Bosio, G., Malinverno, E., Collareta, A., Di Celma, C., Gioncada, A., Parente, M., Berra, F., Marx, F.G., Vertino, A., Urbina, M. & Bianucci, G. (2019). Strontium Isotope Stratigraphy and the thermophilic fossil fauna from the middle Miocene of the East Pisco Basin (Peru). Journal of South American Earth Sciences, 102399.

DOI: https://doi.org/10.1016/j.jsames.2019.102399

Strontium Isotope Stratigraphy and the thermophilic fossil fauna from the middle Miocene of the East Pisco Basin (Peru)

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Highlights

- We applied Sr isotope dating to carbonates and phosphates from the East Pisco Basin
- $^{87}Sr/^{86}Sr$ values date the lower Pisco strata (P0 sequence) to the middle Miocene
- Paleontological data indicate a warm water paleoenvironment for P0
- Our results suggest a middle-late Miocene strengthening of the Humboldt Current

Abstract

New age estimates obtained via Strontium Isotope ($^{87}Sr/^{86}Sr$) Stratigraphy and new paleoclimatic data are here presented for the Miocene Chilcatay and Pisco formations exposed in the East Pisco Basin, an Andean forearc basin of southern Peru, which is renowned worldwide for its exceptional content of fossil marine vertebrates. Mollusk and barnacle shells, carbonate nodules, and shark teeth were collected along three stratigraphic sections for applying Strontium Isotope Stratigraphy on both carbonates and phosphates. To avoid diagenetic biases, mollusk and barnacle shells were analyzed in detail by means of optical and scanning electron microscopy, cathodoluminescence, and inductively coupled plasma-optical emission spectrometry, whereas only the enameloid from the best-preserved shark teeth was sampled. The obtained $^{87}Sr/^{86}Sr$ ages confirm a late early Miocene (Burdigalian) age for the Chilcatay strata, and reveal middle Miocene (Langhian to Serravallian) ages for the lower Pisco unit (i.e., the P0 sequence) – a result that matches the relatively archaic aspect of its cetacean fossil assemblage. New and literature data about the fossil assemblage of the lower Pisco beds highlight the presence of several thermophilic invertebrates and vertebrates, thus suggesting a warm-water, tropical paleoenvironment for this middle Miocene sequence. Such a paleoenvironmental scenario recalls the warm conditions associated with the Chilcatay Formation, rather than the cooler setting inferred for the remainder of the Pisco Formation (i.e., the P1 and P2 sequences). This pattern likely reflects the late Miocene trend of global cooling or a middle to early late Miocene strengthening of the Humboldt Current.

Keywords

$^{87}Sr/^{86}Sr$ stratigraphy, diagenesis evaluation, Miocene, mollusks, shark teeth, cetaceans

1. Introduction

The $^{87}Sr/^{86}Sr$ ratio of Sr (strontium) isotopes dissolved in the global ocean has varied through the geological time (McArthur, 1994; McArthur et al., 2012). At the same time, the world's seas are homogeneous with respect to $^{87}Sr/^{86}Sr$ ratio, and this is assumed for any geologic time (McArthur et al., 2012). Therefore, Sr isotope composition can be used for dating marine minerals and correlating

marine sedimentary successions worldwide (Faure and Mensing, 2005). Application of Strontium Isotope Stratigraphy (hereinafter: SIS) requires measurement of the Sr isotope ratio of minerals that precipitated from seawater and have not undergone diagenetic alteration of their original $^{87}Sr/^{86}Sr$ value (McArthur et al., 2012). Some intervals of geological time, such as the Miocene, are particularly favorable for implementation of SIS because they display high rates of change of the $^{87}Sr/^{86}Sr$ value and a narrow error band on the empirically defined reference curve (McArthur et al., 2012), which allow for high-resolution age estimates.

