



**Miocene-Holocene paleoenvironmental changes
in the Tinto River estuary (SW Spain)
evidenced by sedimentology, geochemistry and fauna**

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Abstract: This paper investigates the paleoenvironmental evolution of a long core extracted in a small cove located in the Tinto-Odiel estuary (SW Spain). The inferred reconstruction is supported by sedimentological, geochemical, paleontological data and dating. Seven phases have been identified, with the transit from Late Neogene marine environments to a subrecent freshwater pond and a final anthropic filling. On the basis of these data, this area was flooded during the maximum of the MIS-1 transgression (6.5-5.2 cal. kyr BP), with the temporary presence of a subtidal channel with phanerogam meadows. During this evolution, three geochemical peaks have been detected, which correspond to 1) the first evidence of mining activities (~4.5 cal. kyr BP), 2) an interval of intensive mining (1850-1960) and 3) an industrial period (1966-1985), affected by the dumping of highly polluting waste into this estuary.

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**Keywords:**

- texture;
- historical pollution;
- paleontological record;
- shelf-marsh evolution;
- MIS-1 transgression;
- SW Iberian Peninsula

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Résumé : *Mise en évidence de changements paléoenvironnementaux au Miocène-Holocène dans l'estuaire du Rio Tinto (Sud-Ouest de l'Espagne) par des données sédimentologiques, géochimiques et faunistiques.*- Cet article étudie l'évolution paléoenvironnementale des couches d'un carottage d'une vingtaine de mètres effectué dans une petite anse de l'estuaire de Tinto-Odiel (Sud-Ouest de l'Espagne). La reconstitution des environnement est étayée par des données sédimentologiques, géochimiques et paléontologiques, ainsi que par des datations absolues. On y a distingué sept phases avec le passage des milieux marins du Néogène supérieur à un bassin d'eau douce sub-récent et un remplissage anthropique final. D'après ces données, cette zone a été ennoyée lors du maximum de la transgression MIS-1 (6500-5200 ans calibrés AP), avec la présence temporaire d'un chenal infralittoral avec des herbiers à phanérogames. Au cours de cette évolution, trois pics géochimiques ont été détectés ; ils correspondent successivement 1) aux premiers indices d'activités minières (~4500 ans calibrés AP), 2) à un épisode d'exploitation minière intensive (1850-1960) et 3) à une période industrielle (1966-1985) caractérisée par le déversement dans cet estuaire de déchets très polluants.

Mots-clefs :

- texture ;
- pollution historique ;
- registre paléontologique ;
- évolution de plate-forme à marais ;
- transgression MIS-1 ;
- Sud-Ouest de la Péninsule ibérique

1. Introduction

In this century, numerous investigations were focused the paleoenvironment evolution of coastal environments during the Pleistocene (SCIUTO, 2014; SCIUTO *et al.*, 2015) and the Holocene (ZAI-BI *et al.*, 2011; STEPHAN *et al.*, 2019; HARDAGE *et al.*, 2021). For this purpose, different proxies have been used, such as sedimentological analyses (RUIZ *et al.*, 2021; JIWARUNGRUEANKUL *et al.*, 2022), geomorphological mapping (MAYA *et al.*, 2017; MCSWEENEY *et al.*, 2017), the paleontological record (BAICHTAL *et al.*, 2021; PEREIRA *et al.*, 2021) or geophysical datasets (RONCHI *et al.*, 2018; TROBEC *et al.*, 2018), among others. This growing research is very useful to infer the paleogeographic reconstructions of estuaries (DESRUJELLES *et al.*, 2021), lagoons (MARKS *et al.*, 2020), deltas (TA *et al.*, 2021) or barrier island systems (SCHAUMANN *et al.*, 2021). In addition, they are essential to test sea-level changes during the Pleistocene and Holocene (GHANDOUR *et al.*, 2021). Molluscs, foraminifera and ostracods are some of the most used groups in these studies. The analysis of their key species or assemblages can identify ocean water cooling/warming (PEREIRA *et al.*, 2022), salinity variations (GILDEEVA *et al.*, 2021), sea-level changes (WILLIAMS *et al.*, 2021) or even high-energy events (MATAMALES-ANDREU, 2019).

The southwestern Atlantic coast of Spain is made up by large sandy beaches (>250 km long), only interrupted by the presence of estuarine mouths (Guadiana, Piedras, Tinto-Odiel, Guadalquivir, Guadalete). During the maximum of the MIS-1 transgression, approximately 6.5 cal. kyr BP (ZAZO *et al.*, 1994), these estuaries were flooded and a number of large bays and lagoons were formed on a Neogene substrate (DABRIO *et al.*, 2000; BOSKI *et al.*, 2002; POZO *et al.*, 2010). The late Holocene evolution of these coastal systems was characterized by the progressive clogging of these estuaries, due to the growth of sandy spits near their mouths and the appearance of large marshes, mudflats and islands in their innermost areas (RUIZ *et al.*, 2010; CÁCERES *et al.*, 2018; CAPORIZZO *et al.*, 2021).

This paper carries out a multidisciplinary analysis of a continuous sediment core extracted in the Tinto-Odiel estuary. The main objectives are: i) the definition and paleoenvironmental interpretation of its sedimentary facies; ii) the reconstruction of its paleoenvironmental evolution; and iii) the identification of macrofaunal and microfaunal assemblages as proxies of the MIS-1 transgression in this area.

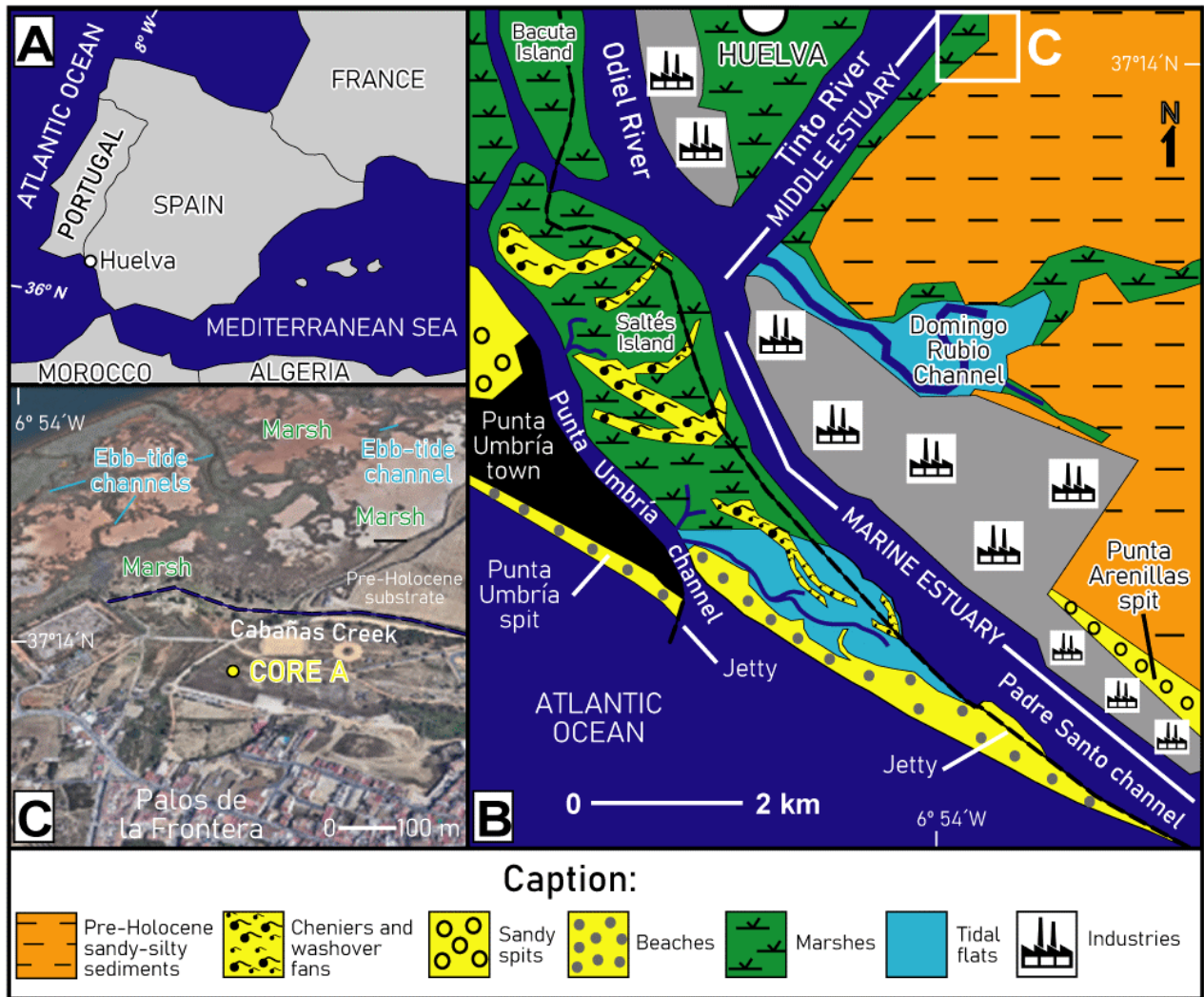


Figure 1: A-B. Location and geomorphological map of the Tinto River estuary, with location of La Fontanilla cove; C. La Fontanilla cove: main geomorphological features and location of core A.

