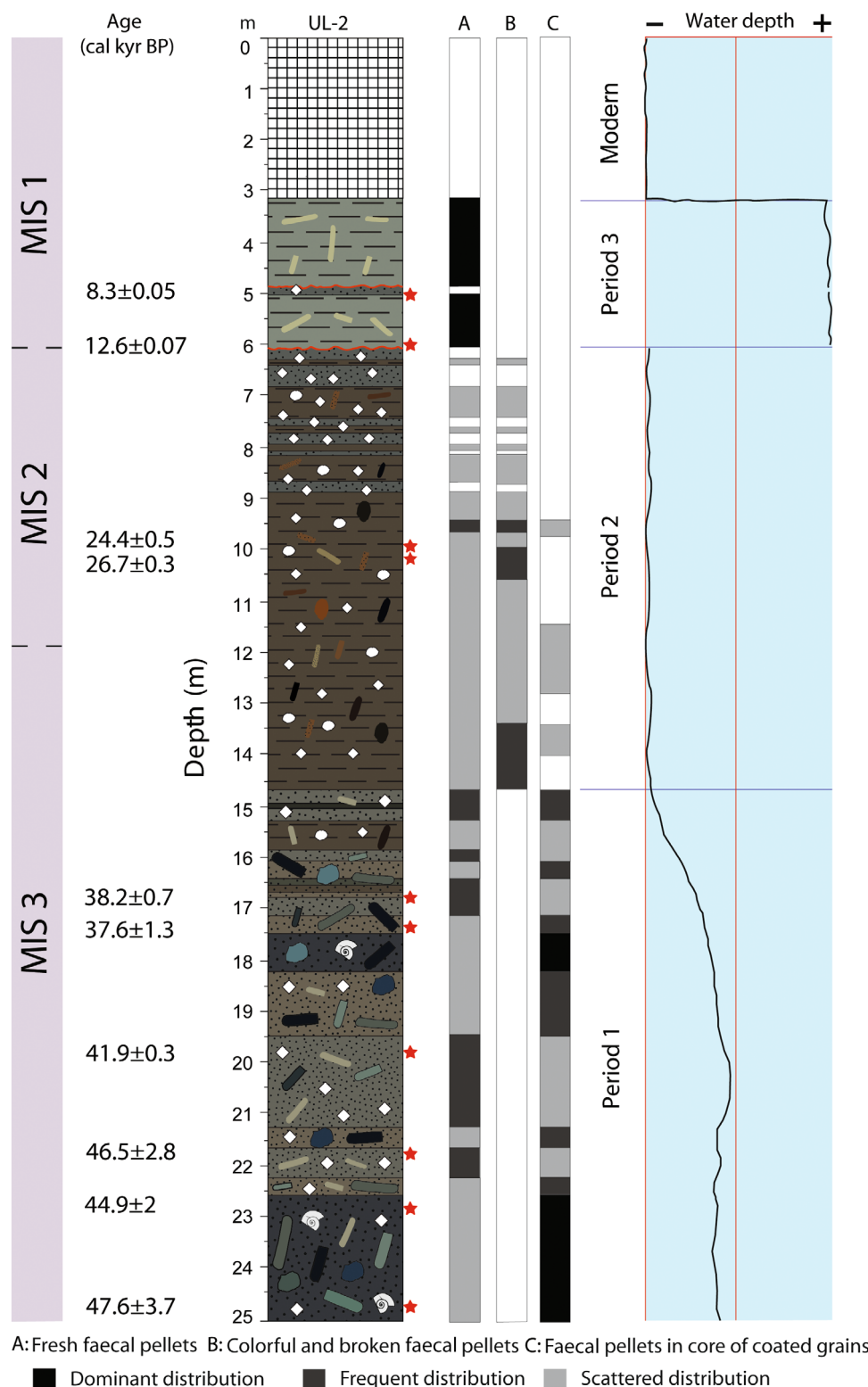


Fig. 8. Relative abundance and different forms of faecal pellets including: fresh and cream-coloured (A); colourful and broken (B); and core of coated grains (C) in the studied core. Interpreted relative Urmia Lake water level fluctuations (black line) according to the identified four main periods of the core.

