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A 6000-year geochemical record of human activities from Alexandria (Egypt)

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ABSTRACT

Recent multidisciplinary investigations of sediment cores from the ancient marine bay of Alexandria (Egypt) have documented local human activities during the Iron Age (circa 900–1000 B.C.) prior to Alexander the Great's arrival in 331 B.C. (Goiran, 2001; Véron et al., 2006; Stanley et al., 2007, 2010), corroborating the existence of the so-called "Rakhotis" as evoked in previous archaeological literature (Jondet, 1916; Weill, 1919; Chauveau, 1999; Baines, 2003). Lead (Pb) Isotopic Analyses (LIA) from Alexandria Bay indicate a possible anthropogenic imprint as early as circa 2300–2650 (±200) B.C. and, to a lesser extent, 3500–3800 (±170) B.C. (Véron et al., 2006). Here we demonstrate that LIA in sediments from the nearby Maryut Lagoon display isotopic anomalies resulting from the release of contaminant Pb into the Lagoon during the Egyptian Early dynastic (at 2897 ± 187 B.C) and Predynastic (at 3520 ± 145 B.C.) periods that corroborate geochemical data from Alexandria Bay. Pb concentrations in Maryut sediments show enrichments that mirror isotopic findings. The absence of contaminant Pb imprints within the sediments from the nearby Nile Canopic branch confirms that isotopic anomalies found in Alexandria Bay and Maryut Lagoon sediments are local and do not originate from long-distance transport of contaminant Pb associated with Nile suspended material and/or wind-derived aerosols. LIA in Alexandria sediment cores correspond to commonly mined Pb and Cu (copper) ores from Turkey (Black Sea region), Cyprus, Crete and the Oman Gulf. LIA substantiate the use of copper-based relics in the Alexandria region during the Pre and Early dynastic periods, and provide insights into metal trading within the Eastern Mediterranean during the Chalcolithic–Early Bronze Age transition, 6000 years ago.

1. Introduction

In 331 B.C. Alexander the Great founded Alexandria ad Aegyptum on the eastern margin of the Nile Delta. It rapidly grew into one of the most densely populated and developed cities of the Mediterranean Basin (Fraser, 1972; Empereur, 1998; Yoyotte et al., 1998). This growth led to the discharge of anthropogenically-derived Pb into the marine bay with a significant peak in pollution at the culmination of the Roman occupation (Goiran, 2001; Véron et al., 2006). Multidisciplinary investigations (including archaeology, stratigraphy, palaeoecology and geochemistry) of sediment sequences from Alexandria's ancient harbours suggest the existence of a pre-Alexander settlement during the Iron Age (Goiran, 2001; Véron et al., 2006; Stanley et al., 2007; Stanley and Bernhard, 2010), referred to by ancient authors as "Rhakotis" (e.g. Herodotus, Pliny and Strabo) (Jondet, 1916; Weill, 1919; Chauveau, 1999; Baines, 2003). Véron et al. (2006) have reported Pb contamination traces in sediments from Alexandria Bay that could indicate the occurrence of human settlements at 2300–2650 (±200) yrs B.C. and, to a lesser extent, 3500–3800 (±170) B.C. Increases in Pb concentration trace the foundation and subsequent growth of Alexandria and are therefore a robust proxy to reconstruct human settlements in the absence of clear archaeological evidence (Véron et al., 2006; Stanley et al., 2007). Nonetheless, uncertainties persist regarding the extension of the so-called "Rhakotis" before its Hellenistic occupation due mainly to the dense urban fabric and the complexity of the pre-Hellenistic archaeological artifacts. Furthermore, the local origin of anthropogenic Pb contamination has been questioned owing to possible long-range transport by wind and fluvial processes. In order to (i) confirm a clear local human origin and (ii) probe metal trade

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networks, we compared isotopic anomalies found in Holocene sediments to Pb isotope source signatures from Pb and Cu ores originating from the Eastern Mediterranean and Middle East regions.

To investigate these questions and test the veracity of a pre-Alexander settlement, we extended our analysis to sediment records from the Maryut Lagoon (core M12), a wetland system that backs onto Alexandria and was formed during the early to mid-Holocene (Flaux et al., 2011). To differentiate between local and long-range transport of contaminant Pb, we also analysed a core collected from the Canopic branch (core M57), a palaeo-branch of the Nile, east of Alexandria (Fig. 1).

Among key anthropogenic markers Pb has proven to be an efficient tracer of past and present human activities. Pb, along with Cu, was amongst the first metals to be refined and used owing to its low melting temperature, malleability and resistance to corrosion (Nriagu, 1983). The oldest Pb artifacts are date back to 8000 years ago in Anatolia and Pb has been extensively used since then in plumbing, ship building, coinage, warfare, pigments, cosmetics, jewellery, glazes, masonry, glass, writing tablets and tombs (see references in Lucas and Harris, 1962; Nriagu, 1983). Its stable isotopes, ^{204}Pb, ^{206}Pb, ^{207}Pb, ^{208}Pb, of which the last three are end-members of the uranium (U)-thorium (Th) decay chains, allow to elucidate and discriminate the use of Pb-containing ores from various geographical origins depending on the age and the initial U–Th content of their primary geological reservoirs (Doe, 1970). Ratios of Pb isotopes will vary according to the origin of ores used to manufacture metal artifacts, either from Pb mining (Brill and Wampler, 1967; Brill et al., 1973; Gale and Stos-Fertner, 1978; Gale, 1979; Chamberlain and Gale, 1980; Stos-Gale and Gale, 1981, 1982, 2009; Gale and Stos-Gale, 1982a, 2000; Pernicka and Bachmann, 1983) or Cu mining (Gale and Stos-Gale, 1982b, 1986, 1999; Stos-Gale et al., 1986, 1997; Hauptmann, 1992; Stos-Gale and Gale, 1994; Gale, 1999; Niederschlag et al., 2003; Begemann et al., 2010). Consequently these isotopic imprints can be used to reconstruct ancient human activities recorded in environmental archives such as peatbogs (Shotyk et al., 1998; Dunlap et al., 1999; Cortizas et al., 2002), marine and lacustrine sediments (Chow et al., 1973; Shirahata et al., 1980; Renberg et al., 1994; Monna et al., 1999; Brännvall et al., 2001; Le Roux et al., 2003; Véron et al., 2006; Stanley et al., 2007; Thevenon et al., 2011).