Figure 1 : A-B. Carte de localisation et géomorphologique de l'estuaire du fleuve Tinto, avec la localisation de l'anse de La Fontanilla; C. Anse de La Fontanilla : Principales caractéristiques géomorphologiques et localisation de la carotte A.

2. Study area

The Tinto-Odiel River estuary is a 25-km-long incised bar-built system (FAIRBRIDGE, 1980) located on the southwestern Spanish coast (Fig. 1.A-B). This coastal environment is characterized by wide tidal flats, salt marshes and barrier islands, some of which are composed of a complex system of cheniers and washover fans (Fig. 1.B: Saltés Island; GONZÁLEZ-REGALADO *et al.*, 2019a). These inner areas are connected with the Atlantic Ocean through two main channels (Punta Umbría channel and Padre Santo channel) and they are partially protected by two sandy spits: i) the Punta Umbría spit, to the west; and ii) the Punta Arenillas spit, developed on the eastern margin and directly linked with Neogene cliffs, which include some inner small coves (e.g., Fig. 1.B-C: La Fontanilla cove) excavated by the fluvial dynamics during the Pleistocene. The Holocene estuarine sediments of this estuary were deposited on Mio-

cene-Pliocene siliciclastic sediments deposited in marine environments (CIVIS *et al.*, 1987). This Tertiary succession is composed of Miocene clays and silts (Gibráleon Clay Formation) and Pliocene fine sandy silts and sands (Huelva Sand Formation).

Fluvial discharge and tidal regime are the fundamental hydrodynamic processes in the Tinto-Odiel Estuary. The freshwater discharge of the Tinto River is subject to seasonal changes. The highest runoff occurs from December to February, while this discharge extremely scarce during the summer months (~1 hm³/month). The tidal regime is mesotidal (mean range: 2.15 m) and semidiurnal with a low diurnal amplitude (BORREGO *et al.*, 1995). Interaction between both fresh and marine waters delimits three sectors (BORREGO, 1992): i) Fluvial estuary, dominated by the riverine action; b) Middle Estuary, well mixed during spring tides and partially stratified during neap tides; and iii) Marine Estuary, a tide-dominated zone.

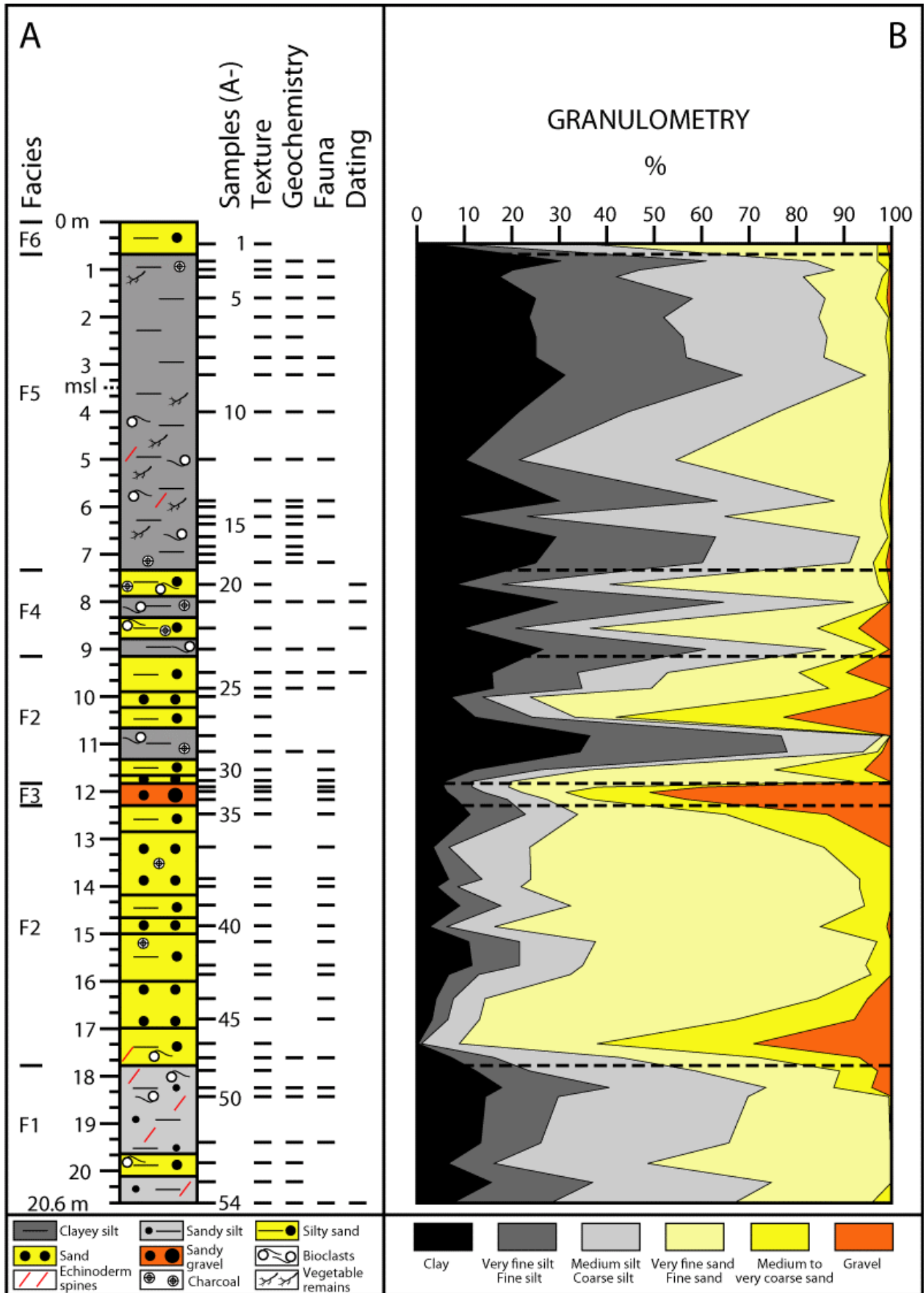


Figure 2: A. Core A: Log and multidisciplinary sampling; B. Core A: Grain size distribution.

Figure 2 : A. Carotte A : Colonne et échantillonnage multidisciplinaire ; B. Carotte A : Granulométrie.



The drainage basin of the Tinto River includes the Iberian Pyrite Belt, one of the most important mining-metallurgical provinces in the world. These deposits have been mined for at least 4,500 years, with two main periods: i) Roman period (2.1-1.7 kyr BP), with more than 20 Mt extracted (TORNOS, 2008); and ii) Recent period (1850-1960), with almost 300 Mt extracted in more than twenty mines. This historical contamination has been increased since 1966 due to the effluents coming from two industrial concentrations located in its margins (Fig. 1.B). The final results of these two pollution sources is the presence of heavily polluted bottom sediments by trace elements such as Cu (up to 3,000 mg kg⁻¹), Pb (up to 4,860 mg kg⁻¹) or Zn (up to 3,300 mg kg⁻¹) (RUIZ, 2001; CARRO *et al.*, 2011).

3. Material and methods

Core A (Fig. 2.A: 20.6 m length; 37°13'58"N-6°53'49"W; + 4 m m.s.l.) was extracted in 2013 near the transition between the alluvial deposits and the marshes located in the middle estuary of the Tinto River, near the town of Palos de la Frontera (Fig. 1.C). It was obtained by usual rotary drilling techniques with a continuous recovery of sediment and a barrel diameter of 11.6 mm. Fifty-four samples (2 cm thickness) have been collected for a multidisciplinary analysis, according to the visual study of the different sedimentary facies and their boundaries. These selected samples cover all sedimentary facies observed in core A and the main bioclastic levels. The visual lithological description has been complemented with the grain-size analysis of fifty samples (Fig. 2.A), selected according to the vertical distribution of sedimentary facies. Grain-size distribution was determined by wet sieving for the coarser fractions, which were sieved with pressured water and floated using sieves of 63 µm, 125 µm, 250 µm, 500 µm, 1000 µm, and 2000 µm diameter. Fractions lesser than 63 µm were analyzed with a particle counter (model Mastersizer-2000) at the CIDERTA center (University of Huelva, Spain). Na-hexametaphosphate has been used as a dispersing agent.

The geochemical contents of twenty-nine samples (Fig. 2.A) were performed and certified by MS Analytical, Langley (Canada). In a first step, an acid digestion (hydrochloric, nitric, perchloric and hydrofluoric acids) was applied to each sample. Concentrations of five major elements (in %; Al, Ca, Fe, Na, S), four trace metals (in mg kg⁻¹; As, Cu, Pb, Zn) and phosphorus (in mg kg⁻¹) were obtained by ICP Atomic Emission Spectrometry, with less than 5% variation between different replicate samples and a quality control based on more than 30 sample references (*e.g.*, OREAS 904). In these samples, detection limits are: Al

(0.01%), Ca (0.01%), Fe (0.01%), Na (0.01%), S (0.01%), As (0.02 mg kg⁻¹), Cu (0.2 mg kg⁻¹), Pb (0.5 mg kg⁻¹), Zn (2 mg kg⁻¹) and P (10 mg kg⁻¹).