Overall, the $\delta^{18}\text{O}$ and $\delta^{13}\text{C}$ values show a positive covariance (0.6177) along the UL-2 core. Elsewhere co-varying isotopes have been interpreted to reflect the precipitation versus evaporation balance in closed lakes (e.g. Kelts & Talbot, 1990; Li & Ku, 1997; Leng & Marshall, 2004; Roberts *et al.*, 2008; Çağatay *et al.*, 2014). The range of $\delta^{18}\text{O}$ in faecal pellets (mean value: 0.2; Table S2; Fig. 6) in MIS 3 is almost similar to the range previously reported by Stevens *et al.* (2012) for the period of the Last Glacial Maximum. In contrast, $\delta^{13}\text{C}$ values are more negative (mean value: -2.5 ; Table S2; Fig. 6) than the counterpart values (mean value: 1.5) from Stevens *et al.* (2012). Most probably different analytical methods between the two studies is the reason for this difference. For example, in our study faecal pellet samples have been heated to over 600°C for 4 h to destroy any organic carbon compounds prior to carbon isotope measurements. Such a step is not reported by Stevens *et al.* (2012). Stevens *et al.* (2012) also stated that arid conditions at Urmia Lake began at 60 kyr BP.

In MIS 3, three relatively stable isotope minima ($\delta^{18}\text{O}$ and $\delta^{13}\text{C}$) are obvious; at 22.35 m core depth (*ca* 45 cal kyr BP), at 18.25 m core depth (shortly before *ca* 37 cal kyr BP) and at 16.31 m core depth (shortly after *ca* 38 cal kyr BP; Fig. 6). This would mean that at times of prevailing arid conditions with high evaporation, the UL-2 core record has captured three short events of higher precipitation or fluvial input into the lake during MIS 3, the last of which was stronger than the others. Strikingly, this also has been observed at the well-dated and much deeper Lake Van, which is located 150 km west of Urmia Lake (Çağatay *et al.*, 2014). Both lakes show very similar stable isotope trends as shown in Fig. 6; arrowed lines in this figure connect similar successions of relative maxima and minima. Given the maximum error bar with dating bulk material of this period's sediments (up to ± 3745 years; Table 1) this suggests that the two lakes might have captured the same events (Fig. 6). The other reason for the age offset of the same event between these two lakes might be related to the altitude difference; the mean elevation of Urmia Lake is 1270 m.a.s.l., whereas the Lake Van mean elevation is 1646 m.a.s.l. (Litt *et al.*, 2014; Mohammadi *et al.*, 2023).

Marine Isotope Stage 2 (29 to 12 kyr BP)

The presence of dark brownish muddy-salty deposits (Facies 2) is associated with a relative

lake level lowstand in the middle part of period 2, which may include partial exposure of the lake floor in early MIS 2 (Fig. 8). Coarse-grained volcanic lithics, volcanic glass and detritic grains are common in Facies 2. The XRD analysis of the multicoloured faecal pellets reveals that the dominantly micritic pellets have clastic material admixed; they contain different portions of quartz, calcite, volcanic lithics, volcanic glass and clay minerals (Table S5; Fig. 2B). It may reflect the riverine sediment input on the *Artemia* diet at times of lower lake levels during early MIS 2. This could be a local feature of faecal pellets in Urmia Lake, because the nearest river (Zola Chay) drains Yigit Dagi volcano (on the border of Turkey and Iran) and the detrital fraction might be supplied from the volcanic slopes (Allen *et al.*, 2011). In the lower part of period 2 (Facies 2), faecal pellets are generally broken, which points to recycling of deposited pellets from the exposed lake floor by erosion and transport by floodwaters and rivers. During early MIS 2, physical processes dominate and the fluvial input delivers a greater portion of terrigenous sediment to the core site (Fig. 2B). In addition, the presence of rounded diagenetic sulphate minerals suggests that more ancient large sulphate minerals have been eroded and recycled (Fig. 2B) under similar conditions as stated above.

Similarly, the late MIS 2 (upper part of period 2), is associated with a lake lowstand which may include partial exposure of the lake floor (Fig. 8). The main difference between Facies 3 and Facies 2 is the presence of a sizeable portion of large euhedral diagenetic sulphate minerals in Facies 3 compared to rounded and small sulphate minerals in Facies 2. This is most probably related to the faecal pellet-rich overlaying layer, where the pressure-dissolution of faecal pellets in the early stage of diagenesis provides Ca^{+2} cations to the underlying SO_4^{-2} anion-rich deposits for the formation of large sulphate minerals.

