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Shallow hypersaline lakes as paleoclimate archives: A case study from the Laguna Salada, Málaga province, southern Spain

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ABSTRACT

Although numerous studies concerning the Holocene climate of the southern Iberian Peninsula were accomplished within the last few decades, the climate history of this region is still poorly understood. Various studies deal with a combination of proxies, which are neither easy to compare nor is their connection easy to explain, e.g., due to spatial patterns and time transgression. Within this study, the suitability of the lacustrine sediments from the Laguna Salada (Andalucía region, southern Spain) as a paleoclimate archive is investigated. The lake sediments were evaluated using a multi-proxy approach including sedimentological, mineralogical, geochemical and biological analyses. The sediments reflect the evolution of the lake from pre-Medieval times onwards and Characeae as well as Ostracod analyses give an indication of paleosalinity. Moreover does the geochemical composition provide profound information concerning changes of elemental and mineralogical composition. Nevertheless, a robust, high-resolution chronology could not be achieved owed to the scarcity of material available for radiocarbon dating and contamination problems. Furthermore, poor preservation of pollen restricted the reconstruction of vegetation history, which could have complemented important information concerning climatic changes and human activity.

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1. Introduction

Studies of Holocene climate variability (e.g. Mayewski et al., 2004; Davis and Stevenson, 2007; Anderson et al., 2011; Bard, 2013) have shown that climatic shifts in the Holocene were numerous, though weaker in amplitude compared to the last glacial cycle. Although the Iberian Peninsula has been a focus for paleo-environmental research during the last few decades, the regional character of Holocene climate change and human impact is still poorly understood. Today, the climate of Southern Spain is Mediterranean and known to be highly variable on annual to centennial time scales (Jalut et al., 2009). This is due to its location in the southwestern Mediterranean, which is influenced by the arid

climate of northern Africa and the mid-latitude North Atlantic westerly system (Reed, 1998; Lionello et al., 2006). Strong seasonality is expressed by warm and arid summers alternating with mild and wet winters (Lionello et al., 2006; Martín-Puertas et al., 2010). This is in contrast to the behaviour in former times (e.g. Davis and Stevenson, 2007).

In our study we have chosen a lake as paleoclimate archive, because (i) former studies have shown that lacustrine sediments in Southern Spain can provide high-resolution information of past environments (e.g. Reed et al., 2001; Valero-Garcés et al., 2006; Martín-Puertas et al., 2010; Anderson et al., 2011; Giralt et al., 2011; Martín-Puertas et al., 2011; Moreno et al., 2012; Höbig et al., 2016), (ii) sedimentation rates in lakes are usually higher than those in the ocean (Cohen, 2003) and, (iii) shallow endorheic salt lakes can be very sensitive to climate changes (Laird et al., 1996; Beklioglu et al., 2007; Davis and Stevenson, 2007; Höbig et al., 2016).

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Several investigations on climate archives in southern Spain and adjacent areas were performed (Reed et al., 2001; Frigola et al., 2007; Cortés-Sánchez et al., 2008; Combourieu Nebout et al., 2009; Carrión et al., 2010; Corella et al., 2010; Martín-Puertas et al., 2011; Valero-Garcés and Moreno, 2011; Moreno et al., 2012; Valero-Garcés et al., 2014). Nevertheless, climate response thresholds of the wide range of proxies differ temporally and spatially and problems of chronology and/or discontinuous data occur (Cohen, 2003; Magri et al., 2004; Verschuren et al., 2004; Anderson et al., 2012; Fritz and Anderson, 2013). Additionally, most Spanish salt lakes are located within marine or continental evaporites yielding a high alkalinity. This results in a tendency for lacustrine remains to suffer from reservoir effects (Davis and Stevenson, 2007), making radiocarbon dating of non-terrestrial organic matter difficult. Furthermore, many lakes are dominated by groundwater inflow, so that groundwater residence time, activity of sulphate reducing bacteria and reworking of sediments likely influence the datable material (Höbig et al., 2016).

Our motivation is to focus on a contribution to the understanding of the climatic and environmental history of southern Spain during Holocene times and focuses on multi-proxy analyses of the Laguna Salada, a shallow, hypersaline lake in the Andalucía region. This study area with its numerous shallow and endorheic lakes is and has been focus for palaeolimnological research. It was furthermore chosen because of its size, catchment and location, its prospective depth (and consequently lower probability of drying out) and low anthropogenic overprint.

2. Study site

The Laguna Salada (37°02'15"N, 4°50'33"W, 452 m asl) is a small, shallow endorheic salt lake (De Vicente et al., 2012) in Spain,

located in the municipality of Antequera in the region of Andalucía. It has an area of 0.18 km², an approximately three times bigger watershed, a pH value of 8.5 and an average depth of 1.3 m (Rodríguez-Rodríguez et al., 2006). The Laguna Salada constitutes one of the “Campillos” playa lakes, which also comprise the Laguna Dulce, Redonda, del Cerero and de Camuñas next to the city of Campillos (Fig. 1). It is part of the Natural Reserve “Laguna de la Fuente de Piedra”, named after its biggest salt lake, which deemed unsuitable for lake sediment studies e.g. due to its shallowness and anthropogenic overprint. The seasonal to perennial salt lakes are characteristic of arid and semiarid environments (Cohen, 2003) such as southern Spain, where they occur due to tectonic activity and subsidence of underlying rocks (Rodríguez-Rodríguez et al., 2006). The “Campillos” playa lakes lie within a Neogene intra-montane basin in the Betic Cordilleras, evolved due to late orogenic tectonic extension (Linares and Rendón, 1998). The Betic Cordilleras, representing the westernmost part of the Alpine orogeny system and northern branch of the Gibraltar Arc (Reicherter, 2001), were formed from Paleogene to Pliocene times (Gibbons and Moreno, 2002) within the African-European convergence zone (Reicherter and Peters, 2005). During Late Triassic and Late Jurassic times, the largest sedimentation of shallow-marine evaporites took place (Gutiérrez et al., 2008) in the marginal framework of the Tethys. Continental evaporites were deposited in lakes during the Paleogene and Neogene as a result of the dissolution and reprecipitation of older evaporite sequences (Gutiérrez et al., 2008). These basins were filled up with late Miocene materials whereupon conditions changed from marine to continental in Tortonian/Messinian times (Kohfahl et al., 2008).

The abundant salt lakes of the Antequera region are related to dissolution through erosion (Comin and Alonso, 1988; Gutiérrez et al., 2008) and subsidence of the underlying Triassic evaporites