Thirty-seven samples were selected for microfaunistic analysis (Fig. 2.A). In each sample, twenty grams of sediment were separated and levigated through a 125 µm mesh sieve. This sieve was selected for three reasons: a) this sieve retains practically all of the malacofauna, for whose study sieves with a larger mesh diameter are usually used (> 250 µm mesh diameter in most cases; ALEXANDROWICZ, 2017; CRISTINI & DE FRANCESCO, 2017); b) the benthic foraminiferal assemblages picked from this fraction provide useful information on prevailing environmental conditions and it is useful for an overview of paleoenvironmental changes in coastal settings (LO GIUDICE CAPELLI & AUSTIN, 2019), two of the main objectives of this paper; and c) this sieve is adequate for screening of the ostracod fauna and prevent the presence of juveniles (stages 1-4) of these microcrustaceans, with important difficulties in identifying them (DANIELOPOL *et al.*, 2002).

The residue was dried in an oven at a constant temperature of 40°C for a period of not less than one day. The microfaunal content (foraminifera and ostracods) was also extracted and determined. Foraminifera were classified according to LOEBLICH & TAPPAN (1987), MILKER & SCHMIEDL (2012), and the World Record of Marine Species (WoRMS). Ostracoda were classified according to RUIZ *et al.* (1997, 2000). This micropaleontological record has been compared with: (i) the assemblages of both groups present in the Neogene formations found in the core A vicinity (GONZÁLEZ-REGALADO & RUIZ, 1996, RUIZ & GONZÁLEZ-REGALADO, 1996), in order to identify reworked specimens; and (ii) the distribution of both groups in the estuaries of southwestern Spain (RUIZ *et al.*, 2000; GONZÁLEZ-REGALADO *et al.*, 2001), a database to recognize paleoenvironmental changes or anthropogenic impacts in this core. These studies have analyzed the foraminiferal and ostracod assemblages of the main sedimentary facies of these estuaries (high salt marsh, low salt marsh, ebb-tide channel, main channel, tidal plain, beach).

Five dates were produced at the National Center of Accelerators (CNA, Seville, Spain), with a subsequent calibration using CALIB version 8.2 and the reservoir effect correction (-108 ± 31 ¹⁴C yr) calculated by MARTINS & SOARES (2013) for this area. The final results correspond to calibrated ages using 2σ intervals. These calibrated ages have been completed with the comparison between the vertical geochemical variations and the main periods of mining extraction and industrial discharges (see Study Area), in order to obtain additional age references.

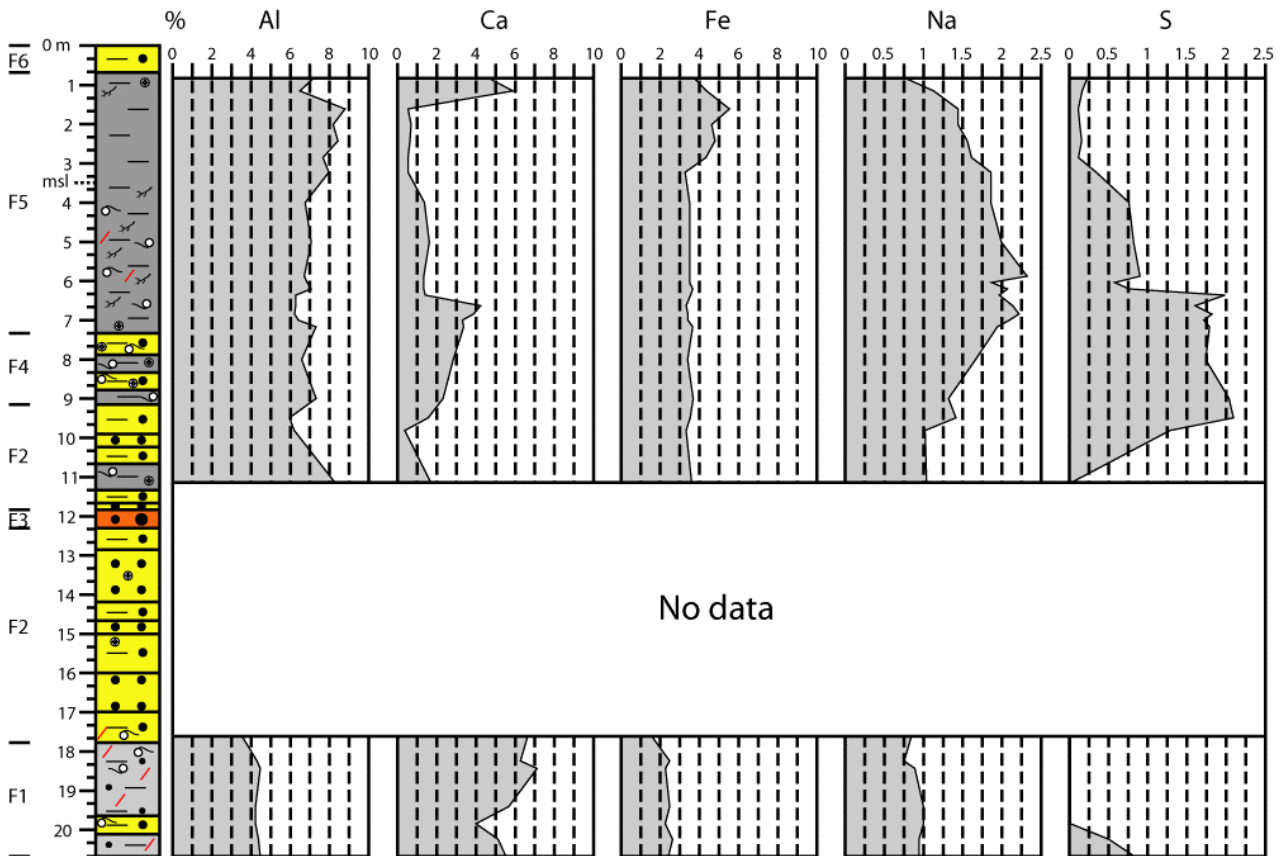


Figure 3: Down-core variations of major elements in core A.
Figure 3 : Variations des principaux éléments de la carotte A.

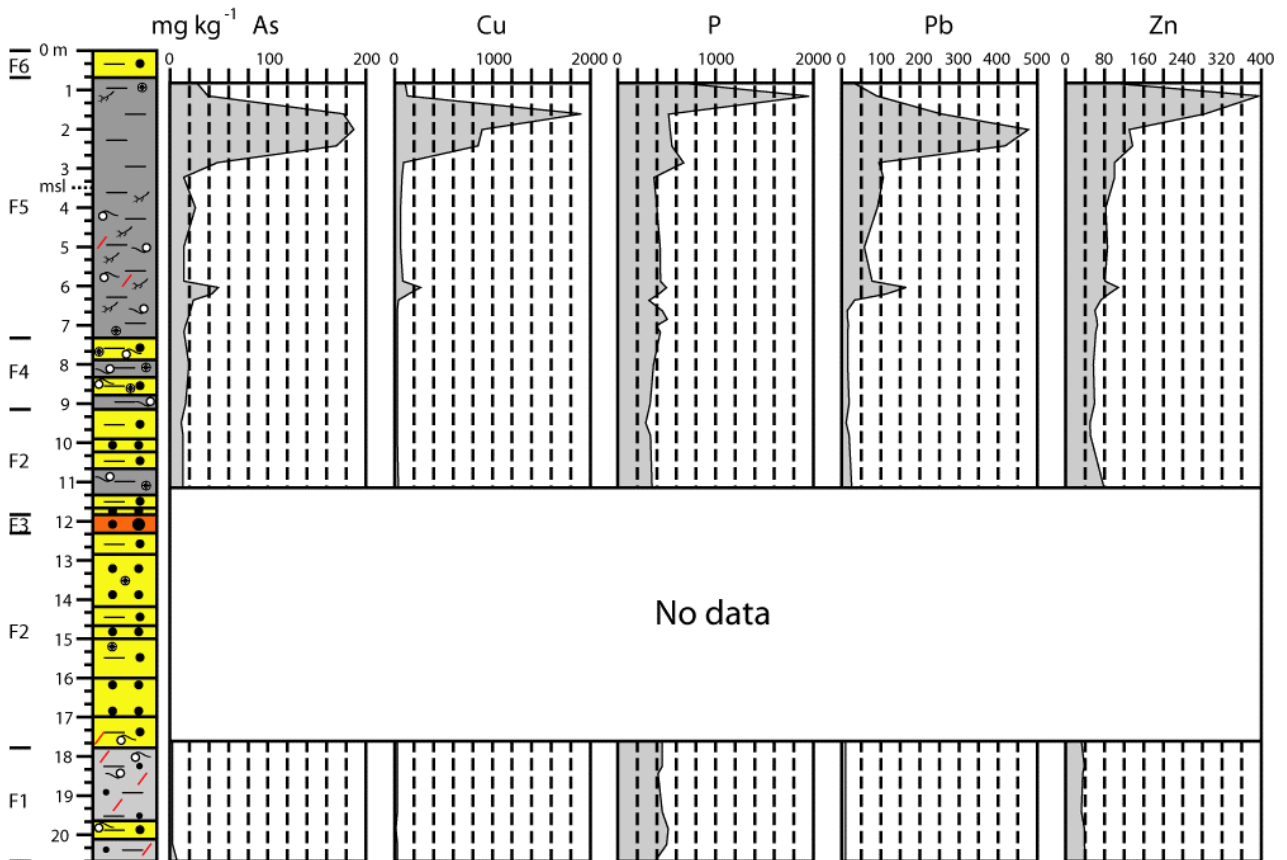


Figure 4: Down-core variations of trace elements in core A.
Figure 4 : Variations des éléments traces dans la carotte A.