It is noteworthy that, for Facies 2 and 3, a partial lake shore exposure is assumed, which means a playa-lake condition was prevailing during MIS 2, as already described by Kelts & Shahrabi (1986). Although faecal pellets can be observed at the relevant stratigraphic position, the sedimentary environment changes notably on a decimetre-scale in the sediment record (Fig. S3).

Stevens *et al.* (2012) stated that arid conditions in Urmia Lake began at 60 kyr BP and lasted until the Holocene, which is consistent with our

inferred lake level lowering (Fig. 8). However, Kong *et al.* (2022) reconstructed two high lake level periods at 29.9 to 20.2 cal kyr and during the Bölling/Alleröd warming period (15.3 to 13.3 cal kyr); they observed particularly prominent carbonate deposition during these times, as seen in sediment cores from the south-western part of Urmia Lake. However, in Urmia Lake, variation in carbonate amount is not a good indicator for water level fluctuation. For example, aragonite is deposited in the highstand periods in the form of faecal pellets and in lowstand periods in the form of coated grains. The aforementioned high lake levels at 29.9 to 20.2 cal kyr and 15.3 to 13.3 cal kyr were not observed in the studied core, where during these age periods the lake floor was partially exposed (Fig. 8).

The Younger Dryas (*ca* 12.9 to 11.6 kyr), which is interpreted as a period of extreme aridity with steppe vegetation according to Djamali *et al.* (2008), is observed in core UL-2 with a sharp unconformity (hiatus) exactly before 12.6 cal kyr. This unconformity occurs at the top of Facies 3 at 6.07 m depth and is overlain by fresh faecal pellet-rich greenish mud (Figs 8 and S3). The recorded 24 subaerial terraces of Urmia Lake, at various elevations, provide clear evidence of scores of lake level changes in Late Quaternary time (Yamani *et al.*, 2015; Salehipour Milani *et al.*, 2020).

Marine Isotope Stage 1 (12 kyr BP to recent)

Facies 4 starts with an unconformity at the lower contact (6.07 m) and a second unconformity interrupts this facies at 4.90 m (Fig. 8). The second unconformity is most probably related to variations in the lake hydrodynamic conditions. The second unconformity starts with a 0.10 m thick sand layer with abundant diagenetic sulphate minerals (Fig. S4). In this layer, faecal pellets are absent. This unconformity is not recorded in other sediment cores of Urmia Lake (e.g. Kong *et al.*, 2022). The UL-2 core site is located close to the lake margin (Fig. 1) and the unconformities in the sediment record might be caused by local changes in the lake hydrodynamic conditions. The overlying sediments are poor in clastic and diagenetic sulphate minerals, and rich in greenish micrite and fresh cream-coloured faecal pellets (>50% of bulk sediment) thus pointing to favourable ecological conditions for the growth and bloom of *Artemia* (Fig. S4). A relatively high lake level is inferred from this for much of the Holocene (Fig. 8).

The TIC values are comparably high in Facies 4 (Fig. 5) because increased lake water level

would promote ideal conditions for *Artemia* habitat and the production of more aragonitic faecal pellets. Djamali *et al.* (2008) showed that a large amount of aragonitic faecal pellets go parallel with high values of arboreal pollen/non-arboreal pollen (AP/NAP) counts, thereby confirming a more humid climate around the lake during MIS 1. During the Holocene (period 3), more humid conditions and higher lake water levels are reflected by the faecal pellet enrichment and relatively low $\delta^{18}\text{O}$ and $\delta^{13}\text{C}$ values (Figs 6 and 8; Haghipour *et al.*, 2016).

Modern

Facies 5 depicts a lake-level lowering over the last 25 years (Mohammadi *et al.*, 2021, 2023). The presence of a maximum of 3.20 m of pure evaporite deposits (>97% halite) covering the lake floor demonstrates that the lake undergoes a drastic change from a terrigenous-biochemical hypersaline lake to a chemical playa environment (Mohammadi *et al.*, 2019, 2021).

Urmia Lake water level fluctuation

Despite some studies on the Late Pleistocene–Holocene lake level fluctuations in Urmia Lake (Djamali *et al.*, 2008; Stevens *et al.*, 2012; Kong *et al.*, 2022), the discussion continues. Stevens *et al.* (2012) reconstructed climate conditions and relative lake water levels for the last 185 kyr based on sedimentological characteristics and isotopic data ($\delta^{18}\text{O}$ and $\delta^{13}\text{C}$) from *Artemia* faecal pellets. The same 100 m long core from the central part of Urmia Lake was analysed for the vegetation history covering the last 200 kyr BP (Djamali *et al.*, 2008). In a recent study, Kong *et al.* (2022) used a 12.5 m core from the western margin of the lake to infer palaeoclimate and palaeoenvironmental conditions, including relative lake water levels for the last 30 kyr; that study focused on mineralogical and sedimentological characteristics of lake sediments.