2. Archaeological context

The Nile Delta has long been considered a hostile wetland for human settlements and therefore very few surveys have been conducted in this area. Archaeological research on the Nile Delta has been hindered by recurrent flooding which has buried/eroded archaeological evidences (Stanley and Warne, 1993; Butzer, 2002; Flaux et al., 2013), and archaeological biases that have favoured excavations in the more attractive central Nile valley. Nonetheless, Montet (1942) and Vandier (1952) raised the question of complex communities on the Nile Delta during the Egyptian Predynastic period (circa 5500–3100 B.C.) after the discovery of the Predynastic settlement of Merimde Beni-Salâme (ca 4900–4300 B.C.; Junker, 1929) on the southwestern art of the delta. Human settlements were also evidenced in the Memphite region as early as 5400 B.C. (Fayum culture, see references in Hendrickx, 1999; Midant-Reynes, 2003; Tristant, 2005, 2006). Further communities were discovered at Buto in the northwestern delta and Maadi in the Memphite area that developed at the end of the Merimde Beni-Salâme period, circa 3900 B.C. (Bovier-Lapierre, 1926; Köhler, 1998; Tristant and Midant-

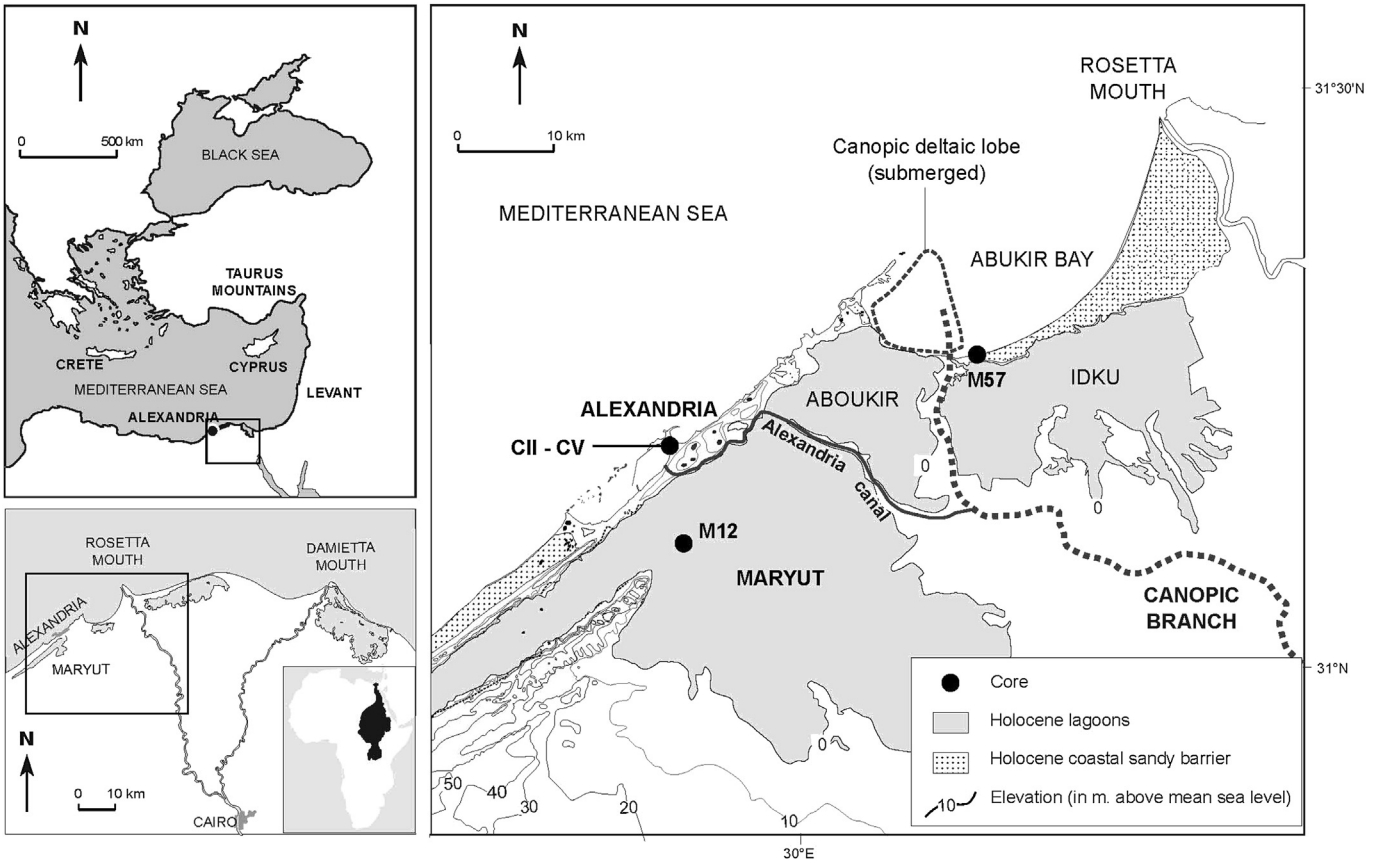

Fig. 1. Map of the sampling sites in the Alexandria region: Alexandria Bay (core CII, CV), Maryut lagoon (M12) and the Nile Canopic branch (M57).