4. Results

4.1. Sedimentary facies

Six main sedimentary facies have been distinguished within core A (Fig. 2.A). The lowermost 2.8 m of this core consist of grayish to greenish massive sandy silts (F1; MUNSELL scale: 10Y 7/2 to 7/6) with intercalations of silty sands (MUNSELL scale: 10Y 9/8). Macrofauna is very scarce, with rare fragments of the bivalve *Parvamussium cristatellum* (DAUTZENBERG & BAVAY, 1912) and isolated remains of scaphopods. The upper boundary of F1 (Fig. 2.A: 17.8 m depth) is characterized by an erosional discontinuity, which separates it from the overlying brownish silty sands (F2; 17.8-12.3 m depth and 11.7-9.1 m depth; MUNSELL scale: 10Y 3/0) with some muddy intercalations (11.3-10.7 m depth). F2 presents frequent quartzite clasts (up to 2 mm diameter) and some charcoal-rich layers, while no macrofaunal remains have been observed. F3 (12.3-11.8 m depth) has an erosive base and it is made up of yellow to whitish quartzite sandy gravels (MUNSELL scale: 5Y 8/8) within a silty-clayey matrix (gravel: 55%; sand: 25%; silt+clay: 20%). Macrofauna is absent in this facies.

F4 (9.1-7.3 m depth) consists of bioclastic greenish silty sands and sandy silts (MUNSELL scale: 10Y 3/2) with important percentages of clays (up to 30%). This facies includes frequent quartzite clasts and fragments of charcoal. Macrofauna is abundant, with numerous specimens of the gastropods *Peringia ulvae* (PENNANT, 1877) and *Bittium reticulatum* (MENDES da COSTA, 1778). The overlying clayey silts of F5 (7.3-0.7 m depth; MUNSELL scale: 5Y 8/4) can be divided into two subfacies: i) subfacies F5a (7.3-4 m depth), characterized by numerous roots and frequent specimens of *Peringia ulvae*; and ii) subfacies F5b (4-0.7 m depth), with very massive sediments, higher silty percentages and absence of macrofaunal remains (Fig. 2.B). The uppermost 0.7 m of core A are mainly composed of azoic silty sands (F6; sand: 56%; MUNSELL scale: 10YR 7/10).

4.2. Geochemical evolution

4.2.1. Major elements

F1 is characterized by high percentages of Ca (Fig. 3: 4-7%), as well as low to very low contents of Al (3.5-4.5%), Fe (1.5-2.6%), Na (0.75-1%) and S (0.04-0.82%). These last four elements increase in F2 (Al: 6-8.2%; Fe: 3.2-3.7%; Na: 1-1.4%; S: up to 2.1%), while Ca decreases noticeably (0.4-1.7%). Transition from F2 to F4 is marked by a progressive increase in Ca (up to 3.3%) and Na (up to 1.9%).

Calcium decreases upward within F5a from more than 4% near its base to less than 1.4% near the limit with F5b (4 m depth). This decrease is very abrupt for S (2% to 0.5%) and milder for Na (2.2% to 1.8%). The F5a-F5b boundary is clearly delimited by a significant increase in Fe (3.5% to 4.7-5.5%) and Al (up to 8.8%), while S decreases in F5b (<0.2%). Except for sulphur, all other major elements decrease near the boundary between F5b and F6.

4.2.2. Trace elements

The vertical variations of the five elements show similar trends (Fig. 4). The lowest values of As (< 20 mg kg⁻¹), Cu (< 20 mg kg⁻¹), P (< 500 mg kg⁻¹), Pb (< 20 mg kg⁻¹) and Zn (< 70 mg kg⁻¹ in most samples) were measured in F1, F2 (except in some muddy intercalations) and F4, although these contents increase upward. A first peak of four trace elements is observed within F5a at 6.2-6 m depth (As: 42-49 mg kg⁻¹; Cu: 155-252 mg kg⁻¹; Pb: 117-164 mg kg⁻¹; Zn: 89-110 mg kg⁻¹). Concentrations of these elements decrease remarkably in the remaining samples of F5a, although the last two elements slowly increase to the limit between F5a and F5b.

The highest trace element contents were measured within F5b, with two peaks. The second geochemical peak of core A (Fig. 4: 2.4-1.6 m depth) is characterized by the highest concentrations of As (170-187 mg kg⁻¹), Cu (853-1893 mg kg⁻¹) and Pb (253-475 mg kg⁻¹), as well as by high values of Zn (131-285 mg kg⁻¹). A third peak (1.2 m depth) is located near the F5a-F5b boundary, with the highest concentrations of P (up to 1940 mg kg⁻¹) and Zn (up to 394 mg kg⁻¹). In this boundary, all these contents decrease (As: 27 mg kg⁻¹; Cu: 97 mg kg⁻¹; P: 843 mg kg⁻¹; Pb: 35 mg kg⁻¹; 85 mg kg⁻¹).

4.3. Microfaunal record

4.3.1. General remarks

The microfossil distribution can be consulted in Table 1. Microfauna is abundant in F1, with numerous individuals of benthic foraminifera and frequent planktonic species of this group (mainly *Globigerina bulloides* ORBIGNY, 1826, *Globigerinoides obliquus* BOLLI, 1957, and *Orbulina universa* ORBIGNY, 1839). These planktonic species are not found in the remaining facies. In addition, this facies presents numerous spines of echinoderms and some undifferentiated fragments of ostracods. Conversely, F2 is almost devoid of microfauna, which has only been observed near its lower (sample A-47; 17.6 m depth) and upper (sample A-24; 9.5 m depth) limits. No microfossils have been found in F3 and most samples of F5b, while benthic foraminifera are well represented in F4 and, to a lesser extent, in F5a.



Table 1: Distribution of benthic foraminifera and ostracoda in core A.

Tableau 1 : Distribution des foraminifères benthiques et des ostracodes dans la carotte A.

FORAMINIFERA																	
SPECIES/SAMPLES	A-2	A-3	A-5	A-10	A-11	A-12	A-14	A-19	A-20	A-21	A-23	A-24	A-47	A-49	A_50	A-51	A-54
<i>Ammonia beccarii</i> (LINNAEUS, 1758)									4	9				2			
<i>Ammonia inflata</i> (SEGUENZA, 1862)												4			11	12	4
<i>Ammonia tepida</i> (CUSHMAN, 1926)				8	7	10	20	17	35	25	22	4					
<i>Bannerella gibbosa</i> (ORBIGNY, 1826)														3	3		
<i>Bolivina catanensis</i> (SEGUENZA, 1862)															3	5	
<i>Bolivina spathulata</i> (WILLIAMSON, 1858)															5	10	
<i>Bulimina elongata</i> ORBIGNY, 1846									1						11	24	2
<i>Cassidulina carinata</i> SILVESTRI, 1896															11	3	
<i>Cibicides pseudoungerianus</i> (CUSHMAN, 1922)																1	
<i>Criboelphidium williamsonii</i> (HAYNES, 1973)									1								
<i>Elphidium advenum</i> (CUSHMAN, 1922)								1	1	1							
<i>Elphidium macellum</i> (FICHTEL & MOLL, 1798)									2								
<i>Fursenkoina acuta</i> (CZJZEK, 1848)															14	3	
<i>Globobulimina auriculata</i> (BAILEY, 1894)													1		5	3	
<i>Hansenisca soldanii</i> (ORBIGNY, 1826)												14				2	
<i>Heterolepa bellincionii</i> (GIANNINI & TAVANI, 1960)													1		7	6	1
<i>Haynesina depressula</i> (WALKER & JACOB, 1798)				2	3	8	11	10	29	16	5	3					
<i>Haynesina germanica</i> (EHRENBERG, 1840)				2	3	4	7	3	30	24	3	1					
<i>Hopkinsina bononiensis</i> (FORNASINI, 1888)																2	
<i>Lenticulina calcar</i> (LINNAEUS, 1758)													1	12	4	5	1
<i>Lenticulina cultrata</i> (MONTFORT, 1808)														17	4	3	4
<i>Lenticulina curvisepta</i> (SEGUENZA, 1880)														1			
<i>Lenticulina inornata</i> (ORBIGNY, 1846)														4	4		1
<i>Marginulinopsis costata</i> (BATSCH, 1791)													1	9	10	3	
<i>Melonis pompilioides</i> (FICHTEL & MOLL, 1798)																2	
<i>Nonion faba</i> (FICHTEL & MOLL, 1798)		1										3			8	21	2
<i>Orthomorphina tenuicostata</i> (COSTA, 1853)			1										1				
<i>Pullenia bulloides</i> (ORBIGNY, 1846)															7	2	
<i>Quinqueloculina seminulum</i> (LINNAEUS, 1758)			1														
<i>Sahulina neorugosa</i> (THALMANN, 1950)															7	3	
<i>Trochammina inflata</i> (MONTAGU, 1808)										3	3						
<i>Vaginulina striatissima</i> SCHRODT, 1890															3	1	
<i>Valvulineria bradyana</i> (FORNASINI, 1900)																3	
NUMBER OF INDIVIDUALS/20 g	3	12	13	22	38	31	98	71	42	28	9	48	121	110	15		
NUMBER OF SPECIES	3	3	3	3	3	4	6	6	5	6	6	7	19	18	7		
OSTRACODA																	
SPECIES/SAMPLES	A-2	A-3	A-5	A-10	A-11	A-12	A-14	A-19	A-20	A-21	A-23	A-24	A-47	A-49	A_50	A-51	A-54
<i>Cyprideis torosa</i> (JONES, 1850)			1														
<i>Cypridopsis vidua</i> (O.F. MULLER, 1776)	17																
<i>Cytherois fischeri</i> (SARS, 1866)									1	3							
<i>Ilyocypris gibba</i> (RAMDOHR, 1808)	1																
<i>Leptocythere castanea</i> (SARS, 1866)										3							
<i>Loxoconcha elliptica</i> BRADY, 1867				1					3	61							
NUMBER OF INDIVIDUALS/20 g	18	1	1						4	67							
NUMBER OF SPECIES	2	1	1						2	3							
FACIES	F5b		F5a					F4		F2		F1					