Stevens *et al.* (2012) interpreted that arid conditions and low lake levels prevailed after 60 kyr and persisted until 14 kyr BP. However, several episodes with increased moisture and rising relative lake water levels were recorded during the Last Glacial. The relative lake level lowered after 14 kyr BP when the Younger Dryas is expressed as an episode with increased aridity and a relatively low lake level at Urmia Lake (Kong *et al.*, 2022). Finally, more humid conditions dominated over the last 10 kyr (i.e.

Holocene) which is expressed in an increasing relative lake level. Unlike Stevens *et al.* (2012), according to Kong *et al.* (2022), the Holocene lake does not reach the relatively high MIS 3 levels.

Faecal pellet data including O and C isotopic ratios from the UL-2 record suggest a lowering of the lake water level and dry climate conditions over MIS 3 and MIS 2, and a more humid climate with a higher lake level in MIS 1 (Holocene; Fig. 8). A similar succession of dry and humid climatic episodes and associated lake level variation was observed by Stevens *et al.* (2012). In contrast to Stevens *et al.* (2012) and the UL-2 faecal pellet record (this study), Kong *et al.* (2022) disagreed with dry climate conditions and relatively low lake water levels during late MIS 3 and MIS 2. This discrepancy most probably is related to the different coring locations. The sediment cores used by Stevens *et al.* (2012) and the UL-2 site are located in the central and deep part of the north-western lake where sedimentary conditions are dominantly lacustrine (Fig. 1). On the contrary, the cores used by Kong *et al.* (2022) were collected from the exposed margin of the lake close to Shahar Chay River delta (Fig. 1); most probably the lateral migration of the delta influenced the core site. The amount of detritus within the sediment column, as reported by Kong *et al.* (2022), is much higher than the reported detrital material by Stevens *et al.* (2012) and in the UL-2 record, which in turn indicates that the delta environment had an impact on the sedimentation pattern in the core site of Kong *et al.* (2022). The other reason for the aforementioned difference is the application of different materials for palaeoclimate conditions and lake water level reconstruction. For example, in this study (UL-2 core) and Stevens *et al.* (2012) the frequency, mineralogy and $\delta^{18}\text{O}$ composition of faecal pellets is applied as a reliable proxy for palaeoclimate conditions and lake water level reconstruction, while Kong *et al.* (2022) used the frequency of bulk aragonite, dolomite and clay minerals crystallinity index as lake water level indicators. It is noteworthy that the bulk aragonite amount is not a good indicator, at least in Urmia Lake, for past lake level reconstruction because in Urmia Lake aragonite is deposited during both high lake levels (in the form of faecal pellets) and low lake levels (in the form of the cortex of coated grains). Similarly, Kong *et al.* (2022) assumed the majority of dolomite to precipitate from the lake water as primary (authigenic) dolomite under dry climate conditions, and high evaporation and a relatively low lake level. However, primary

dolomite precipitation from lake water has not been reported. Interestingly, under present-day conditions and rapid lake level decrease a thick salt crust has formed (>3 m) with dolomite being absent, as documented in extensive XRD data of the salt crust depth profile (Davari, 2014). The high amount of Mg^{2+} cations (1012 meq/l; Lak *et al.*, 2022) in the lake brine is in agreement with no deposition of primary dolomite from lake water with very low lake levels at the same time. Nevertheless, diagenetic (secondary) dolomite is reported from lake sediments by Kelts & Shahrabi (1986) and Darvishi Khatouni *et al.* (2015). Similarly, a principal component analysis (PCA) from bulk XRD of core UL-2 illustrates a positive relation between diagenetic gypsum and dolomite, which further supports the interpretation that both gypsum and dolomite have an early diagenesis origin in Urmia Lake (Schwamborn *et al.* submitted). Therefore, the confusion of identifying diagenetic dolomite falsely to be primary dolomite (Kong *et al.*, 2022) could be another reason for the difference in reconstructing the lake water level and palaeoclimate implications for the late MIS 3 and MIS 2 (Stevens *et al.*, 2012; this study).