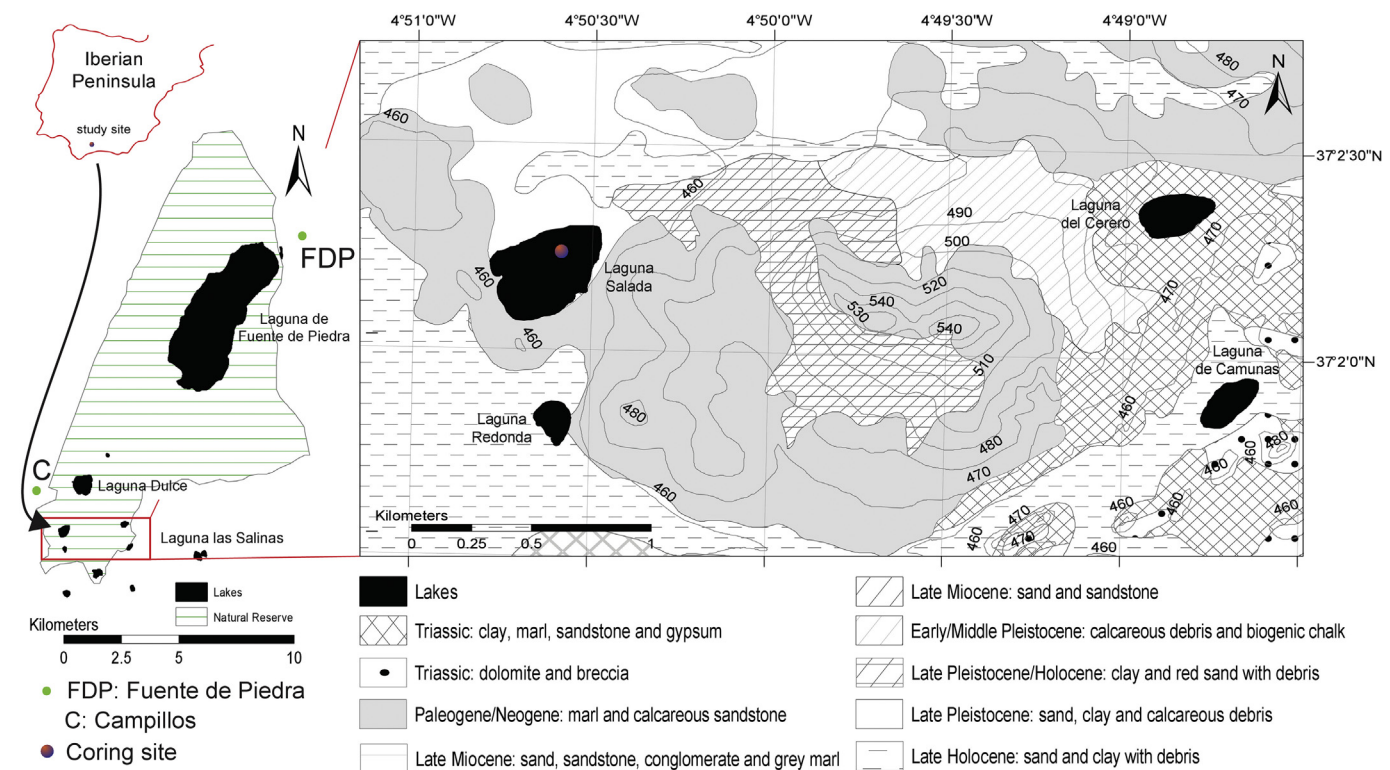


Fig. 1. Top left: Overview map of the Laguna Salada, left: Laguna Salada and the setting within the natural reserve and surrounding lakes, included are the cities Campillos (C) and Fuente de Piedra (FDP), right: Geological setting, coring site as well as the locations of the Laguna del Cerero, Redonda and de Camuñas (based on geological map 1:50 000, (IGME, 1982)).

at or beneath the surface (Kohfahl et al., 2008; Reed, 1998). Therefore, most Spanish salt lakes are rich in sodium-chloride (Na-(Mg)-Cl-(SO₄), typical for marine evaporites) or magnesium sulphate (Mg-(Na)-SO₄-(Cl), continental evaporites) (Baltanás et al., 1990; Comin and Alonso, 1988). The Laguna Salada is underlain by a sequence of Triassic sediments called the “Trias of Antequera” (TA) (Alcalá-García et al., 2001), a Triassic megabreccia partly composed of gypsum at the surface and halite and calcium sulphates at greater depth, resedimented during the Miocene. The TA is mainly composed of reddish-green clays, basaltic volcanic rocks (ophites), sandstones and evaporites (Linares and Rendón, 1998; Kohfahl et al., 2008).

The playa lakes are situated around small hills, which are built up of permeable materials dominated by sand and calcareous sandstones of Paleogene, Neogene and Quaternary age (IGME, 1982). This results in a dome shaped water-table that discharges into the surrounding areas. Salts and other compounds are delivered to the lake via ground- or surface water from the nearby uplands (Rodríguez-Rodríguez et al., 2006). They precipitate as lake sediments, when the evaporitic concentration of the brine is high enough (Eugster, 1980). The endorheic basins are characterized by evaporation, which presupposes again an arid to semiarid climate (Eugster, 1980). As the Mediterranean climate is determined by a long and intense drought during summer (Magri et al., 2004), the salt lakes of Campillos usually desiccate within that season (Rodríguez-Rodríguez et al., 2006). Most of the Spanish salt lakes are temporary and below 1 m water depth (Comin and Alonso, 1988). The annual inflow and the exceeding moisture loss through evaporation result in lake-level variations of the hydrologically closed systems and furthermore lead to a variability in the amount of soluble particles (Battarbee, 2000; Rodó et al., 2002) as lakes are dynamic systems which react very sensitive to variations of climatic or human influence (Rodríguez-Rodríguez et al., 2006).

3. Material and methods

The core SAL 2 was taken from a floating platform by the use of a Russian corer in the north-eastern part of lake Laguna Salada in autumn 2012 (Fig. 1). The core composite covers the upper 2 m of the sediment record, consisting of three drives of 1 m sections with an overlap of 0.5 m. The core sections were opened, photographed and described lithologically in the laboratory of the Institute of Neotectonics and Natural Hazards of the RWTH Aachen University with focus on grainsize, colour, layer boundaries and gypsum content. This was followed by high-resolution X-ray Fluorescence (XRF) scanning of the cores, subsampling and analyses of discrete samples for pollen, characeae, ostracods, macro fossils, organic matter, CNS, TIC/TOC and mineralogy. Smear slides for microscopic observation of sediment composition and texture were taken at centimetre intervals throughout the sequence.

3.1. Geochemical investigations

Continuous down-core XRF measurements of the geochemical composition were carried out with an ITRAX core scanner (Cox Analytical Systems Sweden; Cr-tube, tube voltage 30 kV, current setting 30 mA, exposure time 10 s and spatial distribution 5 mm) at the University of Cologne. This reflects elements of the upper millimetres (Löwemark et al., 2011). In addition, XRF measurements were performed on dried and homogenized samples for comparison with the down-core measurements. The biogeochemical indicators total carbon (TC), nitrogen (TN) and sulphur (TS) contents were measured with a Vario Micro Cube (Elementar Corp.) in which 5 mg of sediment were combusted at 1200 °C. The total inorganic

(TIC) and organic carbon (TOC) contents were measured on parallel samples of 35 mg using a Dimatoc 2000 (Dimatec Corp.) with 10 mg distilled water.

3.2. Mineralogical investigations

XRD analysis on one samples was done at the IGME in Madrid, using a XPERT PRO MPD of PANalytical (40 mA, 40 kV, software HighScore version 3.0.4 by PANalytical). Supplemental XRD analysis on 10 samples was performed for verification and significance at the University of Cologne on powder pellets using the diffractometer Bruker D8 Discover with Cu tubes ($\lambda = 1.5418 \text{ \AA}$, 40 kV, 30 mA) and the detector LYNXE_XE. The spectrum from 5° to 90° was measured in 4155 steps of 1 s exposure time. The evaluation of the minerals was computed using Match! and X-POW. The mineral concentration was evaluated using Rietveld.

3.3. Biological investigations

Pollen samples were prepared following a standard procedure on a sample volume of around 2 ml, employing 10% HCl, 10% KOH, 40% HF digestion, acetolysis (acetic anhydride and sulphuric acid, 9:1), ultrasonic treatment, the addition of *Lycopodium* spores and the final preparation of slides with glycerine. Additional pre-treatment because of low pollen concentration comparable to those used by Davis and Stevenson (2007) was not performed, as most of the minerogenic material could be removed already by the applied treatment. Samples were taken at prospective parts of the core with apparently higher organic matter content at 0.19–0.20, 0.39–0.40, 0.59–0.60, 0.60–0.62, 0.79–0.80, 1.19–1.20, 1.39–1.40, 1.55–1.56, 1.59–1.60, 1.79–1.80 and 1.99–2.00 m depth.

Stonewort (Charophyceae, family of characeae) and ostracod sample preparation followed the instructions of Vangerow (1981): addition of 7% hydrogen peroxide for 24 h followed by wet levigation using two meshes of 0.125 mm and 0.63 mm mesh size and drying at 40 °C. The ostracods were picked and identified under a stereomicroscope (magnification 64-fold). The taxonomy and ecological interpretation followed the studies by (Meisch, 2000; Mezquita et al., 2005; Rasouli et al., 2016). Characeae samples were additionally treated with 20% acetic acid to remove calcium carbonate remains and sonicated. The oospores of the Characeae were determined after Vedder (2004), measurements were done according to Horn af Ratzien (1956, 1959) and Soulié-Marsche and García (2015) at the University of Rostock.

3.4. Chronology

Radiocarbon age measurements were performed on concentrated pollen remains (treatment following preparation of pollen samples without the addition of *Lycopodium* and glycerine) at the Center for Accelerator Mass Spectrometry at the University of Cologne (CologneAMS). OxCal 4.2 was used for calibration (Ramsey, 2009) with the IntCal 13 curve (Reimer et al., 2013). Age determination on bulk sediment was not performed because of potential hard water effects (high carbonate carbon concentration) (Meyers and Lallier-Vergès, 1999).