Reynes, 2011). Numerous copper artefacts and architectural designs found at these sites suggest relationships with the Levant and, in particular, with the mining regions of the Sinai (see references in Perrot, 1984; Rizkana and Seeher, 1989; Tristant, 2006). Evidence of trade between Egypt and the Levant has been found in numerous archaeological excavations from the eastern delta where more than 60 sites were investigated, leaving the western delta largely unexplored (Schott et al., 1932; Saad, 1969; Redmount, 1986; Chlodnicki et al., 1992; Van den Brink, 1993; Köhler, 1998; Chlodnicki and Cialowicz, 2003; Midant-Reynes et al., 2003; Tristant, 2005; Tristant and Midant-Reynes, 2011; Abdel-Motelib et al., 2012). Artefacts found at Buto and Maddi during the 4th millennium not only linked the so-called Lower Egypt cultures (Faltings, 1998; Cialowicz, 2005; Tristant, 2005) to Palestine, but also possibly to Mesopotamia and Anatolia (Margueron, 1991; Miroschedji, 1991; Faltings et al., 2000; Bavay et al., 2004; Guyot, 2004; Tristant, 2005). At the end of the Predynastic period (3600–2700 BC), we observe a full extension of Levantine trade and the growing influence of the southern Naqadian culture (Rizkana and Seeher, 1984; Abdel-Raziq et al., 2002; Meurice and Tristant, 2004; Tristant and Midant-Reynes, 2011).

Pb isotopic analyses of Holocene sediments may provide further evidence for trade exchanges between the Egyptian communities and the Levant region during the Pre and Early dynastic periods from the poorly investigated western Nile Delta, and possibly raise clues on interactions with other Mediterranean regions.

3. The study site

Here, we present the results of Pb concentration and its stable isotopes from one of the cores (M12) that shows continuous sedimentation for the past 6000 years. Palaeoecological and sedimentological analyses suggest a typical lagoon environment with intermittent marine inputs (Flaux et al., 2011, 2013). A clear confinement between 7500 B.P. and 4800 B.P. with pronounced Nile influence is followed by increasing marine inflows.

This marine influence is favoured by lower Nile floods due to an increased aridification of the Nile Basin starting circa 5000 B.P. (Bell, 1970; Woodward et al., 2007; Flaux et al., 2013; Marriner et al., 2013). During this period, the source of dominant Nile sediment inputs shifted from the White Nile (Precambrian metamorphic basement from the Lake Victoria region) to the Blue Nile (flood basalts of Tertiary age from the Ethiopian Highs) (Krom et al., 2002) Further stratigraphic descriptions of the Maryut core M12 are presented in Flaux et al. (2011).

The age model is based on 15 radiocarbon dates, compiled from cores taken in the northwestern Nile Delta area. Carbonate samples comprise connected lagoon shells (*Cerastoderma glaucum*). Organic samples include wood and organic-rich lenses taken within the sediment sequence. All of these samples were taken at stratigraphic boundaries, each of them being dated by two to six independent radiocarbon measurements. The mean age $\pm 2\sigma$ was used for calibration. Details and reservoir ages used for calibration are provided and discussed in Flaux et al. (2013). Sedimentation rates in cores M12 were calculated based on the calibrated ages of stratigraphic boundaries (Table 1).

A core (M57, Fig. 1) was also sampled in the Nile Canopic branch, near Idku Lagoon, at the edge of the Canopic deltaic lobe. This Nile channel was active for at least the past 6000 years (Chen et al., 1992; Stanley and Warne, 1993; Stanley et al., 2004) and recorded past Nile influences on the western delta. M57 is a 10 m core comprising fluvial sands and lagoon mud as the main environmental deposits separated by a 60 cm thick organic layer dated circa 5000–6000 B.P. (Stanley et al., 1996). No accurate stratigraphy could be obtained from this core due to the depositional context and uncertainties in the age model that need to be resolved. Pb was analysed between 150 and 850 cm depth (i.e. circa 3000–7000 B.P.) in order to investigate Pb isotopic imprints during the Early Bronze Age period and possible anthropogenic influences associated with long-distance fluvial or aeolian transport to the Alexandria region.

4. Methods

Sediment from the Maryut Lagoon and the Nile Canopic branch were dissolved with concentrated acids (HNO_3, HCl, HF) at 130 °C to extract total Pb from each sample (bulk Pb) in HEPA-filtered class 100 clean laboratories using acid-cleaned teflon ware at CEREGE (Aix Marseille University) and GEOTOP (University of Quebec at Montreal, Canada). Procedural Pb blanks were always below $0.2 \cdot 10^{-9}$ g (Alleman et al., 2000; Poirier, 2006) and negligible as compared to the amount of Pb extracted from sediment samples ($10–100 \cdot 10^{-9}$ g). An exchangeable fraction of the sediments was also leached using 0.5 M HNO_3 in order to extract Pb that comprises an exogenous fraction attached to mineral and organic particles (as opposed to mineral Pb bound within silicates) and of some mineral Pb extracted from weakly structured crustal constituents (see references in Tessier et al., 1979; Shirahata et al., 1980; Trefry and Metz, 1984). The exogenous fraction usually corresponds to contaminant Pb that became attached to sediment particles when it was released into the basin from human activities. Thus, leached

Table 1
Maryut Lagoon sediments (M12): ^{14}C dates, Pb and Fe concentrations in the bulk fraction, Pb isotopic ratios for the bulk and leached fractions.