Urmia Lake late Pleistocene–Holocene palaeoclimate records and global climate changes

Despite the low temporal resolution of the Urmia Lake isotopic record, three hemisphere-scale palaeoclimate events in the Late Pleistocene and Holocene are recognizable. There are two minimum events in the early part of MIS 3; around 46 kyr and 34 kyr BP, and a minimum event at the end of MIS 2 coinciding with the Younger Dryas. Other long palaeoclimate time series back to MIS 3 including oxygen isotopic records are known from Middle Eastern lakes and caves; namely from the Dead Sea and the Soreq Cave in Israel and Lake Van in Turkey (Fig. 9). In Urmia Lake, the two MIS 3 events coincide with a similar palaeoclimate double event in the Lake Van sediment record. According to Çağatay *et al.* (2014), Lake Van experienced a lower lake level (relative to the present day) during 60 to 34 kyr BP. However, this regressive period is interrupted by transgressive episodes around 46 kyr and 35 kyr BP. It has been suggested that these transgressive episodes correspond to Dansgaard-Oeschger climatic events involving the discharge of large amounts of melt-water into the lake. Urmia Lake might have reacted in a similar way to Lake Van. However, unknown reservoir effects over time, and age

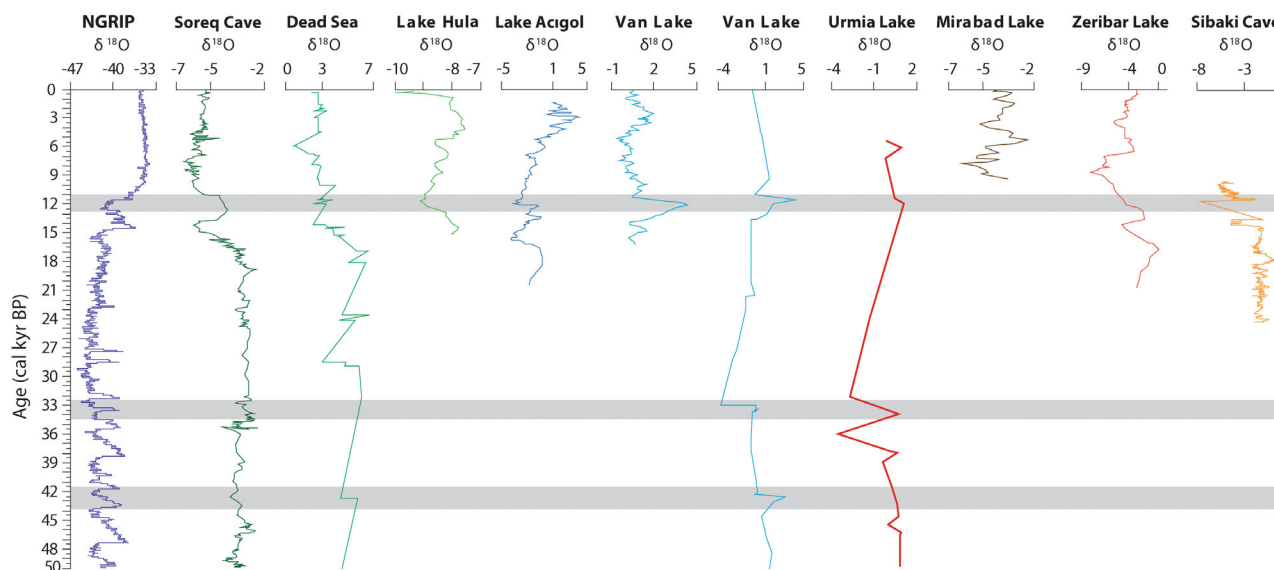


Fig. 9. Correlation of the Urmia Lake oxygen isotopic records with the NGRIP curve – North Greenland Ice Core Project members (2004) – and some lakes and caves of the Middle East in the past 50 kyr BP. Oxygen data were redrawn from Soreq Cave (Bar-Matthews *et al.*, 1999), Dead Sea (Goldstein *et al.*, 2020), Lake Huleh (Stiller & Hutchinson, 1980), Lake Acigöl (Roberts *et al.*, 2001), Lake Van (Wick *et al.*, 2003; Çağatay *et al.*, 2014), Lake Mirabad (Stevens *et al.*, 2006), Lake Zeribar (Stevens *et al.*, 2001) and Sibaki Cave (Soleimani *et al.*, 2023).

uncertainties with the absolute dating of Urmia Lake sediments, do not allow for an unerring palaeoenvironmental interpretation when synchronizing with other records.