In addition, nine ostracod samples were used for amino acid racemization (AAR) from cleaned and translucent shells of *Eucypris mareotica* at the Laboratio de Estratigrafía Biomolecular at Madrid, Spain. For this purpose, ostracod samples were carefully sonicated and cleaned. The concentration of amino acids was measured using High-Performance Liquid Chromatography (HPLC), following sample preparation according to Kaufman and Manley (1998) and Kaufman (2000). Age measurements were made with an Agilent

HPLC-1100 (fluorescence detector, 335 nm excitation and wavelength 445). The mean aspartic acid (Asp) and glutamic acid D/L racemization ratios on ostracod shells were used for the calculation of the numerical ages. Thus, Asp D/L values were introduced in the age calculation algorithm of Ortiz et al. (2015). The calculated ages from the ostracod shells constitute the average of the numerical dates of the samples from that particular depth. The age uncertainty is one standard deviate of all the numerical ages calculated from the amino acid D/L values of each sample.

4. Results and interpretation

4.1. Chronology

According to the age estimates and supporting data (Table 1), the 2 m long sediment sequence of the Laguna de Salada probably extends back to pre-Medieval age, but the results do not provide strong chronological control. The age reversals in case of radiocarbon dating may be explained by microbial growth after core recovery, which can add modern atmospheric carbon to the sediment and rejuvenate the radiocarbon (Colman et al., 1996). Younger ages can also be generated if younger sediments are transported to deeper parts of the lake sediments in consequence of bioturbation e.g. through penetration of roots or organisms (Walker, 2005). Older ages can be explained by the reworking of older sediments and by aquatic plants that incorporate old carbon during photosynthesis, resulting in a reservoir effect (Cohen, 2003; Walker, 2005). Pollen are mainly of terrestrial origin and therefore not influenced by this reservoir effect. However, pollen dating might be defective because prepared samples can still contain other organic material apart from pollen (Cohen, 2003). Samples no. 5, 8, 10, 11 and 12 (AAR) had to be rejected for age calculation because of the cut-off value of 0.8 in the concentration of L-Ser to that of L-aspartic acid (Kaufman, 2006), as Ser decomposes rapidly and excessive amounts of this amino acid indicate contamination of valves by modern amino acids. The ostracod dating could slightly differ in terms of the age calculation algorithm which was initially established for the species *Cyprideis torosa* (Ortiz et al., 2013), which may have a different aspartic acid racemization rate than *Eucypris mareotica*. Sample no. 9 was too small to be dated with the radiocarbon method. Taking the results available, there is a hiatus between sample nos. 6 and 7, which differ by 2100 years despite a small sediment interval of 7 cm (0.93–0.96 to 0.87–0.89 m). The erosion can likely be traced back to repeated or long-lasting lake drying in a rather dry phase, taking into account that the very plain area argues against mass movement and that the shallow lake nowadays still dries out in very dry seasons (Rodríguez-Rodríguez et al., 2006). Sample nos. 4 and 3, 15 cm apart, are not continuous

but both belong to the 14th century. This suggests a higher sedimentation rate compared to other periods documented in the record. The two near-surface samples are not successional in time as well. In summary, no explicit reason can be given for each defective age of the potentially pre-Medieval core sequence.

4.2. Sedimentological results

The lithology of the core SAL2 comprises nine unconsolidated sediment layers mostly consisting of mud, intercalated with clay and sandy mud layers (Fig. 2).

Gypsum in variable amounts, grain sizes and degrees of roundness is incorporated in the whole sediment column (Fig. 3). Gypsum grain size distribution can be a result of coupled sorting and abrasion, with decreasing grain size and progressive roundness at increasing distance, supplying information about source and transport (Jerolmack et al., 2011). Prismatic, angular gypsum grains are indicative of in-situ formation and deposition in a deep water phase, while reworked (more rounded and lenticular) gypsum minerals are very common in salt lakes due to re-deposition from the margin zone to more central parts of the lake during times of low lake level (Schnurrenberger et al., 2003; Valero-Garcés et al., 2014). After Davis and Stevenson (2007), a low lake level and related salinity rises may also cause the precipitation of gypsum minerals. However, in times of high lake level, a redeposition of angular gypsum grains from the lake margin is also possible.

Between 2 m and 1.65 m, the core consists of beige to grey mud, a mixture of clay and silt. Gypsum grains are mostly angular with a small grain sizes of approximately 30 µm at the bottom and up to 300 µm at around 1.70 m depth. This is followed by a 10 cm thick sandy mud sequence of beige colour which includes again small to larger angular to sub-angular gypsum crystals up to 400 µm. Shallower lake phases are indicated after Harrison and Digerfeldt (1993) by increasing grain size. However, continuous sedimentation of smaller sediment particles may be deflected by precipitated prismatic, angular salt and gypsum crystals (Davis and Stevenson, 2007). Up to 0.70 m depth, the sediment consist again of beige grey mud. Roundness of gypsum grains varies from angular to rounded and ranges between 20 µm and 400 µm, therefore not giving distinct information about gypsum transport or lake level. This interval is followed by a 3 cm thick clay layer comprising very angular to angular gypsum grains up to 300 µm in size and six cm sandy mud, again of beige colour and with the same size and type of rounded gypsum grains as before. In between 0.60 m and 0.31 m depth, the core is again made up of mud and incorporates very angular to angular 40 µm–400 µm large gypsum grains. A clay layer is documented up to 0.225 m depth including very angular to angular gypsum, followed by a sandy mud layer up to 0.155 m. The

Table 1
Radiocarbon dates, Asp and Glu D/L values of ostracod shells and corresponding numerical ages of the core SAL2.

Nr	Depth [m]	¹⁴ C age [yrs BP]	¹⁴ C age ± 2 σ [cal yrs BP]	D/L Asp	D/L Glu	Age ± 2 σ [yrs BP]	Material
1	0.25–0.28			0.090	0.026	365	Ostracods
2	0.32–0.35			0.088 ± 0.002	0.027 ± 0.002	300 ± 47	Ostracods
3	0.47–0.50			0.097	0.024	584	Ostracods
4	0.62–0.65	488 ± 34	562 ± 57				Pollen
5	0.73–0.75			0.120	0.031	(**)	Ostracods
6	0.87–0.89	640 ± 34	610 ± 58				Pollen
7	0.93–0.96			0.134	0.033	2710	Ostracods
8	1.31–1.35			0.099 ± 0.005	0.028 ± 0.002	(**)	Ostracods
9	1.38–1.41	(*)	(*)				Pollen
10	1.51–1.54			0.083 ± 0.001	0.030 ± 0.000	(**)	Ostracods
11	1.75–1.79			0.157 ± 0.016	0.051 ± 0.005	(**)	Ostracods
12	1.85–1.90			0.1453 ± 0.020	0.039 ± 0.001	(**)	Ostracods

(*) not measured, too small (**) L-Ser/L-Asp > 0.8, not measured.