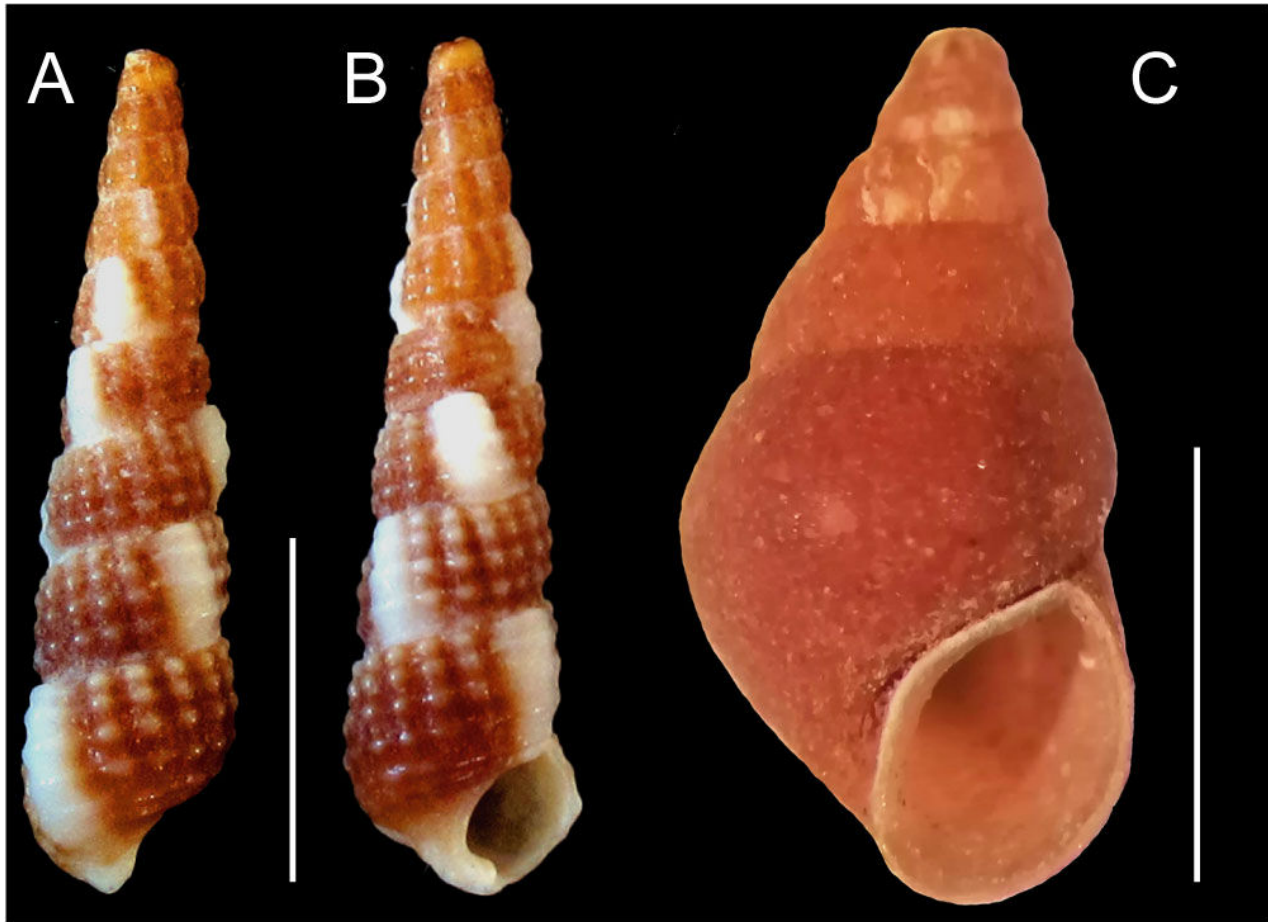


Figure 5: Gastropods. **A-B:** *Bittium reticulatum* (MENDES da COSTA, 1778); **C:** *Peringia ulvae* (PENNANT, 1877). Scale: 1 cm.
Figure 5 : Gastropodes. **A-B :** *Bittium reticulatum* (MENDES da COSTA, 1778) ; **C :** *Peringia ulvae* (PENNANT, 1877). Échelle : 1 cm.

4.3.2. Benthic foraminifera

A total of 661 shells of benthic foraminifera belonging to 33 species were extracted from fifteen samples, with more than 76% of the individuals concentrated in F1 and F4 (Table 1). The foraminiferal record of F1 (48-121 individuals/20 g; 7-19 species/sample) is dominated by *Ammonia inflata* (SEGUENZA, 1862), *Bulimina elongata* ORBIGNY, 1846, *Marginulinopsis costata* (BATSCH, 1791), *Nonion faba* (FICHTEL & MOLL, 1798) and some species of *Lenticulina*. Some of them are also present in the basal sample of F2 (A-47; 17.6 m depth), together with scarce shells of other species also observed in F1, such as *Globobulimina auriculata* (BAILEY, 1894) or *Heterolepa bellincionii* (GIANNINI & TAVANI, 1960). In the remaining samples of this facies, benthic foraminifera are only observed near the upper boundary with F4 (sample A-24; 9.5 m depth), with very frequent shells of *Hansenisca soldanii* (ORBIGNY, 1826) and rare specimens of *Ammonia tepida* (CUSHMAN, 1926), *Haynesina* spp., *Nonion faba* (FICHTEL & MOLL, 1798) and *Trochammina inflata* (MONTAGU, 1808). These microorganisms are absent in F3.

The foraminiferal record of F4 (42-98 individuals/20 g; 5-6 species/sample) is mainly composed of *Ammonia tepida* (CUSHMAN, 1926) (22-35 individuals/20 g), *Haynesina depressula* (WALKER & JACOB, 1798) and *Haynesina germanica* (EHRENBERG, 1840), with an increasing number of individuals upward. These species are accompanied by *Ammonia beccarii* (LINNAEUS, 1758) and scarce elphidids. The three first species are the almost only benthic foraminifera observed in F5a (12-38 individuals/20 g; 3-4 species/sample), with a decreasing density towards the top of this subfacies. On the contrary, samples of F5b are characterized by the absence of these microorganisms and the presence of rare individuals of *Nonion faba* (FICHTEL & MOLL, 1798), *Ortomorphina tenuicostata* (COSTA, 1853) and *Quinqueloculina seminulum* (LINNAEUS, 1758).

4.3.3. Ostracoda

This group is represented by six species and 91 valves and carapaces, although no specimens were found in F2 and F3. This group is abundant in the lower-middle samples of F4 (9-8 m depth), with numerous adults and moults of *Loxoconcha*



elliptica BRADY, 1867, together with rare valves of *Cytherois fischeri* (SARS, 1866) and *Leptocythere castanea* (SARS, 1866). An isolated valve of *Loxocconcha elliptica* BRADY, 1867, was extracted in the uppermost sample of F5a, while *Cypridopsis vidua* (O.F. MÜLLER, 1776) is the main species of the uppermost sample of F5b (sample A-2; 17 individuals/20 g), together with isolated valves of *Ilyocypris gibba* (RAMDOHR, 1808) and *Cyprideis torosa* (JONES, 1850).

Table 2: Radiocarbon database of core A.
Tableau 2 : Datations de la carotte A.

Laboratory code	Sample material	δ 13C‰	14C age (yr BP)	Calibrated yr BP/ IntCal20/Marine20 (2σ)	Mean calibrated age (kyr BP)
CNA-4277	Organic matter	-26.7	6407 ± 32	5470–5325	5.4
CNA-4278	Organic matter	-26.2	6993 ± 33	5980–5785	5.88
CNA-4279	Shell	-0.4	5690 ± 30	6190–5990	6.09
CNA-4280	Organic matter	-28.0	7437 ± 33	6390–6240	6.31
CNA-4283	Shell	-1.2	Out of range	Out of range	Out of range

5. Discussion

5.1. Autoecology and biostratigraphy of the main species

5.1.1. Molluscs

F1 is characterized by fragmented valves of the bivalve *Parvamussium cristatellum* (DAUTZENBERG & BAVAY, 1912), a highly mobile, epifaunal species (DANISE *et al.*, 2010). It was very common [as *Amussium cristatum* (BRONN, 1827)] in the Miocene silty-clayey sediments of the Gibrleon Clay Formation (CIVIS *et al.*, 1987), deposited from slope to shelf paleoenvironments (GONZÁLEZ-REGALADO *et al.*, 2019c). It has been also found in Pliocene monospecific-paucispecific beds of southeastern Spain formed in mesotrophic mid-shelf settings (AGUIRRE, 2017), although it has been cited in Miocene and Pleistocene shallower paleoenvironments of Italy (BERNASCONI & ROBBA, 1993).