Depth (cm)	^{14}C corr. (years BC)	Pb ppm	Fe ppm	EF	$^{206}Pb/^{204}Pb$ bulk	$^{206}Pb/^{207}Pb$ bulk	$^{208}Pb/^{206}Pb$ bulk	$^{206}Pb/^{204}Pb$ leach	$^{206}Pb/^{207}Pb$ leach	$^{208}Pb/^{206}Pb$ leach
385–420	1167	6.01	47,085	0.7	18.921	1.2076	2.0588	18.957	1.2094	2.0548
420–455	1351	7.07	32,359	0.8	18.938	1.2080	2.0588	18.970	1.2095	2.0558
455–490	1535	5.33	43,275	0.9	18.908	1.2063	2.0599	18.953	1.2088	2.0566
490–525	1720	9.54	42,082	1.2	18.911	1.2067	2.0595	18.937	1.2076	2.0577
525–560	1904	7.37	56,803	1.0	18.885	1.2051	2.0628	18.957	1.2094	2.0554
560–595	2088	9.65	53,551	0.9	18.840	1.2028	2.0637	18.955	1.2099	2.0544
600–630	2377	10.3	58,678	1.1	18.819	1.2013	2.0657	18.887	1.2053	2.0600
630–665	2647	10.8	49,235	1.0	18.848	1.2028	2.0649	18.951	1.2090	2.0563
665–700	2897	13.5	73,920	1.5	18.686	1.1932	2.0722	18.615	1.1890	2.0755
700–735	3229	12.7	45,274	1.0	18.915	1.2062	2.0608	18.959	1.2102	2.0530
735–770	3520	12.2	57,261	1.5	18.731	1.1957	2.0694	18.775	1.1979	2.0667
805–840	4102	10.9	49,574	1.1	18.908	1.2058	2.0616	18.924	1.2077	2.0568
840–875	4393	10.3	46,885	1.2	18.934	1.2066	2.0690	18.964	1.2089	2.0575
910–945	4974	10.1	17,668	1.2	18.902	1.2048	2.0648	18.975	1.2096	2.0564
990–995	5559	5.30	53,530	1.7	18.832	1.2020	2.0712	18.976	1.2095	2.0558
995–1015	5675	10.6	47,085	1.1	18.851	1.2019	2.0666	19.032	1.2126	2.0532

fractions of contaminated sediments provide valuable clues on the isotopic signature of the ore from which contaminant Pb is derived owing to the absence of isotopic fractionation in surficial reservoirs (Ault et al., 1970; Flegal and Smith, 1995) and the previously described isotope specificity of each ore field. Pb and iron (Fe) concentrations from bulk sediments were measured by Inductively Coupled Plasma Mass Spectrometry (Thermo Scientific Xseries II ICP-MS) at the Laboratoire des Mécanismes et des Transferts en Géologie (LMTG, Paul Sabatier University, Toulouse, France) and are presented in Table 1. The standard deviation for these concentrations was below 3%. Enrichment factors (EF) were calculated as the ratio of Pb/Fe in bulk samples as compared to the crustal background for each sediment core. It reveals possible Pb enrichment within the sediment sequence with EF > 1. The EF can be used as an index to establish if sediments are enriched with "non crustal" Pb, but it cannot provide insights into anthropogenic sources owing to natural mineralogical variations and the slight Pb contamination generally encountered in these ancient deposits. Aliquots from total acid digestion and leached fractions were purified on AG1X8 anionic exchange resins (Manhès et al., 1978) for subsequent stable Pb isotopic analysis. Pb recovery from AG1X8 extraction was generally higher than 92% (Alleman, 1997). Isotopic ratios ($^{206}Pb/^{204}Pb$, $^{206}Pb/^{207}Pb$, $^{208}Pb/^{206}Pb$, Tables 1 and 2) were determined by Multi-Collector-Inductively Coupled Plasma Mass Spectrometry (MC-ICPMS, Belshaw et al., 1998) at GEOTOP on an IsoProbe instrument using an Aridus desolvation membrane as the introduction system. Data were normalized to multiple measurements of the NBS-981 international standard low temperature TIMS values from Doucelance and Manhès (2001) reported in Thirlwall (2002), interspersed throughout each analytical session. Long-term reproducibility assessment from internal standard measurements was better than 0.03% for all Pb isotopic ratios.

Pb isotopic signature of contaminated bulk and leached sediment fractions can be used to trace the geographical origin(s) of metal ores whose imprints are recorded in sediments (see references in Shirahata et al., 1980; Véron et al., 2006). In the absence of archaeological data, these geochemical signatures provide insights into both human settlements and the provenance of Cu or Pb ores. Several difficulties have arisen regarding the use of LIA for provenance studies of ingots and artifacts that include: (1) the heterogeneity of ore deposits; (2) isotopic fractionation during smelting that can affect isotopic ratios; and, (3) re-smelting/recycling of artifacts from diverse origins, and pooling of various ores from different locations to produce ingots.

Geological heterogeneity rarely exceeds 0.2–0.3% for ores of archaeological significance (Barnes et al., 1974; Stos-Gale and Gale, 2009). The definition of an ore field signature requires a large amount of LIA due to possible overlapping of isotopic imprints that occurs between geographically distant ore fields. It can only be resolved by expanding the Pb isotope data set (Pernicka et al., 1990; Gale et al., 1997; Stos-Gale et al., 1997; Gale, 1999; Baxter et al., 2000). To this end, we have included a large range of LIA for each potential ore source region to investigate the possible provenance of contaminant Pb found in Alexandrian sediments (Figs. 4 and 5).

Isotopic fractionation is not suitable for LIA provenance studies (Budd et al., 1995a; Pollard and Heron, 2008). This fractionation has been predicted from thermodynamic models (Mulliken and Harkins, 1922) that did not properly describe the smelting processes. More realistic and experimental models dismiss this issue and show that no measurable fractionation takes place during smelting (Barnes et al., 1978; Pernicka and Bachmann, 1983; Gale and Stos-Gale, 1996; Macfarlane, 1999; Baron et al., 2009; Stos-Gale and Gale, 2009; Cui and Wu, 2011).