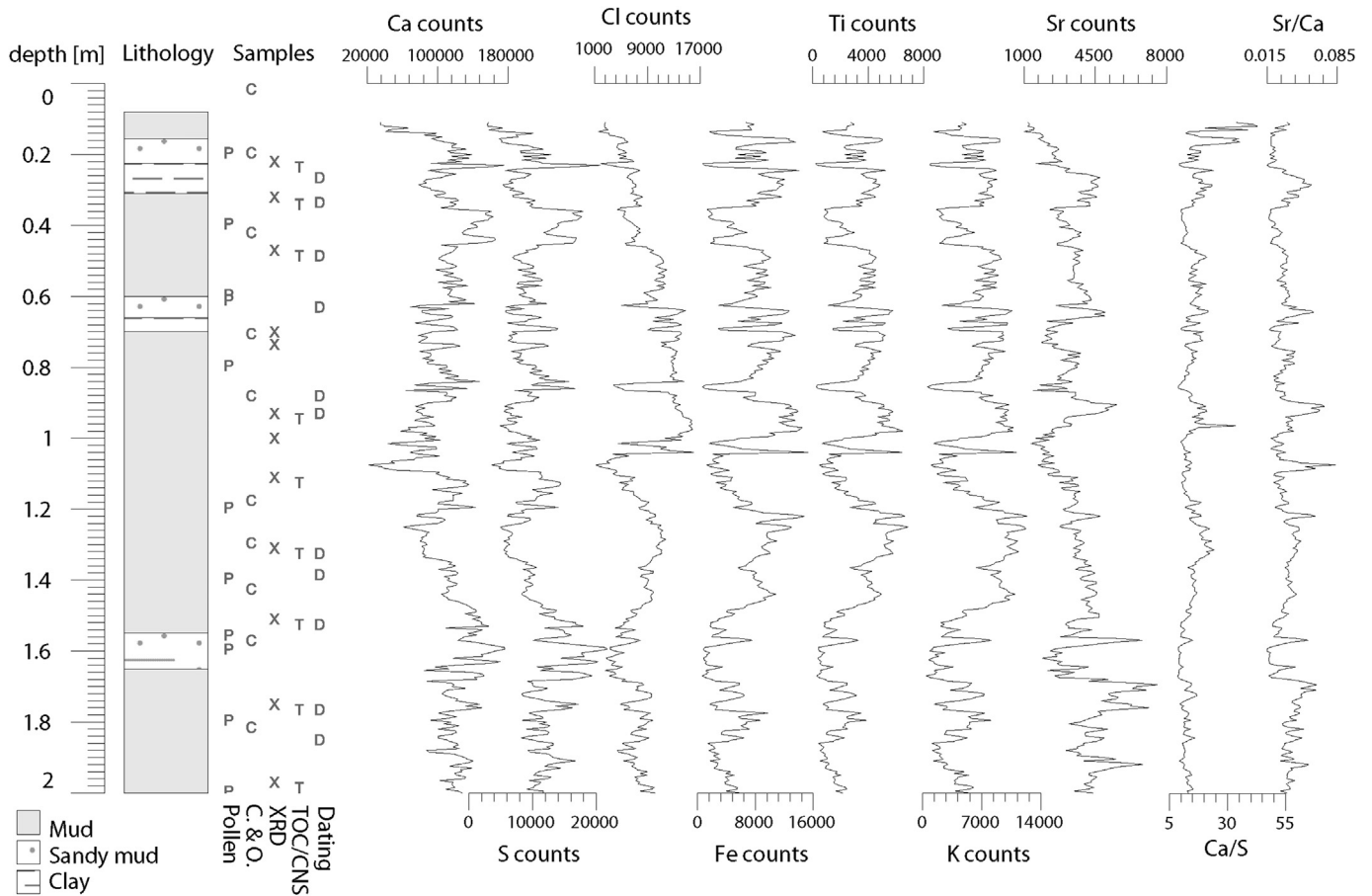


Fig. 2. Lithology of the core SAL 2, display of samples (dating, TOC/TIC, CNS, XRD, characeae, ostracods and pollen) & sample depth, as well as display of calcium, sulphur, chloride, iron, titanium, potassium and strontium counts as well as the Ca/S and Sr/Ca ratio.

very top contains a mud with very angular to angular gypsum grains with a grain size up to 200 μm . Within the core SAL2, angular grains do occur within “deeper” lake phases and times of clay sedimentation but also in “shallower” phases with coarser sediments, as for example between 1.70 and 1.60 m depth. In addition, both angular and lenticular grains were found at the same depth.

Except for the repeated beige-coloured sandy mud layer, the sediments are of beige grey colour. The more or less invariant sediment colour consequently gives no indication of changing redox conditions or periodic desiccation. A variation or occurrence of bluish or reddish sediments would in contrast allow a statement about both phenomena (Davis and Stevenson, 2007; Eusterhues et al., 2005). Erosion surfaces, representing desiccation phases of the lake, could not be identified. There is no considerable crust, nor are there obvious changes of sediment colour, which helped to identify desiccation phases together with magnetic susceptibility and TOC values at the Lake Sanabria in north western Spain (Giralt et al., 2011). Furthermore, the upper layers of SAL2 reveal traces of bioturbation and there are no internal sedimentary structures due the shallowness of the lake and respective bioturbation of the upper layers observed. The layer contacts are gradational, except for the contacts between the clay and sandy mud layers.

4.3. Geochemical results

The sediments within the core SAL2 mainly consist of Calcium (Ca) and Sulphur (S), minor components are Chloride (Cl), Iron (Fe), Titanium (Ti), Silicon (Si), Aluminium (Al), Potassium (K) and

Strontium (Sr) (Fig. 2). Ca and S vary significantly throughout the core and show a similar pattern of the correlation coefficient ($R = 0.87$, Table 2). This is contrasted by the opposite trend of Al, Si, K, Ti and Fe and their highly negative correlation coefficients to Ca and S. These elements can be associated with allochthonous minerals like clay, quartz and feldspar (Rodó et al., 2002; Cohen, 2003; Eusterhues et al., 2005; Martín-Puertas et al., 2011; Neugebauer et al., 2015) indicating their detrital source and catchment runoff. Fe cannot be used as redox signifier (Davies et al., 2015), as the water depth is too low for a thermal or chemical stratification.

High Ca and S contents can be explained by the surrounding geology (carbonates and gypsum) and related saline and sulphate-rich groundwater inflow which results in gypsum precipitation when evaporation of lake water takes place. High values, therefore, may indicate a low lake level (Martín-Puertas et al., 2011; Davis and Stevenson, 2007). However, it requires a sufficient supply of both elements in preceding times. The assessment of Ca and S being mostly incorporated in gypsum is supported by the XRD results (Fig. 3). Excess Ca, shown by an elevated Ca/S ratio (Fig. 2), can presumably trace back to calcite precipitation, as visible in XRD data. Generally, detrital carbonates are derived by erosion from the surrounding, while biogenic carbonate remains from skeletons of organisms like molluscs or phytoplankton (Tucker and Wright, 1990). Ostracods and Characeae within SAL2 reveal an additional occurrence of Ca within their shells.

Strontium is commonly associated with Ca in arid and carbonate environments (Davies et al., 2015), within this XRF-data set it shows a low negative correlation coefficient of -0.27 to Calcium.

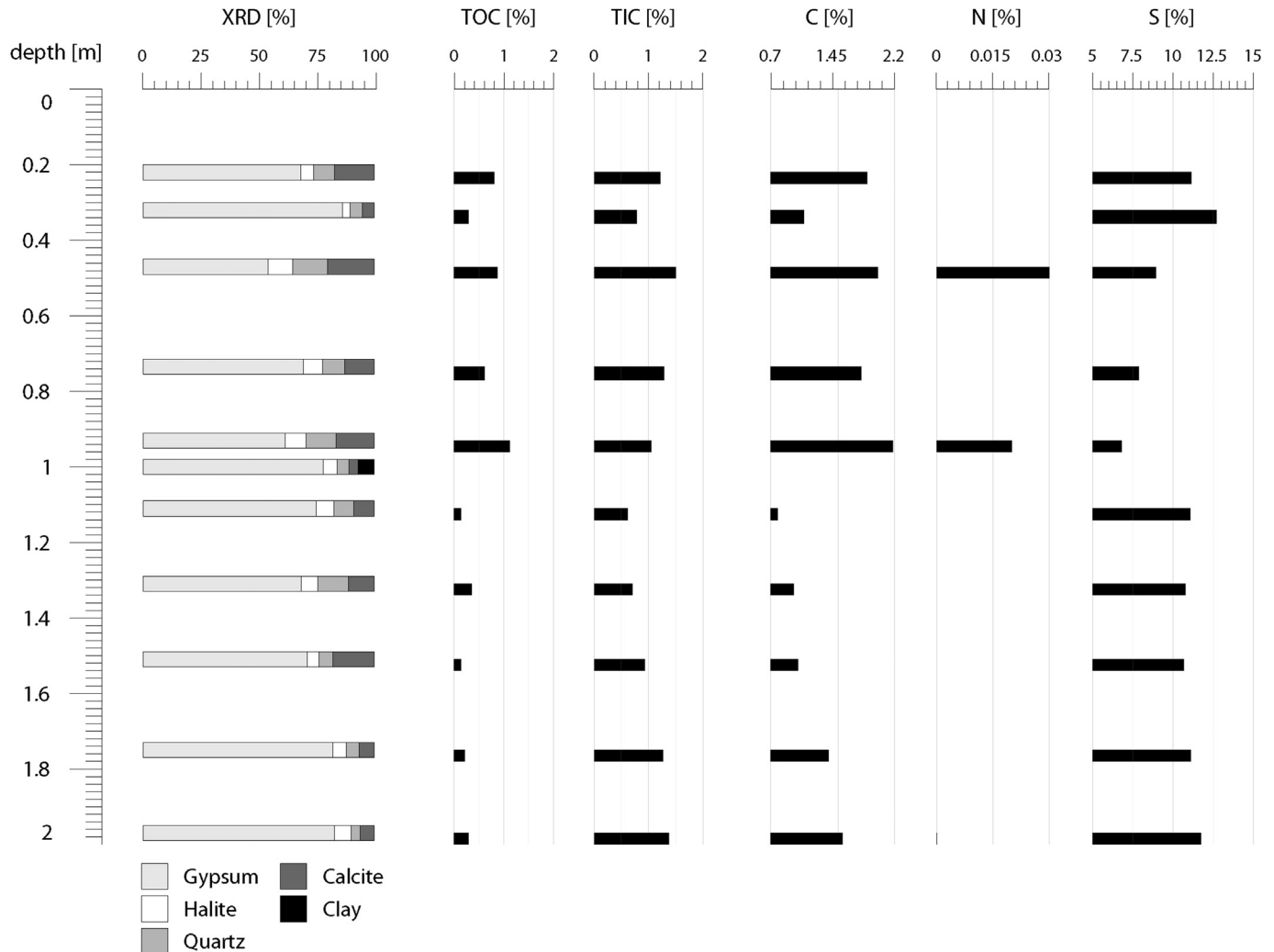


Fig. 3. Results of the XRD, TOC, TIC as well as CNS measurements.

