

## INTEGRATED CHRONOSTRATIGRAPHY AND TAPHONOMIC STUDIES IN MIOCENE SEDIMENTARY SUCCESSIONS OF THE EAST PISCO BASIN (PERU)

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### GEOLOGY OF THE PISCO BASIN: A REVIEW

Along the western side of the Ica River (Ica Desert, Peru), the Miocene Chilcatay and Pisco formations of the East Pisco Basin crop out. These units are characterized by an extraordinarily preserved marine vertebrate fossil assemblage, especially cetaceans. This work has the purpose of making chronostratigraphic and taphonomic studies on these formations, cropping out in the Ica River valley.

The Pisco Basin, in southern Peru, is one of the fault-bounded Cenozoic sedimentary basins along the Peruvian coast, located at 14°30' S of latitude. Along the coast, the most easterly Outer Shelf High separates the forearc Pisco Basin into an offshore and an onshore portion, referred to as the West Pisco Basin, still submerged, and East Pisco Basin (Fig. 1a). The sedimentary infill of the East Pisco Basin has been described by Dunbar *et al.* (1990) and DeVries (1998), and consists of five lithostratigraphic units: the Eocene Caballas Formation, the middle to upper Eocene Paracas Formation (subdivided in Los Choros and Yumaque members), the uppermost Eocene-lower Oligocene Otuma Formation, the uppermost Oligocene to lower Miocene Chilcatay Formation, and the upper Miocene to Pliocene Pisco Formation (Fig. 1b) (*e.g.*, Dunbar *et al.*, 1990; DeVries, 1998; DeVries & Jud, 2018).

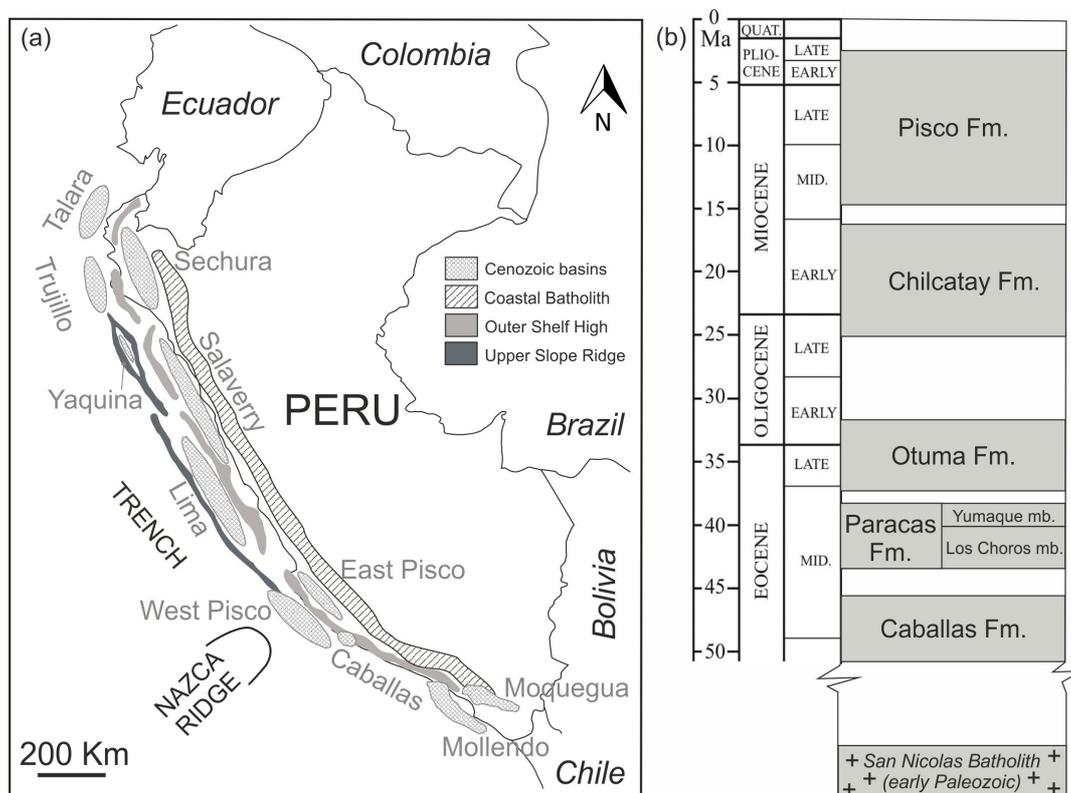


Fig. 1 - a) Map of the major sedimentary basins along the Peruvian coast. Sedimentary basins are indicated with ellipses. Major structural highs, *i.e.* the Coastal Batholith, the Outer Shelf High and the Upper Slope Ridge, are represented in oblique lines, light gray and dark gray, respectively. b) Schematic stratigraphic log with the main stratigraphic units of the East Pisco Basin. From Bosio *et al.* (2019).