Although not common, there is some evidence for metal recycling and pooling during the late Bronze Age (Gale and Stos-Gale, 1995; Pernicka, 1995; Sayre et al., 1995; Budd et al., 1995b; Stos-Gale et al., 1997; Knapp, 2000; Gale, 2001). The examination of isotopic mixing lines provides insightful clues on ore pooling (Gale, 2001). Indeed, when mixed, ore signatures plot along a straight line that connects end-members in isotope plots. In the case of contaminant Pb accumulated in sediments, one of the end-members is constrained by the natural background imprint. Mixing between this background and the isotopic signature of Pb-derived ores generates a line that allows ore signatures in each sediment reservoir to be differentiated for given periods.

Table 2
Pb isotopic ratios in sediments (bulk fraction) from the Nile Canopic branch (core M57). (*) corresponds to a date determined from stratigraphic correlation with other local cores (Stanley et al., 2004; Flaux et al., 2011, 2013).

Depth (cm)	^{14}C corr. years BC	$^{206}Pb/^{204}Pb$ total fract.	$^{206}Pb/^{207}Pb$ total fract.	$^{208}Pb/^{206}Pb$ total fract.
150	1030–1130	18.751	1.2000	2.0598
165		18.766	1.2000	2.0539
175		18.892	1.2080	2.0499
200		18.957	1.2120	2.0443
220		18.831	1.2030	2.0595
280		18.697	1.1960	2.0630
300		18.778	1.2000	2.0607
350		18.841	1.2040	2.0565
400		18.939	1.2100	2.0499
450		18.896	1.2070	2.0551
500		18.891	1.2060	2.0611
520		18.910	1.2070	2.0593
570	2800 ± 200	18.887	1.2050	2.0586
590		18.872	1.2030	2.0656
600	3000–3400*	18.830	1.2010	2.0695
670		18.436	1.1780	2.0734
700		18.344	1.1750	2.0827
750		18.274	1.1710	2.0898
800	4800 ± 200	18.325	1.1740	2.0726
850		18.318	1.1730	2.0827

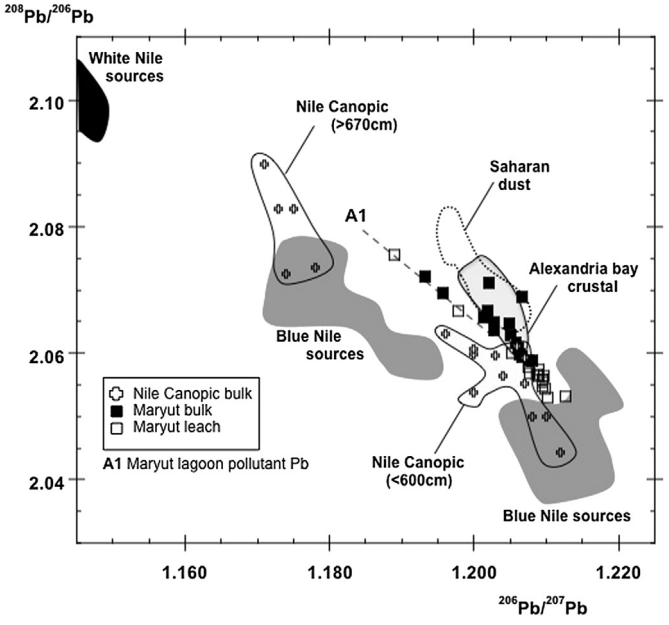

Fig. 2. Pb isotopic plot showing the geochemical background imprints (Saharan dust, Blue and White Nile sediments, Alexandria Bay crustal sediments, cores CII and CV) along with sediment isotope signatures from Maryut Lagoon (total bulk and leached fractions) and the Canopic branch of the Nile (bulk total fraction). A1 is the mixing line of peculiar non-crustal leached and bulk fractions from the Maryut sediment sequence. The Canopic Branch sequence is shown below 670 cm and above 600 cm.

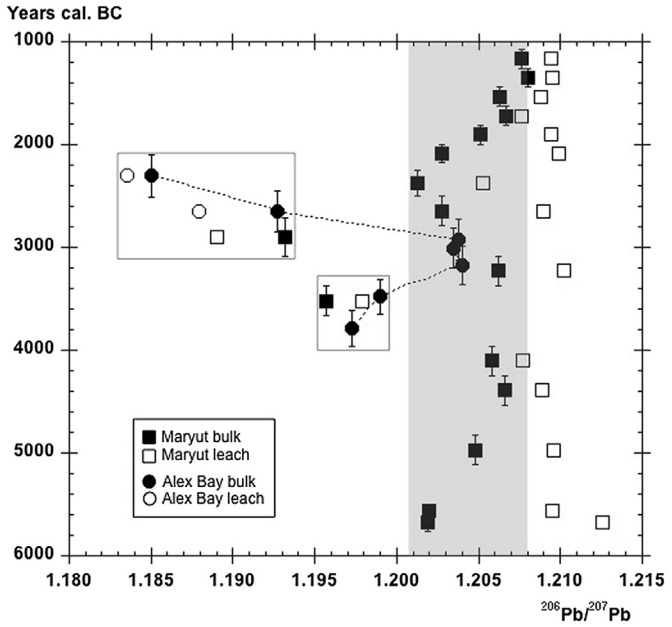

Fig. 3. Profile of $^{206}Pb/^{207}Pb$ ratios vs. time (calculated 14C age corrected) in Maryut Lagoon (M12) and Alexandria Bay (core CII). Maryut Lagoon leached (open squares) and total bulk (closed squares) fractions are presented with bulk total fraction from Alexandria Bay sediments (closed circles). The grey area corresponds to the commonly observed crustal imprint in Alexandria Bay and bulk Maryut sediment sequences.