The gastropods *Peringia ulvae* (PENNANT, 1877) (Fig. 5.A-B) and *Bittium reticulatum* (MENDES DA COSTA, 1778) (Fig. 5.C) are very abundant in F4. The first species is frequent in the inner, silty-sandy mesolittoral areas of estuaries and marshes with low hydrodynamics and important fluvial inputs (GÓMEZ, 2017). It is specially adapted to these changing conditions and it crawl about on the surface of the mud then the burrow for several hours during low tide (NEWELL, 2009). This species has been mainly cited in Pleistocene to Holocene sediments around Europe (GOFAS *et al.*, 2001; DANUKALOVA *et al.*, 2015).

Both species coexist in mesolittoral phanerogam meadows or in its vicinity, since *Bittium reticulatum* (MENDES DA COSTA, 1778) feeds on the organic matter that covers the beam of the leaves and the soil of these meadows (BORJA, 1986; LUQUE & TEMPLADO, 2004). In Spain, this species has been found present in Miocene to Holocene sedi-

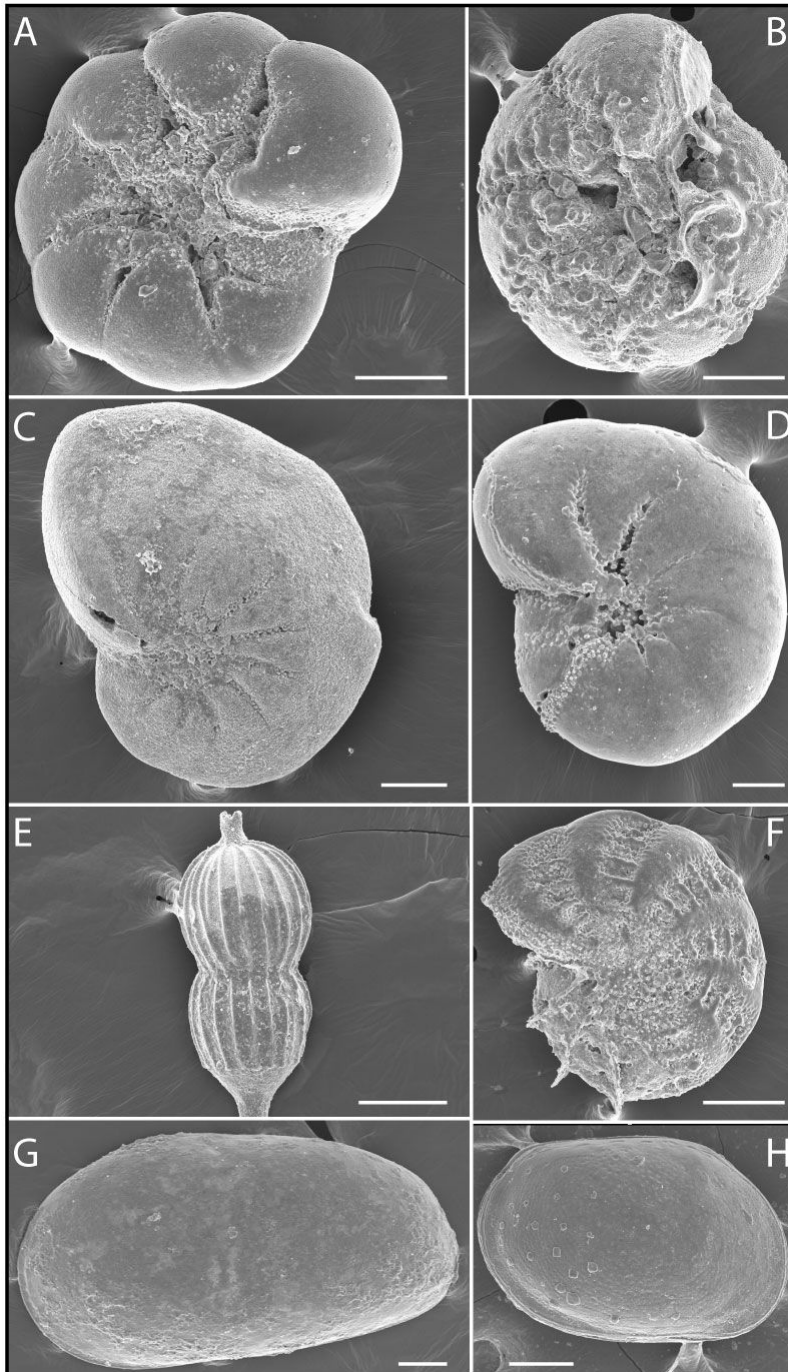
4.4. Dating

Five dates have been obtained from F1, F2 and F4 (Table 2). According to these data, a first approximation to the ages of the different facies can be made: i) F1 was deposited more than 50,000 years ago; ii) the ages of F2 and F3 are older than ~6.31 cal. kyr BP; iii) the bioclastic deposits of F4 were deposited between ~6.2 cal. kyr BP and ~5.4 cal. kyr BP; and iv) the ages of the uppermost facies (F5a, F5b and F6) are younger than 5.4 cal. kyr BP.

ments (GONZÁLEZ DELGADO, 1986; VÁZQUEZ *et al.*, 1990; GONZÁLEZ-REGALADO *et al.*, 2019b).

5.1.2. Benthic foraminifera

Ammonia inflata (SEGUENZA, 1862), *Bulimina elongata* ORBIGNY, 1846, *Marginulinopsis costata* (BATSCH, 1791) and *Nonion faba* (FICHEL & MOLL, 1798) (Fig. 6.C) are the most abundant species of F1. *Ammonia inflata* (SEGUENZA, 1862) is a temporary motile epifaunal species (morphotype B *sensu* LANGER, 1993) that prefers shallow marine (paleo-)environments (COLOM, 1974; MATEU-VICENS *et al.*, 2014). In southern Spain, it is common in Miocene silty-clayey sediments and Pliocene sandy silts (GONZÁLEZ-REGALADO, 1989; CORBÍ, 2010). *Bulimina elongata* ORBIGNY, 1846, and *Nonion faba* (FICHEL & MOLL, 1798) are currently found together in the outer inner shelf (12–40 m depth) of the southwestern Spanish-Portuguese littoral (MENDES *et al.*, 2004), although these species have been cited from deeper Miocene paleoenvironments (up to 80–100 m depth) of southeastern Germany (PIPPER & REICHENBAKER, 2009) and Morocco (TULBURE *et al.*, 2017) and recent sediments from the Alboran Sea (GARCÍA SANZ & USERA, 2015). *Bulimina elongata* ORBIGNY, 1846, is common in low-oxygen (paleo-)environments (cf. RÖGL & SPEZZAFERRI, 2003), while *Nonion faba* (FICHEL & MOLL, 1798) is considered intermediate infaunal and associated with eutrophic environments and high organic matter of low quality from terrestrial runoff (see GARCÍA GALLARDO, 2017, and references therein). *Marginulinopsis costata* (BATSCH, 1791) is an infaunal species frequently cited in circalittoral and slope (paleo-)environments under dysoxic conditions (CORBÍ, 2010; AMAKRANE *et al.*, 2016; GONZÁLEZ-REGALADO *et al.*, 2019b), mainly in soft bottoms enriched in organic matter (SCIUTO & BALDANZA, 2020). It has been cited in Late Oligocene to Recent sediments (*e.g.*, CARBONI & PALAGI, 1998).



◀ **Figure 6:** Microfauna. Foraminifera. **A:** *Ammonia tepida* (CUSHMAN, 1926) (ventral view); **B:** *Ammonia beccarii* (LINNAEUS, 1758) (ventral view); **C:** *Nonion faba* (FICHTEL & MOLL, 1798); **D:** *Haynesina germanica* (EHRENBERG, 1840); **E:** *Amphycorina scalaris* (BATSCH, 1791); **F:** *Elphidium advenum* (CUSHMAN, 1922); Ostracoda. **G:** Left valve of *Cyprideis torosa* (JONES, 1850); **H:** Right valve of *Loxoconcha elliptica* BRADY, 1867. Scale: 100 µm.

Figure 6 : Microfaune. Foraminifères. **A :** *Ammonia tepida* (CUSHMAN, 1926) (vue ventrale) ; **B :** *Ammonia beccarii* (LINNÉ, 1758) (vue ventrale) ; **C :** *Nonion faba* (FICHTEL & MOLL, 1798) ; **D :** *Haynesina germanica* (EHRENBERG, 1840) ; **E :** *Amphycorina scalaris* (BATSCH, 1791) ; **F :** *Elphidium advenum* (CUSHMAN, 1922) ; Ostracoda. **G :** valve gauche de *Cyprideis torosa* (JONES, 1850) ; **H :** valve droite de *Loxoconcha elliptica* BRADY, 1867. Échelle : 100 µm.