The Miocene Chilcatay and Pisco formations exposed in the East Pisco Basin of southern Peru are renowned for their exceptional fossil assemblage, comprising cetaceans, seals, sharks, rays, crocodiles, turtles, seabirds, and bony fishes (e.g., Bianucci et al., 2015, 2016a, b, c, 2018a, b; Lambert et al., 2014, 2015b, 2017a, b, 2018; Landini et al., 2017a, b, 2019; Marx et al., 2017a, b; Gioncada et al., 2018b) as well as marine invertebrates (e.g., DeVries and Frassinetti, 2003; DeVries et al., 2006; DeVries, 2016; Coletti et al., 2018, in press; Collareta et al., 2019). In particular, the fossil record of the Pisco Formation is outstanding in terms of both abundance and preservation state of the vertebrate remains. Examples of exceptionally preserved fossil vertebrate specimens from the Pisco strata exposed in the East Pisco Basin include phosphatized baleen bristles and plates of mysticete whales (Brand et al., 2004; Esperante et al., 2008; Gioncada et al., 2016; Marx et al., 2017a), stomach contents and regurgitations of mysticete and odontocete cetaceans (Collareta et al., 2015; Lambert et al., 2015a), and phosphatized skeletons of cartilaginous fishes such as sharks (Collareta et al., 2017).

For interpreting this extraordinary paleontological heritage and enhancing its scientific significance, its placement in a robust stratigraphic context supported by reliable age estimates is crucial. Recent work established a chronostratigraphic framework based on $^{39}Ar–^{40}Ar$ dating, tephrostratigraphy and biostratigraphy, which dates the Chilcatay Formation to the early Miocene and the upper sequences of the Pisco Formation to the late Miocene, respectively (Di Celma et al., 2017, 2018a, b; Gariboldi et al., 2017; Bosio et al., 2019, in press). The seemingly large temporal gap between these two units is striking, but is almost certainly an overestimate caused by a lack of datable ash layers and microfossils from the lowermost portion of the Pisco Formation (the P0 sequence). Previous studies remarked on the significantly archaic aspect of the cetacean fauna from this layer (Di Celma et al. 2017; Marx et al. 2017b), but were unable to draw definitive conclusions as to its age. The peculiar faunal changes that characterize this sequence (Di Celma et al., 2017) make the dating of these strata an issue of prime importance for the ongoing paleontological studies on the Miocene fossil content of the East Pisco Basin.

Here, we present the first attempt to date the lower Pisco beds by means of SIS. Mollusk (i.e., ostreids and pectinids) and barnacle shells, diagenetic cements, bulk sediment samples, and shark teeth were collected to apply this method to both carbonates and phosphates. Several samples from the age-constrained Ct1 sequence of the Chilcatay Formation were dated for checking the reliability of the Sr results in the study areas. The preservation state of the invertebrate remains was preliminary addressed via petrographic, morphological, chemical, and cathodoluminescence analyses in order to quantify the extent of diagenetic alteration and to assess whether the shells were structurally and chemically pristine. Sr isotope ratios were elaborated with the LOWESS Table 5 made for the GTS2012 timescale (McArthur et al., 2012) and SIS ages were obtained.

Furthermore, we investigated the faunal assemblages of both fossil invertebrates and vertebrates for reconstructing the environmental and paleoclimatic significance of the P0 sequence in the light of our age estimates. Having been deposited in a key time interval that saw first-order changes of the climate and oceanographic system at both the global scale (e.g., the Middle Miocene Climatic Optimum and the Middle Miocene Climatic Transition) and the regional scale (e.g., the emergence of the modern upwelling regime along the Pacific margin of South America), the P0 sequence and its newly dated fossil content represent indeed crucial elements for reconstructing the mode and tempo of the major Miocene environmental shift episodes off the coast of Peru – the current location of the outstandingly productive Humboldt Current (Penven et al., 2005).