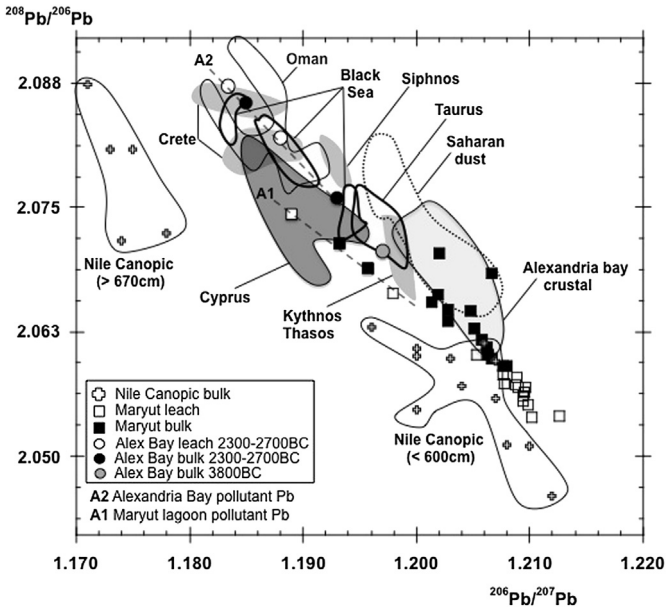

Fig. 5. Pb isotopic plot showing Cu ore imprints of significance to explain isotopic anomalies observed in Maryut Lagoon (A1 mixing line) and Alexandria Bay (A2 mixing line) bulk and leached sediment sequences. The "Oman" field relates to the south-eastern Arabian Cu source imprints.

5. Results and discussion

5.1. Geochemical imprints of early human activities

Lead concentrations in the Maryut Lagoon core M12 vary from 5.3 to 13.5 ppm (Table 1), with a mean of 9.5 ± 2.5 ppm that is consistent with non-contaminated crustal sediments (McLennan, 1995; Véron et al., 2006). A more detailed examination of this profile reveals a shift around 2897 B.C. and 3520 B.C. with a mean concentration (12.8 ± 0.53 ppm) that is statistically different (*t*-test $p < 0.001$) from the mean Pb background concentration (8.7 ± 2.1 ppm). Calculated Enrichment Factors (EF) for these samples also show a slight enrichment in these layers (1.5) in contrast to the rest of the core (1.1 ± 0.2) (Table 1). These enrichments corroborate the possible occurrence of exogenous contaminant Pb that is difficult to assert on the basis of concentration alone due to possible mineralogical variations.

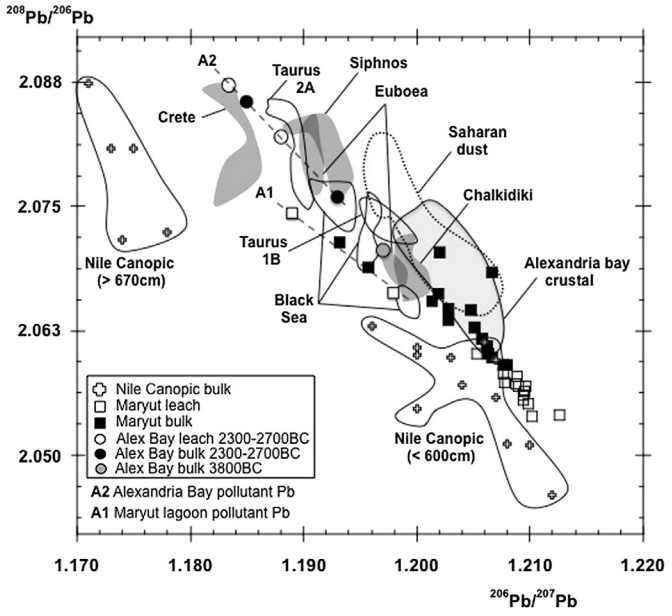

Fig. 4. Pb isotopic plot showing Pb ore imprints of significance to explain isotopic anomalies observed in the Maryut Lagoon (A1 mixing line) and Alexandria Bay (A2 mixing line) bulk and leached sediment sequences.

A well-defined geochemical background is needed to substantiate human activities from sediment records using Pb isotopic imprints. In order to characterize this background for the Maryut Lagoon, we have considered Pb isotope ratios of Holocene non-contaminated sediments from Alexandria Bay (Véron et al., 2006) along with imprints of Saharan dust (Hamelin et al., 1989; Abouchami and Zabel, 2003; Grousset and Biscaye, 2005; Kylander et al., 2010) and sediments of the nearby Nile Canopic branch (Fig. 2). Alexandria Bay and Saharan dust signatures generally overlap and comprise most of the Maryut bulk sediment isotopic ratios measured between 385 and 1015 cm (circa 1200–5700 B.C.) (mean $^{206}Pb/^{207}Pb = 1.205 \pm 0.003$, Table 1), except for two data points that are significantly (*t*-test $p < 0.0001$) less radiogenic ($^{206}Pb/^{207}Pb = 1.193$ and 1.195 at 2897 B.C. and 3520 B.C. respectively; Table 1). The mean $^{206}Pb/^{207}Pb$ "background" ratio of Alexandria Bay (1.203 ± 0.002, Véron et al., 2006) is not significantly different from that of the Maryut Lagoon (1.205 ± 0.003), suggesting similar crustal contributions. Conversely, almost none of the leachings from Maryut sediments belong to this "natural" field. They are significantly more radiogenic with a mean $^{206}Pb/^{207}Pb$ ratio of 1.209 ± 0.001 when excluding two divergent values (1.189 and 1.198) that match the same isotopic deviations observed for Maryut bulk analyses at 2897 ± 187 B.C. and 3520 ± 145 B.C. (Fig. 2).