The East Pisco Basin hosts one of the most important cetacean-bearing Fossil-Lagerstätten worldwide, with exceptionally well preserved fossils of marine vertebrates (Bianucci *et al.*, 2016a, b). In particular, along the western side of the Ica River, the Chilcatay Formation has received a wide attention in the past few years due to numerous significant discoveries of fossil cetaceans, in particular odontocetes (*e.g.*, Lambert *et al.*, 2017). In the same area, strata of the Mio-Pliocene Pisco Formation are characterized by fossil remains of cetaceans, both odontocetes and mysticetes, but also of pinnipeds, seabirds, sea-turtles, crocodiles, sharks, rays and bony fish (*e.g.*, Collareta *et al.*, 2015; Bianucci *et al.*, 2016a, b and references therein; Lambert *et al.*, 2017).

For its paleontological relevance, the East Pisco Basin has been studied for reconstructing a detailed chronostratigraphic framework and for investigating the reasons of the exceptional preservation of this Fossil-Lagerstätte.

## CHRONOSTRATIGRAPHY

### *Chronostratigraphy of the Chilcatay Formation*

The sedimentological and stratigraphic reconstructions of the Chilcatay Formation at Ullujaya and Zamaca localities (14°34'59"S-75°38'27"W; 14°38'3"S-75°38'6"W) depicts a shallow-water paleoenvironment, characterized by three facies associations: the *Ct1c*, composed of massive sandstones and conglomerates with boulder-sized clasts at the bottom; the *Ct1a* recording the coeval deeper parts of the basin (offshore setting) receiving bypass suspended fines from the shallower ramp; and the *Ct1b* recording a distally steepened mixed siliciclastic-bioclastic ramp (Fig. 2). Sited above the Ct1 allomember, the Ct2 allomember consists of two major facies associations comprising shoreface sandstones (*Ct2a*) and offshore silty facies (*Ct2b*).

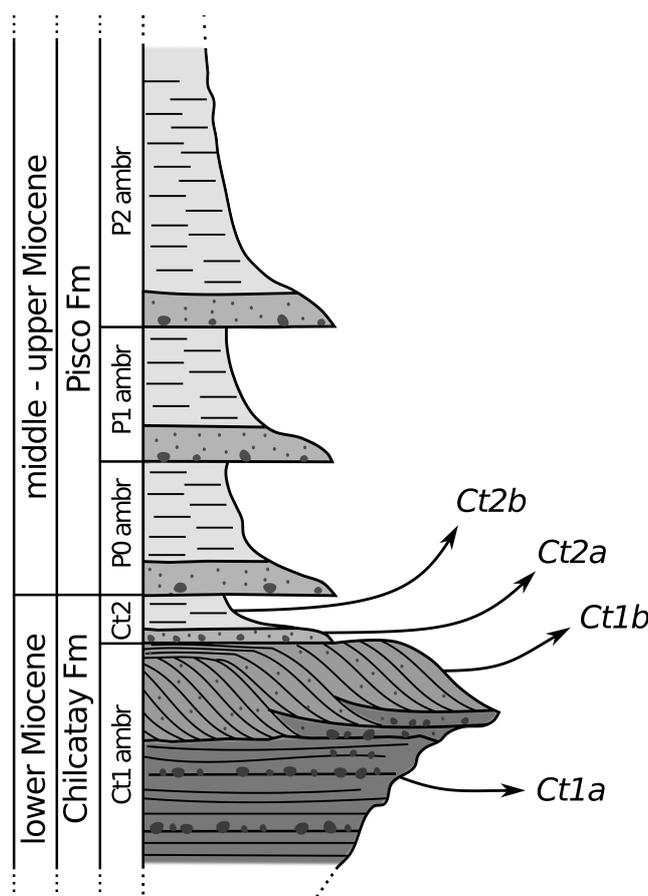


Fig. 2 - Schematic stratigraphic column of the sedimentary succession exposed at Ullujaya, with the Chilcatay and Pisco formations subdivided in allomembers (not to scale). Abbreviations: ambr = allomember; Fm = Formation. Modified from Collareta *et al.* (2019).