Table 2
Correlation coefficient (R) of the elements Aluminium, Silicon, Potassium, Titanium, Iron, Chloride, Calcium, Sulphur and Strontium.

	Al	Si	K	Ti	Fe	Cl	Ca	S	Sr
Al	1,00								
Si	0,92	1,00							
K	0,90	0,98	1,00						
Ti	0,86	0,94	0,96	1,00					
Fe	0,83	0,90	0,94	0,94	1,00				
Cl	0,68	0,76	0,77	0,78	0,79	1,00			
Ca	-0,84	-0,92	-0,94	-0,93	-0,96	-0,90	1,00		
S	-0,83	-0,90	-0,92	-0,88	-0,90	-0,70	0,87	1,00	
Sr	0,20	0,23	0,22	0,15	0,19	0,13	-0,27	-0,31	1,00

Within Fig. 2, the Sr/Ca ratio shows deviation of Strontium being incorporated together with Calcium. The ratio is more or less similar to the Ca/S ratio and points to Sr being incorporated in carbonate related to authigenic carbonate precipitation comparable to the study of Martín-Puertas et al. (2011). Chloride shows positive correlation coefficients to the detrital facies between 0.68 and 0.79 and a stronger, highly negative correlation of -0.90 and 0.70 to Ca and S respectively. However, Cl may be incorporated in the sediment pore water (Neugebauer et al., 2015) and might as well, like the detrital facies elements, be superimposed by the occurrence of

gypsum within the sediments of SAL2. However, a combination of the halite content, derived from XRD measurements and the mean Cl counts (Fig. 4) shows good comparability and verifies, that most Cl is incorporated in NaCl. The sources of salt can be diverse, it can originate from surface or groundwater, hydrothermal water, aeolian dust and older evaporite deposits (Talbot and Allen, 1996). The evaporites are produced from saline solutions through precipitation due to evaporation or in coincidence of interstitial brines and groundwater or sediment (Hardie et al., 1978). In closed basin lakes like the Laguna Salada, salinity can build up over time due to evaporation (Cohen, 2003) and might therefore be related to lake level. This is not applicable for the sediments of SAL2, comparable to the study of the Laguna de Medina (Reed et al., 2001). Within Fig. 4, a comparison of the Gypsum content [%] derived from XRD analysis, as well as the mean Sulphur counts of the same depth range (XRF, wet core), the Sulphur content [%] measured with the Vario Micro Cube and Sulphur counts, measured on dried and homogenized samples with the XRF scanner, are given as well. They show different trends and are difficult to match, which is also related to the unit (cts) of the obtained XRF data which gives no absolute concentration (Boyle et al., 2015). Moreover, micro-XRF analysis does not give quantitative dry mass concentration values, the detected concentration is relative to the water content (Boyle et al., 2015). A comparison of both data sets obtained by XRF

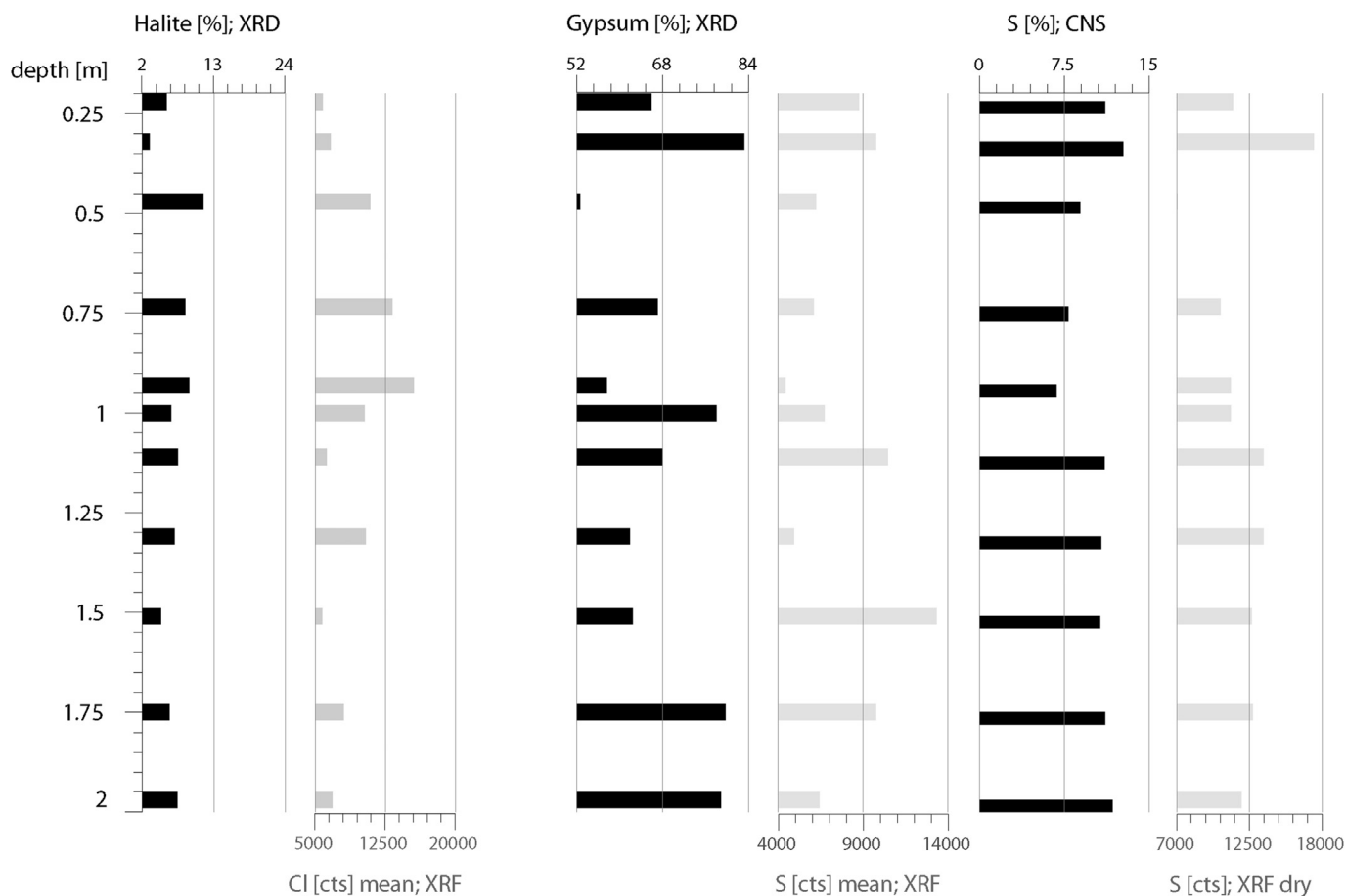


Fig. 4. Combination of XRD Halite content [%] and Cl mean values of the same depth range of the wet core [cts] and XRD Gypsum content [%] together with mean sulphur [cts] obtained by XRF measurements on the wet core, as well as Sulphur content measured by CNS [%] and the Sulphur counts obtained by XRF on dried and homogenized samples.

analysis, down-core measurements on the wet core as well as analysis on dried and homogenized samples, are shown in Fig. 5. Mean values of the XRF data derived from the wet core were calculated for the depth intervals sampled for measurements on dry samples. The Calcite as well as the Sulphur and Strontium counts increase, while the Chloride values decreased within the analysis on the dried and homogenized samples. The Iron, Titanium and Potassium counts differ on a higher level. In general, the deviations might also be related to the high resolution scanning on the core as well grain size and matrix effects.