Ammonia tepida (CUSHMAN, 1926) (Fig. 6.A), *Haynesina depressula* (WALKER & JACOB, 1798) and *Haynesina germanica* (EHRENBERG, 1840) (Fig. 6.D) are well represented in both F4 and F5a. *Ammonia tepida* (CUSHMAN, 1926) is an endopelic species, living in intertidal mudflats of brackish environments, such as estuaries or lagoons (DEBENAY *et al.*, 1998; RUIZ *et al.*, 2012; WUKOVITS *et al.*, 2018; PASCUAL *et al.*, 2019). In southern Europe, this species was described in Miocene to Recent sediments (GONZÁLEZ DELGADO *et al.*, 1986; GONZÁLEZ-REGALADO, 1989; BOSSIO *et al.*, 2003-2004).

Haynesina depressula (WALKER & JACOB, 1798) is one of the most common paralic-shallow water marine species, which tolerates polyhaline waters and stressed environments (see AIELLO *et al.*, 2018, and references therein). It is well represented in marshes, intertidal mudflats or bays with freshwater inputs (CEARRETA, 1989; PASCUAL *et al.*, 2002; CALVO & LANGER, 2010). It was found in Middle Miocene sediments from eastern Spain (GARCÍA-SANZ *et al.*, 2019), as well as in Pliocene-Pleistocene sections from Italy (DI BELLA *et al.*, 2000) and Pliocene clays from Crete (DRINIA *et al.*, 2005).



Haynesina germanica (EHRENBERG, 1840) is a common, shallow-water, benthic foraminifer that has been reported from recent estuaries, bays, salt marshes, and mudflats from around the globe (see GOLDSTEIN & RICHARDSON, 2018, and references therein). These environments have large diurnal salinity variations (0-35‰), a temperature range of 0-32°C and depth from intertidal to 20 m (MURRAY, 2006). It has been collected in Tortonian to Holocene sediments from America, Europe and Asia (GIBSON, 1994; USERA *et al.*, 2000; MELIS *et al.*, 2015).

5.1.3. Ostracoda

Cypridopsis vidua (O.F. MÜLLER, 1776) was only found in the uppermost sample of F5b (0.8 m depth). It is a freshwater, phytophilous species (ROCA & DANIELOPOL, 1991), very frequent in ponds displaying high vegetation cover (MARTÍNEZ *et al.*, 2016b), springs (MARTÍNEZ *et al.*, 2016a), lakes and reservoirs (KULKOYLUOGLU, 2003), among other freshwater (paleo-)environments. In southern Europe, this species has been collected in fossil sediments from the Upper Miocene to the Holocene (BEKKALI *et al.*, 2003; RUIZ *et al.*, 2004a; SCIUTO *et al.*, 2015).

Cytherois fischeri (SARS, 1866), *Leptocythere castanea* (SARS, 1866) and *Loxococoncha elliptica* BRADY, 1867 (Fig. 6.H) are the only ostracod species extracted from F4. *Cytherois fischeri* (SARS, 1866) is an euryhaline species (ATHERSUCH *et al.*, 1990), frequent in silty sand flats, low marshes or lagoons (ZAIBI *et al.*, 2011; MARTÍNEZ *et al.*, 2013; ALTINSACLI *et al.*, 2015). In recent sediment of southwestern Spain, it was found from the middle estuaries to the innermost shelf (RUIZ *et al.*, 1997; MATTOS *et al.*, 2016). This species has been observed in Pleistocene and Holocene cores and sections from Spain, France and Greece (RUIZ *et al.*, 2007; DEVILLERS *et al.*, 2019; PAPADOPOULOU *et al.*, 2019).

Leptocythere castanea (SARS, 1866) is very common in inner estuarine and salt marsh environments (ATHERSUCH *et al.*, 1990), with periodic supply of seawater (MARTÍN-RUBIO *et al.*, 2006) and a highly variable salinity (1‰-32‰; PASCUAL *et al.*, 1999). This species has been found in Pleistocene and Holocene cores and sections from southern Spain (RUIZ *et al.*, 2002; TORRES *et al.*, 2003). *Loxococoncha elliptica* BRADY, 1867, is a euryhaline species, very abundant in lagoons, estuaries, marshes or ebb-tide channels from the Baltic sea to the Turkey (MARTÍN-RUBIO *et al.*, 2004; BASSLER-VEIT *et al.*, 2013; PINT & FRENZEL, 2017). This species has been cited in Pleistocene and Holocene sediments from Germany, Spain, Italy, Tunisia and Turkey (RUIZ *et al.*, 2004b; VIEHBERG *et al.*, 2008; SALIHOGLU *et al.*, 2018; CATANZARITI & DA PRATO, 2020).

5.2. Sedimentation rates, geochemical peaks and episodes of historical pollution

A first, high sedimentation rate (SR: 2.03 cm/yr) can be estimated for F4 between samples A-24 (9 m depth; ~6.09 cal. kyr BP) and A-20 (7.6 m depth; ~5.4 cal. kyr BP). If this SR is extrapolated to the total thickness of F4, this facies would have been deposited between ~6.09 cal. kyr BP and ~5.2 cal. kyr BP.

In the uppermost meters of core A, three geochemical peaks has been observed at 6.2-6 m depth, 2.4-1.6 m depth and 1.2 m depth, respectively (see section B.2. Trace elements). The first peak would correspond to the first evidence of mining activity in this region (~4.5 cal. kyr BP; ROTHENBERG & BLANCO FREIJERO, 1980), according to: i) the age of sample A-20 (7.6 m depth; ~5.4 cal. kyr BP), close to the F4-F5a boundary (7.3 m depth); and ii) the depth of this geochemical peak (~4.2 m depth) detected in other cores of the Tinto estuary (ARROYO *et al.*, 2021b). According to these data and the mean calibrated age of sample A-20 (7.6 m depth; ~5.4 cal. kyr BP), the estimated SR between ~5.4 cal. kyr BP and ~4.5 cal. kyr BP (6.2 m depth) was around 1.55 cm/year. This decreasing trend has been observed in other cores of this estuary after 6.5 cal. kyr BP until the present (LARIO *et al.*, 2002).

The second peak starts at 2.4 m depth and leads to a notable rise (even over an order of magnitude) in the concentrations of As, Cu and Pb, which remain very high in the following 0.8 m above (Fig. 4). These very high contents are attributed to the increase in intensive mining in the Iberian Pyrite Belt, especially from 1850 to 1980. In the middle estuary of the Tinto river, this peak has been observed in other nearby cores at 2 m depth (ARROYO *et al.*, 2021b). Consequently, a sedimentation rate (~0.84 cm/yr) can be estimated between ~4.5 cal. kyr BP (6.2 m depth) and 1850 (~2.4 m depth), which confirms the decreasing trend of SR mentioned above. According to this estimated SR, the calibrated age of the F5a-F5b boundary (4 m depth) is around 1.9 cal. kyr BP.

The third peak (1.2 m depth) is characterized by the highest concentrations of P and Zn. This peak would be due to the wastes dumped by the industrial concentrations located on the banks of the estuary (Fig. 1.B), especially from 1966 to 1985 (RUIZ *et al.*, 2004b). These concentrations include the largest complex in the European Union producing phosphoric acid, as well as Zn-rich pyrite processing plants, which explain the high contents of these elements. Since 1985, this zone is subject of a Corrective Plan for Control of Industrial Waste Disposal, with an improvement in environmental conditions that is reflected in the lower levels of contamination near the limit between F5b and F6 (Fig. 4).



5.3. Miocene-Holocene paleoenvironmental reconstruction of core A

The integrated analysis of core A (sedimentology, geochemistry, paleontology, dating) allows to separate seven phases in the paleoenvironmental evolution of core A (Fig. 7).

Phase 1 (F1; Miocene). The sedimentological and paleontological features of F1 are very similar to those observed in cores and surface sections that include the upper part of the Gibraleón Clay Formation. The main features of these sediments are: i) dominance of predominance of massive silts and sandy silts; ii) frequent presence of the bivalve *Parvamussium cristatellum* (DAUTZENBERG & BAVAY, 1912); iii) a foraminiferal assemblage dominated by *Ammonia* spp., *Bulimina elongata* ORBIGNY, 1846 and *Nonion faba* (FICHEL & MOLL, 1798), very frequent in middle-inner shelf (paleo-) environments; and iv) a Late Messinian age, according to their magnetostratigraphic chronology (CIVIS *et al.*, 1987; GARCÍA, 2002; PÉREZ-ASENSIO *et al.*, 2012; LARRASOÑA *et al.*, 2014).

Phase 2 (F2-F3; Late Pleistocene-6.1 cal. kyr BP). The F1-F2 boundary (17.8 m depth) is characterized by a clear unconformity, indicating a partial erosion of F1 as attested by the presence of F1 benthic foraminifera within the basal sample of F2 (Table 1). In this area, the boundary between the Gibraleón Clay Formation and the overlying Huelva Sand Formation was located around +7 m m.s.l. in surface sections (ARROYO *et al.*, 2021a) and consequently about 25 m of F1 were eroded during this new phase. It is characterized by alluvial sands (F2) and fluvial gravels (F3) with very scarce fauna or absence of macrofaunal remains, also detected in other cores of this estuary and arranged on a Neogene substrate (BORRERO *et al.*, 1999; ARROYO *et al.*, 2021a, 2021b). These coarser sediments would begin to be deposited during the Late Pleistocene-Early Holocene, according to the dating of other cores and due to the domain of fluvial dynamics in this estuary during this period (DABRIO *et al.*, 2000). The geochemical analysis of these two facies has been studied by ARROYO *et al.* (2021a, 2021b) in other cores from this estuary and they show very low values of all the metals studied here, close to the regional background (RUIZ *et al.*, 1997).