For reconstructing a chronostratigraphic framework of all these units, tephra were analyzed with  $^{39}\text{Ar}$ – $^{40}\text{Ar}$  dating method on biotite phenocrysts. All the dated tephra were classified as calcalkaline, based on the biotite chemistry, and rhyolitic, based on the glass composition. Despite some problems caused by a variable alteration of the biotite phenocrysts, all the samples give Burdigalian ages (early Miocene).

At the base, the PN-T2 tephra, 5 m above the Chilcatay basal unconformity at Roca Negra ( $14^{\circ}39'04''\text{S}$ – $75^{\circ}38'54''\text{W}$ ), gives an age of  $19.25 \pm 0.05$  Ma, which agrees with the lowest ash layer (UJA-T35) of *Ct1a* at Ullujaya, which is dated at  $19.00 \pm 0.28$  Ma, despite the slight alteration (Fig. 3).

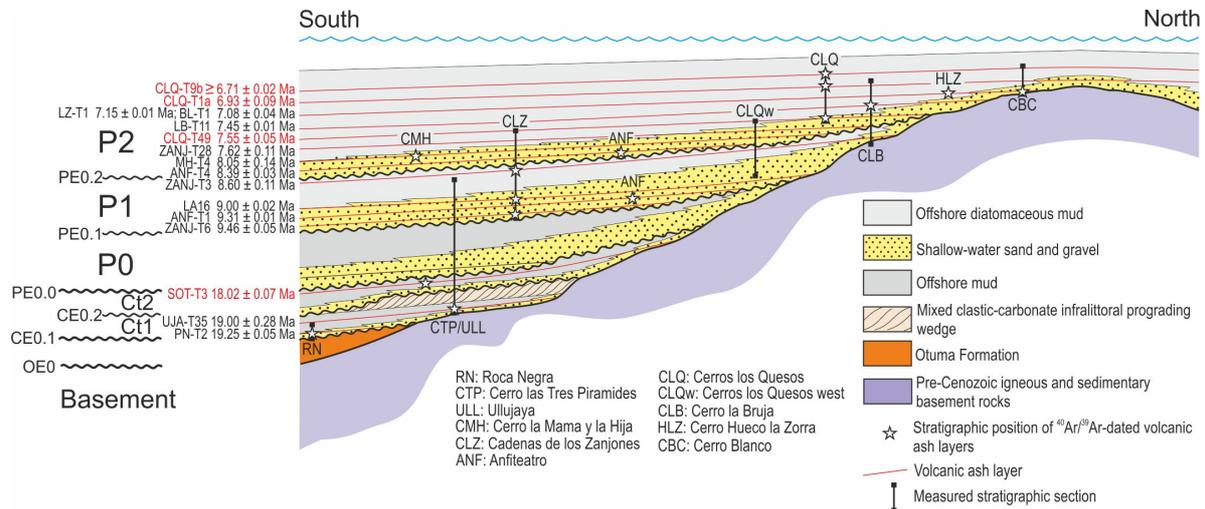


Fig. 3 - Schematic, dip-oriented stratigraphic diagram for the Chilcatay and Pisco formations showing both the position of measured stratigraphic sections (vertical black lines) and the stratigraphic position of dated tephra layers (stars). Red lines show dated volcanic ash layers.  $^{39}\text{Ar}$ – $^{40}\text{Ar}$  ages are reported for each tephra. From Bosio *et al.* (2019).

These ages agree with the silicoflagellate biostratigraphic data, which assigned an age of 18–19 Ma to the lower part of the formation. The top of the Chilcatay Formation has an age of  $18.02 \pm 0.07$  Ma dating the ash layer SOT-T3, sampled 1 m below the Pisco basal unconformity at Los Dos Cerritos ( $14^{\circ}35'40''\text{S}$ – $75^{\circ}40'05''\text{W}$ ) (Fig. 3). This  $^{39}\text{Ar}$ – $^{40}\text{Ar}$  age is close but not overlapping with the biostratigraphic data based on diatoms, which suggest an age younger than 17.08 Ma for the top of the formation. Another Burdigalian age was obtained from an ash layer near a new squalodelphinid from the Chilcatay Formation exposed south of Cerro Colorado, dated at  $18.78 \pm 0.08$  Ma.