The CNS, and TIC/TOC measurements were performed on 10 samples evenly distributed throughout the core. Organic matter content constitutes a biogeochemical component to the sediments which is affected by paleoclimate change (Meyers and Lallier-Vergès, 1999) and the TOC bulk value is fundamental to describe the abundance, production and degradation of organic matter (Martín-Puertas et al., 2011; Meyers and Lallier-Vergès, 1999). TOC content within the samples differ between 0.14% at and 1.12%, TIC content varies from 0.61% to 1.37%. These percentages, however, are too low to allow any reasonable paleoclimate interpretation. CNS measurements reveal a low nitrogen content between 0 and 0.03% and sulphur matter between 6.80% and 12.71%. Nitrogen usually allows an interpretation of a terrestrial or lacustrine origin of the organic material, as its concentration differs considerably between them (Cohen, 2003). Due to a sparse nitrogen content, only two of 10 carbon to nitrogen ratios could be calculated, 66 at 0.50–0.47 m, 109 between 0.96 and 0.93 m depth, indicating a land plant origin (Meyers, 1997; Meyers and Lallier-Vergès, 1999; Cohen, 2003). As

there is no more Nitrogen incorporated in the sediments of SAL2, it can neither be stated that the lake level was lowered and the algal production was the major source of organic matter (C/N between 4 and 10), nor that the contribution of reworked sediments from the lake margin was higher (as this would mean elevated values) (Meyers and Lallier-Vergès, 1999) during the according time of sediment deposition. Sulphur content usually varies in relation to the amount of organic matter (Glew et al., 2002). However, in this study it is apparently correlated to the gypsum content. Carbon content measured with the Vario Microtube shows negligible errors from total carbon measured with the Dimatoc 2000 analyzer.

4.4. Investigation of biological proxies

Four of the eleven pollen samples (at 1.79–1.80, 1.59–1.60, 1.55–1.56 and 1.39–1.40 m depth) were devoid of pollen and did only contain some mineral remains, charcoal as well as the added Lycopodium spores. The other seven displayed variable preservation and amount of pollen grains, but were all too sparse to provide reliable pollen counts. Identified pollen grains are Poaceae, *Quercus* sp., *Pinus* sp., *Abies* sp., *Potamogeton* sp., *Phragmites* sp., Cyperaceae, Asteraceae, *Phillyrea* sp., Malvaceae, *Olea europaea*, *Pistacia* sp., *Polygonum raii*, *Caryophyllaceae*, Fabaceae, *Plantago* sp., Ericaceae and *Nuphar lutea*. The conditions to preserve pollen grains in countable amounts were not assured either because of (i) aerobic deposition conditions, which requires a sufficient lake water depth (Bennett and Willis, 2002); (ii) sparse vegetation cover and increased erosion (Pantalón-Cano et al., 2003) and (iii) general

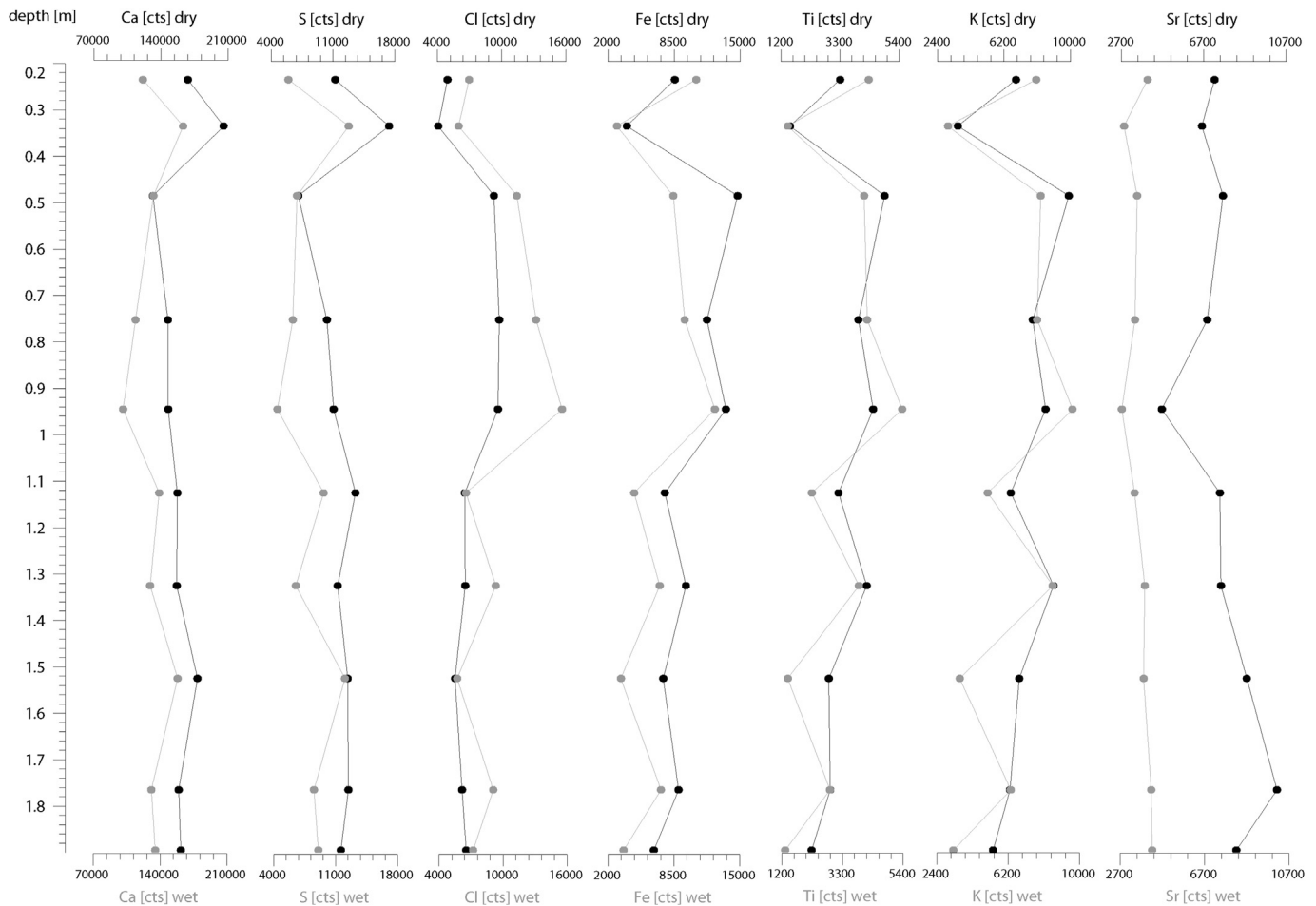


Fig. 5. XRF comparison of measurements on wet cores (mean values [cts] of the depth range) compared to XRF measurements on dried and homogenized samples.

difficulties connected to the saline, episodically desiccating lake environment (Carrión et al., 2009). Nevertheless, a survey of pollen grains, even in a halophilous system will only reflect its value in a try. No overall statement can be made about the pollen preservation potential within these environments (Carrión et al., 2009). The analysis of the pollen slides is not used for further interpretation because of its negligible significance. Still, it has to be mentioned despite of the unfruitful analysis for principle of completeness and to prevent other pollen analysts from wasting time (see Carrión et al., 2009).

Characeae analysis revealed a main occurrence of three species: *Chara aspera* Deth. ex Willd. 1809, *Chara canescens* Loisel. 1809 and *Lamprothamnium papulosum* (Wallr.) J.Gr. 1916 (Tables 3 and 4). *Chara aspera* and *canescens* are combined, as a differentiation of these species based on their morphological features was not possible. At a depth of 1.80 m, only empty lime shells of oospores were found. In the upper layers, *Lamprothamnium* and *Chara aspera/canescens* are mixed. In contrast, below 0.87 m depth a dominance of *Chara aspera/Chara canescens* oospores exists. A few oospores of the species - group *Chara hispida/Chara baltica/Chara globularis/Chara virgata* (Vedder, 2004) were found up to 0.18 m depth. The presence of Characeae shows that in the moment of sedimentation the lake was filled with water (Wray, 1998). Furthermore, the salt content was probably between 12 and 40 PSU because of the generation of calcareous shells (Soulié-Märtsche, 1998, 2008). The existence of small calcareous tubes within these samples is justified by dissolved calcium within the lake water and

related carbonic acid weathering (Grotzinger and Jordan, 2010).