This discrepancy between leached and bulk fractions can be explained by another "natural" source associated with the Blue Nile sediment load which largely derives from young volcanic basalts deposited on the Ethiopian plateau since the Oligocene (Mohr and Zanettin, 1988; Pik et al., 1998). These magmatic outcrops are characterized by higher $^{206}Pb/^{207}Pb$ isotopic imprint (more

radiogenic) than the usual crustal signature (Marty et al., 1993; Pik et al., 1999) (Fig. 2). Pb within this young volcanic matrix is also more easily leached than from older, often metamorphic, crustal rocks and is more easily extracted by our weak acid leaching procedure (Kurtz et al., 2000; Palumbo et al., 2000; Little and Lee, 2010). The Maryut's leached fraction reflects this volcanic imprint, also observed in the Canopic branch sediments in layers above 600 cm (Fig. 2, Table 2).

The isotopic record below 670 cm in the Canopic core significantly diverges from that of the Blue Nile and displays less radiogenic isotopic signatures (Fig. 2, Table 2). This shift is consistent with the influence of the other well-known headwater of the Nile, the White Nile Basin, located within the Equatorial lake region of East-Central Africa that is mostly formed of Precambrian metamorphic rocks (Williams et al., 2003; Woodward et al., 2007). This ancient Nile craton is characterized by less radiogenic imprints (Harms et al., 1990; Stern and Kröner, 1993) that are consistent with the Canopic isotopic signatures below 670 cm (Fig. 2). This transient change in the Canopic core corroborates strontium isotope records observed in the Nile Delta circa 3000 B.C. (Stanley et al., 2003) and is caused by changes in the provenance of sediment loads from the While Nile to the Blue Nile headwaters.

Recent work has suggested that the Blue Nile contribution was enhanced during the early and late Holocene linked to higher spring insolation, while by contrast the White Nile contributed more material between 8 and 4 ka at times of high autumn insolation (Blanchet et al., 2013). Two ^{14}C dates and stratigraphic correlation (Stanley and Warne, 1993; Goodfriend and Stanley, 1996) suggest this shift is recorded circa 3000–3400 B.C. in the Canopic core (Table 2). Other chemical imprints document this variation in sediment delivery during the same period in the Nile Delta (Foucault and Stanley, 1989; Frihy and Komar, 1993; Krom et al., 1999, 2002; Goiran, 2001; Stanley et al., 2003) that was caused by changes in monsoon activity (Joussaume et al., 1999; Gasse, 2000; deMenocal et al., 2000) and subsequent modification in sediment flux from the Nile headwaters (Adamson et al., 1980; Ritchie et al., 1985; Conway and Hulme, 1996; Williams et al., 2000; Woodward et al., 2007; Marriner et al., 2012a,b, 2013).

Variations in $^{206}Pb/^{207}Pb$ isotopic ratios for bulk and leached sediment fractions of the Maryut Lagoon are reported in Fig. 3. The previously explained difference in radiogenic character between Maryut bulk and leached fractions is well attested. The leached and bulk imprints at 2897 B.C. and 3520 B.C. are evident, outside the mean crustal imprint from the Maryut's bulk fraction (see shaded area in Fig. 3; 1.205 ± 0.003). These differences corroborate the isotopic anomalies from Alexandria Bay at 2300–2650 (±200) B.C. (mean $^{206}Pb/^{207}Pb = 1.189 \pm 0.004$) and 3500–3800 (±170) B.C. (mean $^{206}Pb/^{207}Pb = 1.198 \pm 0.001$) (Véron et al., 2006; Fig. 3) that are not well defined using concentration alone (Véron et al., 2006).

5.2. Origin of contaminant Pb

Isotopic anomalies from Alexandria Bay and Maryut Lagoon sediments are compared to Pb isotopic imprints from Pb and Cu ores from the literature (Figs. 4 and 5) (see references in: Barnes et al., 1974; Chamberlain and Gale, 1980; Gale et al., 1981, 1985, 1988, 1997; Stos-Gale and Gale, 1981, 2006; Spooner and Gale, 1982; Pernicka et al., 1984, 1990; Stos-Gale et al., 1984, 1986, 1996, 1997; Gale and Stos-Gale, 1985a, 1985b, 1986; Seeliger et al., 1985; Vavelidis et al., 1985; Wagner et al., 1985, 1986, 2003; Hamelin et al., 1988; Philip, 1991; Yener et al., 1991; Hauptmann, 1992; Sayre et al., 1992, 2001; Hirao et al., 1995; Scaife, 1997; Gale, 1999; Philip et al., 2003; Cattin, 2008; Begemann et al., 2010; Véron et al., 2012). Ore sources from the western Mediterranean (including Sardinia and Spain) are not shown since it does not fit any of the Pb imprints we have measured in Alexandria sediments prior to the 2nd millennium B.C. suggesting no metal exchange between these regions and Alexandria during the Early Bronze Age.