In addition to the tephrochronology, a Strontium Isotope Stratigraphy (SIS) was applied to the Ct1 allomember of the Chilcatay Formation, following McArthur *et al.* (1994). To avoid diagenetic bias, the suitability of shells was first assessed via optical and scanning electron microscopy, cathodoluminescence, and inductively coupled plasma-optical emission spectrometry (ICP-OES). Burdigalian ages, *i.e.* from 18.8 to 18.0 Ma, were obtained applying this method, confirming the  $^{39}\text{Ar}$ – $^{40}\text{Ar}$  and biostratigraphic ages.

#### Chronostratigraphy of the Pisco Formation

The youngest Pisco Formation is divided stratigraphically in three allomembers, P0, P1 and P2, due to recognition of three basin-wide unconformities (called PE0.0, PE0.1 and PE0.2). In each allomember, shallower-water sand-rich deposits rest unconformably on deeper, diatomite-rich deposits (aside for P0), representing transgressive cycles (Fig. 2).

The presence of tephra layers from the Central Andes within the sedimentary succession represents a great opportunity for applying the tephrochronology. Ash layers were dated with  $^{39}\text{Ar}$ – $^{40}\text{Ar}$  method for reconstructing the chronostratigraphy of each unit. At the base of the P1 allomember, ash layers (ZANJ-T6, LA(16)) provide ages of  $9.46 \pm 0.05$  Ma and  $9.00 \pm 0.02$  Ma few meters above the PE0.1 unconformity (Fig. 3). These ages are supported by diatom biostratigraphy in the same measured section of Cerros Cadenas de los Zanjones ( $14^{\circ}34'13''\text{S}$ – $75^{\circ}43'45''\text{W}$ ). The co-occurrence of the diatom marker species *Lithodesmium reynoldsii*

and *Denticulopsis praekatayamae* in the first 15 m of the section constrains the base of the P1 allomember between 9.5 and 8.9 Ma (respectively FO of *D. praekatayamae* and the LO of *L. reynoldsii*). At the top of the P1 allomember, the uppermost ZANJ-T3 tephra at Cerros Cadenas de los Zanjones gives a final age of  $8.60 \pm 0.11$  Ma, despite the alteration that affects the sample. The P1 allomember is entirely Tortonian, in agreement with the previous  $^{39}\text{Ar}$ - $^{40}\text{Ar}$  age of  $9.10 \pm 0.04$  Ma obtained in P1 at Cerro Colorado (Gariboldi *et al.*, 2017).

In the P2 allomember, the base is dated thanks to the MH-T4 and ANF-T4 ash layers, giving an age of  $8.05 \pm 0.14$  Ma and  $8.39 \pm 0.03$  Ma, respectively (Fig. 3). The dated LB-T11 tephra at Cerro la Bruja ( $14^{\circ}31'35''\text{S}$ - $75^{\circ}39'36''\text{W}$ ) gives an age of  $7.45 \pm 0.01$  Ma and is correlated by tephra fingerprinting with an ash layer at Cerro los Quesos located between tephra dated at  $7.55 \pm 0.05$  Ma and  $6.93 \pm 0.09$  Ma (Fig. 3). The P2 allomember is also exposed at Cerros Cadenas de los Zanjones, where one of the uppermost ash layers in the section, ZANJ-T28, gives an age of  $7.62 \pm 0.11$  Ma (Fig. 3). At Cerro Blanco ( $14^{\circ}34'13''\text{S}$ - $75^{\circ}41'31''\text{W}$ ) and Cerro Hueco la Zorra ( $14^{\circ}26'17''\text{S}$ - $75^{\circ}41'37''\text{W}$ ), BL-T1 and LZ-T1 ash layers give  $7.084 \pm 0.044$  Ma and  $7.155 \pm 0.015$  Ma, respectively (Fig. 3). The strata of the P2 allomember exposed in the study area were thus deposited between  $8.39 \pm 0.03$  Ma and  $6.71 \pm 0.02$  Ma (Gariboldi *et al.*, 2017), therefore in the upper Tortonian and the early Messinian.