The ostracod record is almost continuously monospecific and dominated by the euryhaline species, *Eucypris mareotica* (Fischer, 1851). Only a single shell of *Herpetocypris* sp. was identified in sample 1.41–1.43 m. However, both taxa are halotolerant organisms that are collected in modern non-marine shallow waters (Baltanás et al., 1990; Viehberg, 2006; Song and Wang, 2014; Rasouli et al., 2016). *E. mareotica* is mainly distributed in the southern region of the Iberian Peninsula and occurs in chloride rich waters with a salinity range between 12.9 and 80.9 PSU (Baltanás et al., 1990) and after Mezquita et al. (2005) and has a weighted optimum of 36 PSU and a tolerance range between 16 and 81 PSU. Iberian *Herpetocypris* species have a lower salinity preference and tolerance (0.3–1.1 PSU) (Mezquita et al., 2005).

5. Interpretation and conclusion

A multi proxy study is essential to define if a sediment archive is useful as climate archive and endorheic lakes have shown to be very sensitive to climate changes (Laird et al., 1996; Beklioglu et al., 2007; Davis and Stevenson, 2007; Höbig et al., 2016), despite of possible difficulties in climate information (Lane et al., 2013), which have been verified within this study. A comparison to other Iberian lake studies (Frigola et al., 2007; Corella et al., 2010; Martín-Puertas et al., 2011; Moreno et al., 2012; Valero-Garcés et al., 2006, 2014; Höbig et al., 2016) is difficult (Magri et al., 2004), as for example the elements and ratios measured and obtained by XRF data

Table 3

Morphometric data and features of charophyte fructifications identified in the Laguna Salada. LPA (longest polar axis); LED (largest equatorial diameter); ISI (isopolarity index; LPA/LED).

species (Vedder, 2004)		LPA (μm)	LED (μm)	ISI	striae	angle (°)	fossae (μm)	basal impression (μm)
<i>Chara aspera/Chara canescens</i>	min-max	533–822	212–392	1,6–3,8	9–12	57–88	36–62	39–71
	mean value (SD)	627,7 (±56,4)	290,5 (±47,5)	2,2	10	72,4	48 (±5,9)	55,3
	median (SD)	623 (±56,4)	293 (±47,5)	2,1 (±0,5)	10 (±0,7)	72,3 (±5,3)	47 (±5,9)	56,7 (±9,5)
<i>Chara aspera/Chara globularis/Chara virgata</i>	min-max	535–643	222–636	1–2,5	9–12	65–84	42–66	46–73
	mean value (SD)	605,2 (±39,4)	341,9 (±124,3)	1,9 (±0,4)	10 (±1,1)	72,8 (±6,3)	57,4 (±7,1)	62 (±8,6)
	median (SD)	616,6 (±39,4)	307,1 (±124,3)	1,9 (±0,4)	10,5 (±1,1)	70,6 (±6,3)	57 (±7,1)	61,1 (±8,6)
<i>Lamprothamnium papulosum</i>	min-max	576–745	220–410	1,6–2,5	7–10	64–75	48–72	39–88
	mean value (SD)	646,7 (±43,1)	310,8 (±41,1)	2,1 (±0,2)	8,5 (±0,8)	70 (±2,9)	59,5 (±6,2)	64,6 (±10,5)
	median (SD)	646,4 (±43,1)	318,1 (±41,1)	2,1 (±0,2)	8 (±0,8)	70 (±2,9)	59,7 (±6,2)	64,8 (±10,4)
<i>Chara globularis/Chara virgata/Chara hispida/Chara baltica</i>	min-max	692–740	239–374	1,6–2,5	10–11	64–77	46–62	49–70
	mean value (SD)	713,1 (17,4)	323,1 (±51)	2,1 (±0,2)	10,75 (±0,4)	72,3 (±4,9)	55,7 (±6)	63,3 (±8,3)
	median (SD)	709,6 (±17,5)	339,6 (±51,5)	2,1 (±0,2)	11 (±0,4)	73,7 (±4,9)	57,2 (±6)	66,8 (±8,5)

Table 4

Overview of the Characeae sample no., depth and species.

Sample no.	Depth (m)	Chara species
1	0–0.03	Blank
2	0.18–0.21	<i>Lamprothamnium papulosum</i> & <i>Chara aspera/canescens</i> , <i>C. hispida</i> , <i>C. baltica</i> , <i>C. virgata</i> , <i>C. globularis</i>
3	0.41–0.43	<i>Lamprothamnium papulosum</i> & <i>Chara aspera/canescens</i> , <i>C. hispida</i> , <i>C. baltica</i> , <i>C. horrida</i> , <i>C. virgata</i> , <i>C. globularis</i> , <i>C. vulgaris</i>
4	0.69–0.72	<i>Lamprothamnium papulosum</i> & <i>Chara aspera/canescens</i> , <i>C. virgata</i> , <i>C. globularis</i> , <i>C. hispida</i> , <i>C. baltica</i>
5	0.87–0.89	<i>Chara aspera/canescens</i>
6	1.16–1.19	<i>Chara aspera/canescens</i>
7	1.28–1.31	<i>Lamprothamnium papulosum</i> & <i>Chara aspera/canescens</i>
8	1.41–1.43	<i>Lamprothamnium papulosum</i> & <i>Chara aspera/canescens</i>
9	1.55–1.59	Calcareous shells of oospores only
10	1.80–1.82	"

depends on the geological setting and catchment area as well as sediment transport (Talbot and Allen, 1996; Lopez et al., 2006). However, climate records are often synchronized, disregarding spatial patterns and temporal transgressive differences (Giralt et al., 1999; Lane et al., 2013). As wiggle-matching neglects different temporal response times of particular proxies (Brauer et al., 2014) which can be up to a thousand years (Giralt et al., 1999) it was not applied to the data obtained in this study. Instead, a summarizing overview of some proxies (Fig. 6) and evaluation of the all studied proxies will be given. The data of SAL2 provides valuable insights e.g. about the elemental and mineralogical composition as well as salinity and fossil occurrence; but cannot be used for giving meaningful climate information. However, it should not be taken as a dismissal of hypersaline playa lakes being suitable climate archives, but identify problems faced at the interpretation of the core SAL2 of the Laguna Salada.

Carbonates are the first minerals of an evaporitic sequence which precipitate when their saturation coefficient is exceeded (Warren, 2006) and the evaporite minerals of SAL 2 are mostly made of gypsum. This would be followed in a general evaporitic sequence by the deposition of saline minerals (Talbot and Allen,

1996). The halite content, measured by XRD, could be linked to the CI counts obtained by XRF data and shows its significant value. However, it does not give a direct indication of the lake level, as the chemical composition of the lake sediments are overprinted by the high amount of gypsum. Nevertheless, predictions concerning paleosalinity can be made due to Characeae and Ostracod analyses, as the variable occurrence of Characeae species within the lake sediments implies a fluctuating water salinity (Fig. 6). The conservation potential of Characeae regarding changes of temperature and water desiccation is very good (Krause, 1997) and the living species do react to salinity. The presence of oospores of the Characeae species *Chara aspera*, *Chara canescens* and *Lamprothamnium papulosum* indicates saline conditions as they are halophytic species. While *Lamprothamnium papulosum* can occur at salinities of up to 35 PSU, is the optimum of *Chara canescens* at 12 PSU and of *Chara aspera* in a range of 14–18 PSU (Blindow and van de Weyer, 2016). Fructifications of *Lamprothamnium papulosum* indicates phases of salinities between 20 and 40 PSU. It is described as upper limit for sexual reproduction (Soulié-Marsche, 1998). As all samples comprising Characeae incorporated the ostracod species *Eucypris mareotica*, the paleo salinity likely varied above 16 PSU. The increase in Characeae vegetation might indicate a trend to less extreme environments. An overview of a simplified and combined salinity range for both Characeae and Ostracods is given in Fig. 6, which shows the CI curve [cts] (obtained on the wet core and superimposed by the gypsum content). The high salinity ranges explain the occurrence of evaporites within this strongly saline system. It has to be taken into consideration that the analysed sample resolution of Characeae analysis cannot resolve seasonal salinity changes (spring precipitation/summer desiccation) and thus changes in species population densities. Hence, the signal of the characteristic seasonal change in playa lake waters of e.g., increasing ion concentration, decreasing alkalinity needs a higher resolution sampling protocol.

Due to the sparse pollen content, a relation to human influence and activities as e.g. in Valero-Garcés et al. (2006) cannot be given. Nevertheless, pollen are, if present, a suitable proxy for local vegetation history at small lake sites and may show regional trends related to changes of the climate (e.g. Magri et al., 2004). The observation of the shape and size of gypsum grains did not give reason for an interpretation or explanation of fluctuating lake level and gypsum transport or deposition.