Similar to Urmia Lake, the pronounced Younger Dryas to Early Holocene transition has been identified in North Greenland Ice Core (North Greenland Ice Core Project members, 2004), several other lakes (Dead Sea, Lake Huleh, Lake Acigöl, Lake Van, Lake Mirabad, Lake Zeribar and Caspian Sea) and caves (Soreq Cave and Sibaki Cave) in the Middle East (Fig. 9; Stiller & Hutchinson, 1980; Bar-Matthews *et al.*, 1999; Roberts *et al.*, 2001; Stevens *et al.*, 2001, 2006; Wick *et al.*, 2003; Çağatay *et al.*, 2014; Goldstein *et al.*, 2020; Kaveh-Firouz *et al.*, 2023; Soleimani *et al.*, 2023). In Urmia Lake, the Younger Dryas is interpreted as a period of extreme aridity with steppe vegetation (Djamali *et al.*, 2008), which matches the relative maximum in the oxygen isotopic record at the relevant core part (Fig. 9). Yet, care is needed, because the sediment record is interpreted to have an unconformity in the sediment succession (exactly before 12.6 cal kyr); it occurs at the top of a non-faecal-pellet mud and is overlain by faecal pellet-dominant mud (Fig. 8). With regard to the Early Holocene, dry climatic conditions similar to Urmia Lake are also interpreted from the Great Salt Lake record; between 11.5 kyr and 10 kyr by mud poor in *Artemia* faecal pellets and rich in

cysts deposited before it is overlain by mud rich in faecal *Artemia* pellets and poor in cysts (Oviatt *et al.*, 2015).

Significance of *Artemia* faecal pellets in palaeoenvironmental studies

Although hypersaline lakes are distributed over large geographical areas, and although they are sensitive recorders of climate and environmental conditions, they are rarely the subject of palaeoclimate and palaeoenvironment studies due to their low carbon productivity and their limited sediment constituents suitable for chronology (e.g. Williams & Liebhold, 2002; Jones & Deocampo, 2003; Oviatt *et al.*, 2005, 2015, 2021; Oren, 2009; Gajardo & Redón, 2019; Mohammadi *et al.*, 2021, 2023). Except for bulk sediment, pollen and *Artemia* faecal pellets are the only constituents for ^{14}C dating. Pollens provide very important information about palaeoclimate, but due to their allochthonous nature, they do not provide information about palaeolake water salinity and water level fluctuations (e.g. Djamali *et al.*, 2008; Louderback & Rhode, 2009). In contrast, *Artemia* faecal pellets are autochthonous sediment components, and contain information on water salinity, lake level fluctuation and changes in the depositional environment. Brine shrimp *Artemia* has several different species (for example, *urmiana*, *tibetiana*,

persimilis, *monica*, *salina*, *sinica* and *franciscana*) and is widespread in saline and hypersaline lakes and playas all over the world (Van Stappen *et al.*, 2001; Van Stappen, 2002). In times of optimum ecological conditions for *Artemia* to live and bloom, their faecal pellets can form 30 to 50% of total deposited sediments as known from large hypersaline lakes such as the Great Salt Lake and Urmia Lake (e.g. Eardley, 1938; Djamali *et al.*, 2010). Next to organic and inorganic carbon for ^{14}C dating *Artemia* faecal pellets provide suitable material (i.e. carbonate) for carbon and oxygen isotope analysis (Stevens *et al.*, 2012). Furthermore, faecal pellet mineralogy, shape, morphology, colour and relative frequency when compared to the other sediment constituents provide valuable information about lake water physico-chemical parameters. It is worth mentioning that the faecal pellets, by forming coated grain cores, aid in interpreting ancient lake water level fluctuations.