Because of the lack of both microfossils and tephra, Strontium Isotope Stratigraphy was applied for solving the age of the P0 allomember, the lowest unit of the Pisco Formation. Discarding samples affected by diagenesis,  $^{87}\text{Sr}/^{86}\text{Sr}$  values suggest a mean age of 13.4 Ma, with an uncertainty time range spanning between 14.8 and 12.4 Ma. Consequently, the P0 allomember can be placed by means of SIS in the Langhian–Serravallian (middle Miocene).

#### *Tephra fingerprinting for a high resolution stratigraphy*

Due to their regional dispersal and to their geologically instantaneous deposition (Lowe, 2011), tephra layers provide the opportunity not only to date specific layers, when suitable for radiometric age determination, but also to correlate different localities, through the chemical fingerprinting of tephra. Tephra fingerprinting is a unique tool for reconstructing a high resolution stratigraphy (Lowe, 2011; Smith *et al.*, 2011).

In the upper allomember of the Pisco Formation, P2, volcanic ashes were analyzed for correlating stratigraphic sections at distant localities. Despite the similar glass composition and mineral assemblage, together with the shallow marine depositional environment limiting tephra preservation, correlations between distant localities were realized by fingerprinting tephra layers on the basis of petrographic and compositional investigations, grain-size analyses, and glass shard morphology. Major element composition of biotite proved to be a valuable tool for discriminating ash layers and correlating different stratigraphic sections located several kilometers apart from each other, such as Cerro los Quesos ( $14^{\circ}29'57''\text{S}$ - $75^{\circ}43'06''\text{W}$ ) and Cerro la Bruja ( $14^{\circ}31'35''\text{S}$ - $75^{\circ}39'36''\text{W}$ ). In the sections measured at these localities, tephra pairs allow a correlation of high resolution (a few meters).

## INVERTEBRATE AND VERTEBRATE TAPHONOMY

### *Mollusk taphonomy*

Fossil invertebrates are quite common in the Miocene Chilcatay and Pisco formations (Di Celma *et al.*, 2017) and exhibit different preservation modes suggesting two distinct fossilization pathways.

In the early Miocene Chilcatay Formation, invertebrates show a high concentration but a low biodiversity, consisting essentially of bivalves, *i.e.*, pectinids and ostreids, barnacles, serpulids and echinoids. Analyses with optical microscope, SEM-EDS, Raman spectroscopy and cathodoluminescence demonstrate that fossil shell remains are constituted by calcite and show a quite good preservation. Oysters usually display a pristine prismatic microstructure and exhibit layers with different kind of luminescence response, proving that they have pristine calcite layers but also zones characterized by diagenetic calcite precipitation.

In the late Miocene Pisco Formation, the invertebrate content is limited to few mollusk-rich layers characterized by a high abundance and low biodiversity: mainly bivalves of the families Crassatellidae and Veneridae, and gastropods of the family Turritellidae. The process of the invertebrate fossilization is very dissimilar to that of the Chilcatay Formation: in some localities, as Cerro los Quesos and Cerro Cadena de los Zanjones, mollusks are preserved as internal molds or recrystallized shells, and, in some cases, they show geopetal structures. Through aforementioned analyses, we observed that the pristine shell is not preserved: calcite is usually absent. The mineralogical phases are generally represented by dolomite and gypsum, but no-pristine calcite is preserved. Cathodoluminescence analyses, in fact, confirm that the calcite is interested by diagenetic processes also in cases where the pristine structure of the shell seems to be preserved under the microscope.

#### *Barnacle taphonomy*

In the lower allomember (Ct1) of the Chilcatay Formation, two slightly different barnacle facies can be recognized in *Ct1a* and *Ct1b* facies associations. In both facies, the cirripede remains occur as displaced clusters, displaced complete shells and wall plate fragments. The clusters are generally detached from their original substrate (Type C of Nomura & Maeda, 2008; displaced clusters of Doyle *et al.*, 1997). Complete specimens are generally moderate to well preserved, but display evidence of abrasion and lack the opercula. The best preserved specimens retain their pigmentation. The shells are often filled by sand. In the barnacle facies of *Ct1a*, this filling is remarkably rich in bioclastic fragments and has less abundant mineral grains than the rock embedding the specimens. Disarticulated plates are often abraded (Grade 1 of Nielsen & Funder, 2003). Opercula are rare - only a couple of abraded and fragmented scuta have been recovered.