Phase 3 (F4; 6.1-5.2 cal. kyr BP). The abundant macrofaunal and microfaunal record of F4 points to a subtidal brackish paleoenvironment during this phase (see section 5.A), with the presence of phanerogam meadows with abundant individuals of gastropods (*Bittium*, *Peringia*) and frequent foraminifera (*Ammonia*, *Haynesina*) and ostracods (mainly *Loxococoncha*) in the bottom sediments (BORJA, 1986; LUQUE & TEMPLADO, 2004; WUKOVITS *et al.*, 2018; PASCUAL *et al.*, 2019). This phase coincided with the maximum of the MIS-1 transgression in this area (6.5-5 cal. kyr BP; AZO

et al., 1994), which was invaded by the sea with the creation of cliffs formed by Neogene formations (DABRIO *et al.*, 2000).

Phase 4 (F5a; 5.2 cal. kyr BP-1.9 cal. kyr BP). This phase is characterized by clayey-silty sediments, frequent specimens of *Peringia ulvae* (PENNANT, 1877) and a foraminiferal assemblage dominated by *Ammonia tepida* (CUSHMAN, 1926), *Haynesina depressula* (WALKER & JACOB, 1798) and *Haynesina germanica* (EHRENBERG, 1840). These features are similar to those observed in estuarine intertidal channels of southwestern Spain with low hydrodynamic gradient (GONZÁLEZ-REGALADO *et al.*, 2001; GÓMEZ, 2017).

During this phase, the start of mining activities at ~4.5 cal. kyr BP (sample A-14; 6.2 m depth) caused a small pollution of this estuary, mainly by As, Cu, Pb and Zn (Fig. 4). A subsequent decrease in foraminiferal abundance was observed towards the top of this subfacies, as in other polluted estuaries (ALVE, 1995; NAGENDRA & REDDY, 2019). This decrease may also be related to an increasing clogging and lower tidal inputs (MURRAY, 2006; CEARRETA *et al.*, 2006).

Phase 5 (F5b; 1.9 cal. kyr BP-1985). Transit to F5b (4 m depth; ~1.9 cal kyr BP) is marked by: i) a further decrease in grain size (Fig. 2); ii) lower concentrations of Ca, Na and S (Fig. 3); iii) a small increase in Pb content (Fig. 4); iv) the disappearance of macrofauna, foraminifera and spines of echinoderms (Figs. 2.A - 3 - 5). All these features point to an increasing confinement of this area and a transition to marsh paleoenvironments, probably about mean sea level (GONZÁLEZ-REGALADO *et al.*, 2001; RUIZ *et al.*, 2007). The presence of fossil foraminifera (Table 1: sample A-3) are attributed to the erosion of the Neogene formations that make up the slopes near core A.

The higher Pb content could be due to pollution produced by Roman mining activities, very intense around 1900 cal. kyr BP (ARROYO *et al.*, 2021b). In addition, this phase includes the highest concentrations of heavy metals, derived from industrial effluents discharged into this estuary between 1966 and 1985 (Fig. 4; see previous chapter).

Phase 6 (uppermost part of F5b: subrecent). This phase is characterized by lower concentrations of major and trace elements and the presence of freshwater ostracods [mainly *Cypridopsis vidua* (O.F. MÜLLER, 1776)]. This phase would end the regression that started at the end of the MIS-1 transgression, with a transition from a subtidal channel (F4) to this inferred freshwater pond (FERNANDES *et al.*, 2010; MARTÍNEZ *et al.*, 2015), without connection with the tidal dynamics.

Phase 7 (F6: last decades). The uppermost 0.7 m constitute the anthropic filling carried out in recent decades in this area, with the construction of a model airplane circuit and a soccer field.

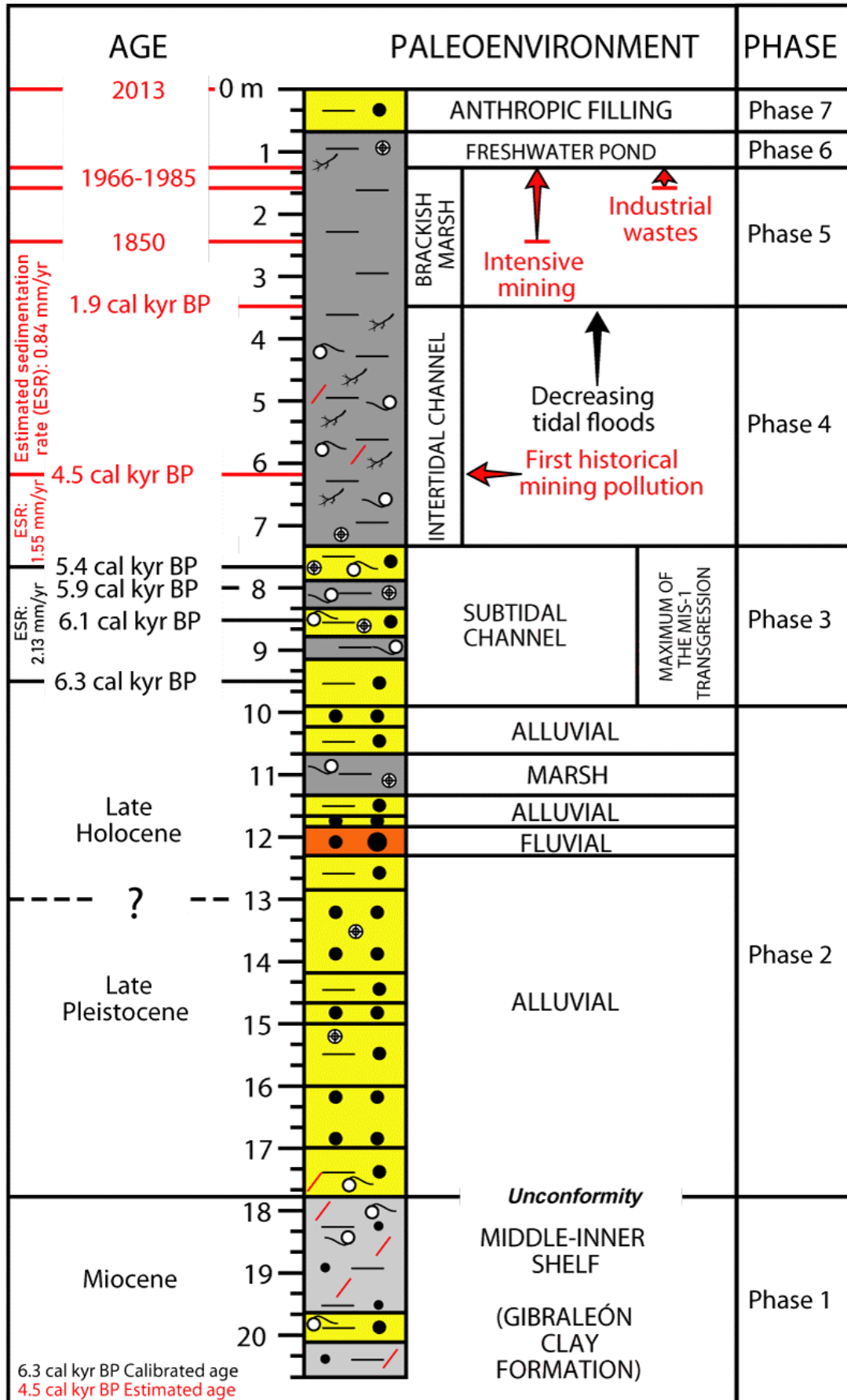


Figure 7: Paleoenviromental reconstruction of core A.
 Figure 7 : Reconstruction paléoenvironnementale de la carotte A.



6. Concluding remarks

The integrated analysis (sedimentology, geochemistry, paleontology, dating) of a long core collected in the middle estuary of the Tinto-Odiel estuary (SW Spain) reveals a complex evolution from marine to freshwater paleoenvironments. The lowermost meters of this core are composed of Miocene sandy silts, which were deposited on a middle-inner shelf. A significant thickness of these Neogene sediments were eroded during the Pleistocene-Early Holocene due the dominant fluvial dynamics during this period, with the deposit of fluvial and alluvial sands and gravels. This middle estuary was flooded during the maximum of the MIS-1 transgression (~6.5-5.2 cal kyr BP), with the implantation of seagrass meadows in a subtidal channel with typical associations of gastropods (*Peringia*, *Bittium*), foraminifera (*Ammonia*, *Haynesina*) and ostracods (mainly *Loxococoncha*). The subsequent regressive evolution included the transit from an intertidal channel to mudflats and finally to a freshwater pond, with a recent anthropic filling in the uppermost meter of the core. This paper also includes one of the oldest historical evidence of mining pollution (~4.5 cal. kyr BP), as well as two strong polluted levels derived from an intensive mining interval during the 19th and 20th centuries and a recent industrial period.

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