Nowadays, under the stress of human manipulation and rapid climate change, freshwater and brackish water lakes especially in arid and semi-arid areas are rapidly changing to saline and hypersaline lakes (e.g. Mohammadi, 2010; Hampton *et al.*, 2018). This change leads to the spread of *Artemia* in a larger area than before and consequently increases potential faecal pellet application importance in palaeoenvironmental studies. The Aral Sea in Central Asia is a typical example of a brackish water lake that changed to a saline–hypersaline lake in the last century due to anthropogenic reasons. By extreme lake water level fall, the lake divided the Aral Sea into Large and Small Aral lakes (e.g. Boomer *et al.*, 2000; Micklin, 2007). With continuous water level decrease, the Large Aral became hypersaline with specific fauna, and among them, the brine shrimp (*Artemia parthenogenetica*) became dominant in the zooplankton population (Aladin & Plotnikov, 2008). According to Mirabdullayev *et al.* (2004), since 2000, *Artemia* has constituted 99% of the zooplankton biomass of the Aral Sea. As a consequence, *Artemia* faecal pellets are a dominant constituent of the recent biochemical sediment in the Aral Sea.

CONCLUSIONS

A 25 m long sediment core recovered from Urmia Lake provides a record of changing lake levels and deposition conditions back to Marine

Isotope Stage (MIS) 3 (ca 48 cal kyr BP). The relative abundance of *Artemia* faecal pellets, supported by other sediment properties, helps to reconstruct the following four main episodes of the lake history.

1 During MIS 3 a period of a slow lake level lowering is represented by a low amount of brownish faecal pellets, a high amount of coated grains, frequently occurring reworked shell fragments and diagenetic sulphate minerals.

2 During MIS 2 the lake level reaches its lowstand of the record (possibly with times of partial lake floor exposure), which is reflected by black, orange and brown faecal pellets with volcanic lithics and volcanic glasses admixed, and an increasing portion of rounded sulphate minerals. At the end of MIS 2 a transition from a playa to a lake environment is contemporaneous with the Younger Dryas period.

3 At the beginning of MIS 1 the lake level rapidly rises to its highest position in the record. During this time the deposits are rich in fresh cream-coloured pellets and do not contain sulphate minerals.

4 A modern lake level lowstand is represented by evaporite minerals forming a 3 m salt crust on the lake floor. Based on stable isotope ($\delta^{18}\text{O}$ and $\delta^{13}\text{C}$) data obtained from faecal pellet micrite, the Urmia Lake record has captured four isotope minimum events, which are also known from the Lake Van sediment record in Turkey.

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DATA AVAILABILITY STATEMENT

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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Supporting Information

Additional information may be found in the online version of this article:

Figure S1. Detailed lithostratigraphy of the studied core (Facies 1). Black stars indicate the examined samples under binocular microscope. Red stars indicate the analysed stable oxygen isotopes ($\delta^{18}\text{O}$), inorganic carbon isotopes ($\delta^{13}\text{C}$), TOC, ROC and TIC samples. Yellow stars indicate the location of ^{14}C dated faecal pellets and bulk sediment samples. Blue stars indicate thin section samples under polarizing microscope. Green stars indicate analysed samples by XRD. Pink stars indicate analysed samples using SEM-EDS.

Figure S2. Detailed lithostratigraphy of the studied core (Facies 2). Legend is the same as Fig. S1.

Figure S3. Detailed lithostratigraphy of the studied core (Facies 3). Legend is the same as Fig. S1.

Figure S4. Detailed lithostratigraphy of the studied core (Facies 4). Legend is the same as Fig. S1.

Figure S5. Thin section images of faecal pellet shell fragments under polarizing microscope: (A) foraminifera from 23.83 m depth, and (B) a grapestone contains shell fragments, pyroxene and gypsum minerals from 3.88 m depth.

Table S1. Total organic carbon (TOC wt%), residual oxidizable carbon (ROC wt%), total inorganic carbon contents (TIC wt%), calculated organic matter (wt%), carbonate (wt%) and other components (wt%) of faecal pellets in the studied sediment core.

Table S2. $\delta^{18}\text{O}$ and $\delta^{13}\text{C}$ values determined from *Artemia* faecal pellets.

Table S3. Depths of different sediment components studied under polarizing microscope using thin sections.

Table S4. SEM-EDS results from selected sediment components from different depths.

Table S5. XRD analysis results of *Artemia* faecal pellets from different depths. The sample marked by a star is coated grain with a faecal pellet core.

Table S6. List of reported ^{14}C ages (cal yr BP) of different materials from different locations and different depths of Urmia Lake sediments. We have calibrated the reported ^{14}C ages of Djamali *et al.* (2008); Manaffar *et al.* (2011); Talebi *et al.* (2016); Sharifi, (2017); Tudryn *et al.* (2021); and Kong *et al.* (2022).