Based on macroscopic features of the wall plates and on their internal microscopic structure, three barnacle taxa have been recognized: cf. *Austromegabalanus* sp., Balanidae indet., and Concavinae indet.

#### *Bone preservation in Chilcatay and Pisco formations*

The extraordinary vertebrate preservation is reknown in both the formations, even the best preserved findings have been discovered in the Pisco Formation, especially in the P1 and P2 allomembers. The reasons of this exceptional fossilization are still scarcely known, therefore the taphonomy of vertebrate remains of both the formations have been investigated.

In the Chilcatay Formation, cetacean bones are well preserved and exhibit a good degree of apatite mineralization. Despite the absence of carbonate nodules, in contrast with those of the Pisco Formation, bone cavities are locally filled by Ca-Mg carbonates. Only in few cases microborings of the B-type (*sensu* Gariboldi *et al.*, 2015) occurred in the compact bone, but larger borings made by benthic invertebrates, such as *Osedax*, are not observed. No remains of whale-fall communities are present. The reason of such a good preservation would be the occurrence of anoxic events. The size distribution of pyrite framboid relics in the Ct1 fine sediments at Ullujaya revealed indeed some fluctuations of euxinic and oxic-dysoxic conditions at the seafloor (Wilkin *et al.*, 1996). Therefore, the fossil vertebrate preservation was favored by the oxygen-deficient bottom conditions that inhibited the action of benthic invertebrates and macro-scavengers and the formation of a developed whale-fall community.

In the Pisco Formation, the reason of the exceptional preservation was first attributed to the rapid burial, but is not the only process that comes in. One of the explanation was proposed by Gariboldi *et al.* (2015), who illustrated how the carbonate nodules, especially dolomite concretions, could prevent the degradation of the bones and diagenetic compression of skeletons during burial, and the erosion of fossils after exhumation (Fig. 4). This process is mainly observed for the carcasses of big dimensions, but small specimens, as odontocetes, usually do not have a concretion around the skeleton. At the sea bottom, indeed, the fall of small-sized cetacean carcass did not induce a local chemical variation of the environment strong enough to reach conditions favorable to the carbonate precipitation. For understanding the preservation of small cetaceans, a small pontoporiid dolphin was analyzed, and we found out that a rapid burial of the specimen is pivotal for the bone preservation, allowing

the early establishment of anoxic processes for degradation of organic matter. The presence of a permeable sediment without any dolomite nodule, together with the availability of phosphorous released from the Fe-reduction processes and the carcass decay in anoxic environment, favored the recrystallization of bone apatite, allowing excellent bone tissue preservation.

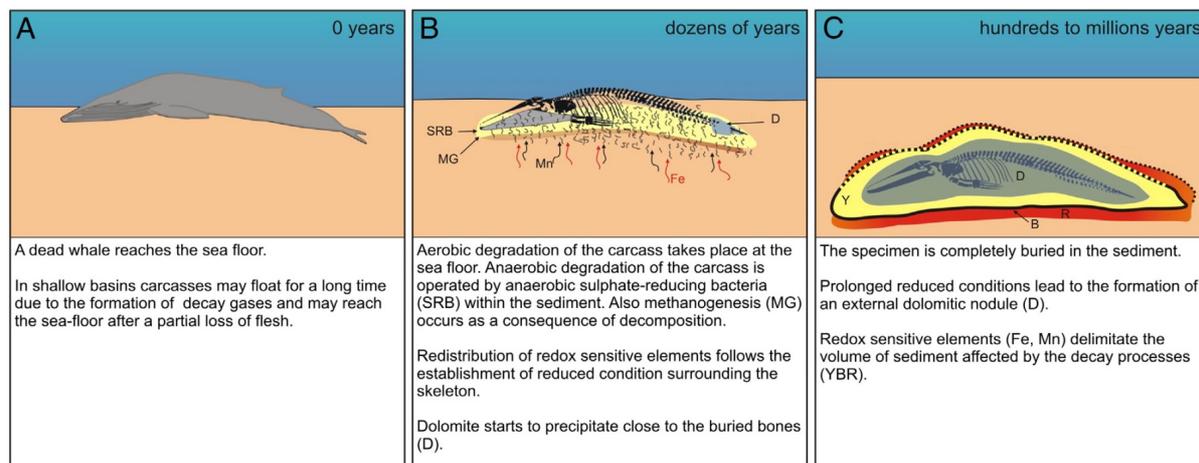


Fig. 4 - Early-diagenetic dolomite formation reconstruction. a) Marine vertebrate deposited at the sea floor. b) The consumption of the soft tissue is rapid compared to lipid degradation. Rapid burial affected the skeleton. Sulphate reducing bacteria are represented. c) The sediment surrounding the skeleton is permanently modified by the consequences of decay. From Gariboldi *et al.* (2015).