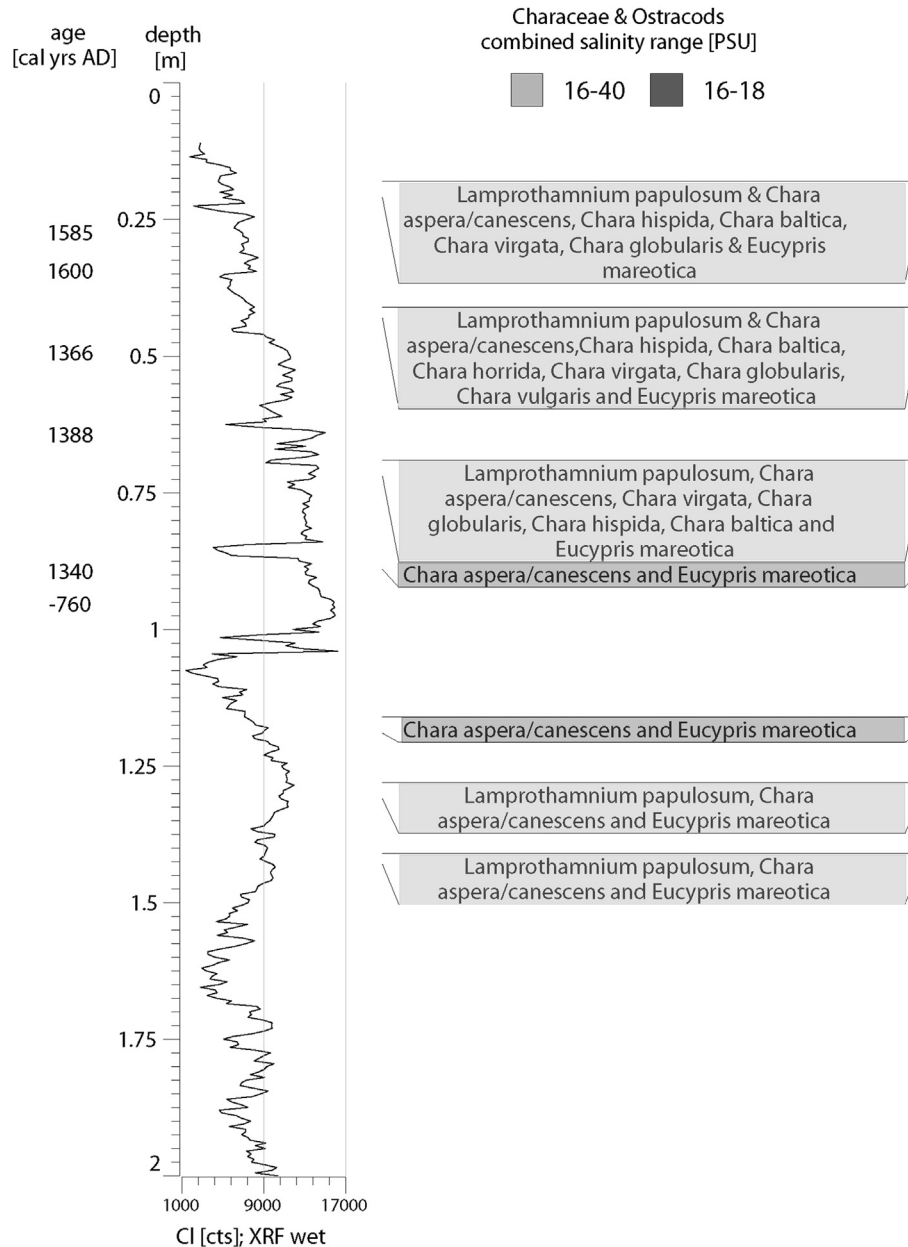


Fig. 6. Overview of the obtained ages [cal yrs AD], CI counts and salinity ranges (PSU) obtained by Characeae and Ostracod analyses.

According to the geochemical data, the gypsum precipitation curve is contrary to the detrital input, but this is based on gypsum overprint. It does not allow an interpretation concerning gypsum precipitation in times of evaporation and arid climate as done by Martín-Puertas et al. (2011) and possible humid times, expressed by higher input of detritus. In general, the detrital input might be interpreted as a lake level reconstruction curve, as the aluminosilicate fraction enters the lake through erosion by precipitation and surface run-off (Rodó et al., 2002; Eusterhues et al., 2005; Lopez et al., 2006; Jimenez-Espejo et al., 2008; Martín-Puertas et al., 2010, 2011), consequently affecting lake level. However, the geochemical data is dependent on sample selection, measurement type, water content, grain size and potential matrix effects (see Fig. 5 and chapter 4.2) etc. which highly influence its comparability and potential interpretation. On that account, measurements of the water content could essentially improve the value of the data.

The dominance of the ostracod species *E. mareotica* throughout the core does not allow the reconstruction of changing lake levels, and overprints the salinity ranges given by Characeae analysis, but it suggested that the water depth was always shallow. After Valero-Garcés et al. (2006), the phase after 1300 AD until the late 19th century was affected by a variable lake level, followed by a dry period until the early 20th century, but it is not safe to say that there happens the same in the region surrounding Laguna Salada due to the lack of pollen data, gypsum precipitation and a consistent age chronology. It is impossible to put paleoclimate information into a wider context, as in many other studies on Iberian saline lakes (e.g., Giralt et al., 1999; Höbig et al., 2016). According to Alvarez Cobelas et al. (2005), this ambiguity is applicable for most Mediterranean lakes, as they do not constitute cold temperate stratified lakes.

In general, the ^{14}C dating on plant remains within the lake

sediments is promising, but in shallow saline lakes, the amount of organic matter is low. Especially the low water level at recent times, gypsum precipitation and bioturbation may have influenced the datable material, implying that in greater sediment depth, the outcome might be quite different if the lake level was higher at the time of sediment deposition. Organic lake sediments are extremely prone to contamination and time representative material is essential for accurate dating. As radiocarbon dates can also be obtained on charcoal (Walker, 2005) and the same is abundant within the lake sediments and less vulnerable to diagenesis and contamination, this might be worth a try. The dating on ostracods might be more feasible, as the amount within the lake sediments is higher. Nevertheless, the dating can be defective as the age calculation algorithm was initially invented for the specific species *Cyprideis torosa* (Ortiz et al., 2013). Potassium-argon dating, Argon-Argon dating, U-series, cosmogenic nuclide dating and the fission track method are not constructive or applicable for dating young lake sediments up to a couple of thousand years (Walker, 2005; Lian and Roberts, 2006). Dating with varve chronology in terms of counting thin annual layers is not possible because of their non-presence within the sediments of the Laguna Salada. Dating on ^{210}Pb as well as ^{137}Cs can only cover a very recent time span up to several hundred years. ^{32}Si dating can cover the last millennia and might be useful for the very upper part of sediments. Radiometric dating like Thermoluminescence or Optically Stimulated Luminescence as well the investigations of palaeomagnetism might be reasonable, but will also be affected by reworking of sediments and drying out of the lake (Walker, 2005) and would also likely give erroneous ages. On that account, an investigation of deeper, less bioturbated and less frequently desiccating lakes with less gypsum deposition might be in general more prospective. Additionally, the low amount of quartz and feldspar within the sediments of the Laguna Salada has to be taken into account, as it decreases the chance of a meaningful age obtainable by luminescence dating.

The interpretation of the data from the Laguna Salada and other paleoenvironmental studies could be enhanced by a monitoring of the local catchment and setting as well as the water-holding capacity of surrounding soils, e.g. as this can strongly influence the dating (Höbig et al., 2016). Furthermore, a survey of possible vegetation patches which can store more water and reduce the erosion through precipitation by 50% and runoff energy by 75% (Hupy, 2004) and monitoring of modern in-lake processes would lead to a better comparability to other Iberian salt lakes and a well-grounded interpretation of proxies. Sampling for TIC/TOC measurements at smaller intervals and optical investigation of the organic material itself could enhance the understanding of its provenance and value, even though the content of TIC and TOC within the sediments of SAL2 is pretty low, related to the high gypsum content. A lake survey by the use of multiple cores could be helpful as well, even more if the lake desiccates in dry years, leading to a non-continuous sedimentation/hiatus and erosion (Carrión et al., 2009). Comparable studies of Laguna de Medina, southern Spain (Reed et al., 2001) and Van't Hoff et al., (2016) (unpublished results) have shown that the same lake, cored multiple times, can show significant differences as for example in pollen preservation and sedimentation rate.

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