While $^{206}Pb/^{207}Pb$ anomalies from Maryut Lagoon (at 2897 ± 187 B.C. and 3520 ± 154 B.C.) fit the corresponding $^{206}Pb/^{207}Pb$ deviations observed in Alexandria Bay at 2300–2650 (±200) B.C. and 3500–3800 (±170) B.C. (Fig. 3), isotopic anomalies from both locations are characterized by two different mixing lines (A1 and A2 for Maryut Lagoon and Alexandria Bay respectively) in a $^{208}Pb/^{206}Pb$ vs. $^{206}Pb/^{207}Pb$ plot (Figs. 4 and 5). Therefore we infer different geographic origins for Pb accumulated in the marine and lacustrine basins. The mixing line A1 has been established using the bulk and leached isotopic anomalies observed at 2897 ± 187 B.C. and 3520 ± 154 B.C. in Maryut Lagoon. When considering Cu and Pb ore imprints, the most likely geographic sources are Cu ores from Cyprus and, to a lesser extent, Cu and Pb ores from Turkey (the Black Sea region and the Taurus Mountains) for A1 (Figs. 4 and 5). The mixing line A2 has been defined using bulk and leached fractions measured at 2300–2650 (±200) B.C. in Alexandria Bay (Figs. 4 and 5). The Alexandria Bay's imprint recorded at 3500–3800 (±170) B.C. may also belong to the mixing line A2. The least radiogenic signature from Alexandria Bay ($^{206}Pb/^{207}Pb = 1.1835$, see mixing line A2 in Figs. 4 and 5) shows the highest bulk EF (=5.3; Véron et al., 2006) of all the data presented in this study, suggesting that 88% of the Pb in this bulk fraction is exogenous. If we consider the bulk $^{206}Pb/^{207}Pb$ ratio for this sample (=1.185, Fig. 3; Véron et al., 2006) and the Alexandria Bay mean crustal background imprint (1.203 ± 0.002), one can calculate an "anthropogenic imprint" (A) as follows:

$$0.88(A) + 0.12(1.203) = 1.185$$

$$(A) = 1.1825$$

This calculated $^{206}Pb/^{207}Pb$ ratio ($A = 1.1825$) is within 0.1% of the measured leached $^{206}Pb/^{207}Pb$ ratio of 1.1835. These two values agree since they are within the lowest isotopic variations that can be generally discussed in sediments (Chow et al., 1973; Ferrand et al., 1999; Miralles et al., 2006; Angelidis et al., 2011) and therefore constitute the lesser radiogenic end-member for anthropogenic ore imprints deposited in Alexandria during the Bronze Age. This end-member matches Pb ore signatures from Crete (Fig. 4) and Cu ores from Crete and Turkey (southern Black Sea region) (Fig. 5). The other leached fraction fits the same geographical Cu ore sources and the more peculiar Cu ore signature from Southeastern Arabia in the Persian and Oman Gulfs (Fig. 5).

6. Conclusions

Are our geochemical findings from Alexandria consistent with known smelting activities from Crete, Cyprus, Turkey and Southeastern Arabia during the 4th and 3rd millennia B.C.? Do they provide archaeological evidence that support trading between these regions and the Nile Delta?

Our geochemical findings do not support the Levant as a source for metal imports to Alexandria in contrast to previous archaeological findings from the eastern delta but rather suggest far-reaching trade with Crete, Cyprus, Turkey and possibly the Arabian peninsula, along the Oman and Persian Gulfs. Cu metallurgy was clearly established during the 3rd millennium in (i) Chrysokamino (Crete) during the Aegean Early Bronze Age (2900–2000 B.C.) (Betancourt et al., 1999; Pryce et al., 2007), (ii) Ambelikou, Enkomi, Kition, Apliki (Cyprus) during the Philia Culture (around 2400 B.C.), (iii) the Taurus Mountains and the Black Sea region (Muhly, 1973, 1976; Wertime, 1973; Seeliger et al., 1985;

Wagner et al., 1986; Yener et al., 1989, 1991; Hirao et al., 1995), and (iv) the Gulf of Oman, during the Hafit (3200–2600 B.C.) and Umm an Nr (2600–2000 B.C.) cultural periods (Hastings et al., 1975; Weisgerber, 1978, 1981; Hauptmann, 1985; Nissen, 1986; Hauptmann et al., 1988; Weeks, 2003; Cleuziou and Tosi, 2007). Metal trading existed between Crete, Laurion, the Aegean Cyclades and Turkey during this period (Stech-Wheeler et al., 1975; Gale and Stos-Gale, 1986; Stos-Gale et al., 1997; Gale, 1999; Web et al., 2006; Stos-Gale and Gale, 2006, 2010). Large scale Cu extraction was conducted in the Sultanate of Oman and the United Arab Emirates in southeastern Arabia during the Bronze Age with established metal export to Mesopotamia, Iran and possibly the Southern Levant (Peake, 1928; Desch, 1929; Potts, 1990, 1993; Weeks, 1999, 2003; Prange, 2001; Begemann et al., 2010). In spite of isotopic similarities between imprints of Alexandria sediment and southeastern Arabia Cu ores, imports from the Oman and Persian Gulf regions to the Nile Delta seem quite unlikely considering the proximity of other sources (such as Turkey and Cyprus) to Alexandria. Furthermore, Cu smelting is attested in Turkey (at Catal Huyuk, Ergani, and the Taurus Mountains) and Cyprus prior to the 3rd millennium B.C. (Braidwood, 1967; Mellaart, 1967; Wertime, 1973; Rapp, 1983, 1988; Yener et al., 1989) suggesting that these two regions were the most probable source for metal imports into Alexandria during the eastern Mediterranean Early Bronze Age period.

However no archaeological findings yet corroborate metal imports from any of these source regions into the western Nile Delta. Temporal changes in Pb concentrations and isotopic compositions measured in sediments from Maryut Lagoon and Alexandria Bay sediments constitute the first evidence of metal use on the Western margin of the Nile Delta during the Predynastic period at 3800–3500 B.C., through to the Early dynastic and Old Egyptian Kingdom at 2900–2300 B.C., suggesting human settlements and potential Cu-ore trading in Alexandria more than 3000 years prior to the arrival of Alexander the Great.

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