Thus, with this work it was demonstrated that a good preservation is possible in such different environments, both anoxic and oxic/dysoxic, and in very different sediments, because of the diverse and multiple diagenetic processes that come in after the deposition of the whale carcass on the seafloor. The fossilization processes and the reasons of this exceptional abundance and extraordinary preservation must continue to be studied in depth for understanding how a Fossil-Lagerstätte such as that of the Pisco Basin could be formed.

## DISCUSSION AND CONCLUSIONS

The multidisciplinary work carried out in these years in the western side of the Ica River resulted in a stratigraphic, paleoecologic, taphonomic and chronostratigraphic studies of the Miocene sedimentary successions such as the Chilcatay and Pisco formations.

Considering chronostratigraphic results, given by  $^{39}\text{Ar}$ - $^{40}\text{Ar}$  dating on tephra, tephrostratigraphy, biostratigraphy, and SIS, the whole Chilcatay Formation in the study area is assigned to the Burdigalian (early Miocene), precisely between 19.25 and 18.02 Ma (or 17.08 Ma following only the diatom biostratigraphy). These results could be tentatively correlated to the Mi-events, the oxygen isotopic maxima in the Miocene (Miller *et al.*, 1991). The Chilcatay Formation would be deposited between the Mi1 or Mi1aa (dated at 23.2 or 20.0 Ma, respectively) and the Mi2 event (dated at 16.3 Ma), with a gap in correspondence of the Mi1ab (dated at 18.7 Ma) that would correspond to the CE0.2 unconformity between Ct1 and Ct2 allomembers.

Regarding the Pisco Formation, chronostratigraphic results assigned this formation to the middle-late Miocene. In particular, Sr isotope analyses on carbonates and phosphates suggest a Langhian-Serravallian age for the P0 allomember (from 14.8 to 12.4 Ma). The P1 allomember was instead assigned entirely to the Tortonian (from 9.46 to 8.60 Ma) and the P2 allomember was dated to the Tortonian-Messinian (from 8.39 to 6.71 Ma). The application of the tephra fingerprinting to the P2 deposits allow to build a high resolution stratigraphy and to correlate sediments exposed in different localities, with important implications for the vertebrate paleontology. The chronostratigraphic framework allows an attempt of correlation with the Mi-events: the Pisco Formation would be deposited after the Mi2 (dated at 16.3 Ma) correlated with the Pisco basal

unconformity. Within the formation, the PE0.1 unconformity can correspond to the Mi6 (10.3 Ma in age) and the PE0.2 unconformity can be related to the Mi7 (dated at 8.7 Ma).

Fossil preservation and diagenetic processes in the Chilcatay Formation are very different from those of the Pisco Formation. Taphonomic studies in the Chilcatay Formation reveal that mollusks and barnacles are usually moderate to well preserved and maintained their pristine calcite shell. In the Pisco Formation, fossil invertebrates show a very bad preservation, with some exceptions in the P0 allomember, where few carbonate shells are still found, but in some cases they are completely recrystallized. In the youngest allomembers, invertebrate shells are absent or completely substituted by gypsum, and mollusks are only present as dolomite/gypsum internal molds or casts.

Both these formations are characterized by an abundant and exceptional concentration of fossil vertebrates, despite the differences in lithologies and paleoenvironments. In the Chilcatay Formation, fossil vertebrates are disarticulated but almost complete, and their bones show a good preservation, probably due to the fluctuation of euxinic and oxic-dysoxic conditions at the seafloor. The Pisco Formation shows an exceptional concentration of fully articulated and complete specimens, and the preservation of delicate tissues, as baleen. Bones are well preserved and exhibit a good permineralization or phosphatization. This extraordinary preservation is due to a rapid burial, an early diagenetic carbonate nodule formation, but especially to the diagenetic processes going on in the sediment enclosing vertebrate remains in the early stages after the burial.

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