2. Geological and paleontological framework

The East Pisco Basin is one of the elongated extensional/pull-apart sedimentary basins along the composite transform-convergent Peruvian margin (e.g., Thornburg and Kulm, 1981; Kulm et al., 1982; Dunbar et al., 1990; León et al., 2008; Zúñiga-Rivero et al., 2010) (Fig. 1A). Its emersion and subaerial exposure is due to the subduction of the nearby Nazca Ridge, a region of topographically high oceanic crust impinging on the Peru-Chile trench (Pilger, 1981; Hsu, 1992; Macharé and Ortlieb, 1992; Hampel, 2002). The basin fill comprises, in stratigraphic order, the Eocene Caballas and Paracas formations, the upper Eocene to lower Oligocene Otuma Formation, and the largely Miocene Chilcatay and Pisco formations (Dunbar et al., 1990; DeVries, 1998, 2017; DeVries et al., 2017; DeVries and Jud, 2018) (Fig. 1C). These units are bounded by regionally extensive unconformities and are also internally divided by less pronounced intraformational unconformities (DeVries, 1998; Di Celma et al., 2017, 2018a, b). As such, this succession can be subdivided using both a lithostratigraphic and an allostratigraphic approach (North American Commission on Stratigraphic Nomenclature [NACSN], 2005). In the present paper, we refer to the formations as "composite sequences" and to their internal informal units as "sequences".

During deposition of the Chilcatay and Pisco formations, the East Pisco Basin was a semi-enclosed, shallow-marine bay, structured along a latitudinal depth gradient, and sheltered seawards by a longshore chain of crystalline basement islands (Fig. 1B) (Marocco and Muizon, 1988; DeVries and Jud, 2018; Bianucci et al., 2018b).

The Chilcatay Formation was deposited during the late early Miocene, between 19 and 17 Ma (Burdigalian) (Belia et al. 2019; Di Celma et al., 2018b; Bosio et al., in press). It is composed of two distinct sequences, namely, Ct1 and Ct2 (Di Celma et al., 2018b, 2019) (Fig. 1C). At Ullujaya (14°34'59"S; 75°38'27"W) and Roca Negra (14°39'04"S; 75°38'54"W), two localities along the western side of the Ica River (Fig. 2A, 3A, B), the lower sequence (Ct1) of the Chilcatay Formation consists of massive sandstones and conglomerates with boulder-sized clasts at the bottom (*Ct1c* facies association), followed by medium- to fine-grained sandstones alternating with conglomerate beds (*Ct1a*), and clinobedded coarse-grained calcirudites at the top (*Ct1b*) (Di Celma et al., 2018b, 2019). These facies associations record deposition in the shoreface, offshore, and on a mixed siliciclastic-carbonate subaqueous delta, respectively (Di Celma et al., 2019), and suggest a semi-protected embayment associated with both a river mouth and the open ocean (Bianucci et al., 2018b). The invertebrate fossil assemblage of the Chilcatay strata exposed at the study sites comprises mollusks, barnacles, serpulids and echinids (Di Celma et al., 2018b, 2019). Bivalves are represented by ostreids, pectinids and venerids. Barnacles mainly belong to three taxa, with *Austromegabalanus carrioli* being the most abundant (Coletti et al., 2018, in press; Collareta et al., 2019). The vertebrate fossil assemblage is dominated by toothed cetaceans (Odontoceti), including kentriodontids (*Kentriodon* sp.), squalodelphinids (*Notocetus vanbenedeni* and *Huaridelphis raimondii*), physeteroids (sperm whales), and the eurhinodelphinid-like *Chilcacetus* (Bianucci et al., 2015, 2018b; Lambert et al., 2014, 2015b; Di Celma et al., 2018b, 2019). In addition, there are sea turtles (large-sized indeterminate dermochelyids), bony fishes and elasmobranchs, with abundant juvenile teeth of the bronze shark *Carcharhinus brachyurus* and the extinct lamniform *Cosmopolitodus hastalis*. Overall, the fossil assemblage points to a coastal community dominated by warm-water mesopredators (Bianucci et al., 2018b).

The Pisco Formation is composed of three fining-upward sequences, designated P0, P1, and P2 from oldest to youngest, which are divided by three unconformities, named PE0.0, PE0.1, PE0.2, respectively (Fig. 1C). Each unit, representing a transgressive cycle, recorded deposition in shoreface (sandstones) and in offshore (siltstones/diatomaceous siltstones) settings. The time of deposition of the P1 and P2 sequences is late Miocene and is well-constrained between 9.5 Ma and 8.6 Ma, and between 8.4 and 6.7 Ma, respectively (Gariboldi et al., 2017; Bosio et al., in press). Due to the lack of tephra layers and microfossils, the depositional age of the lower unit, P0, is to

date very poorly constrained between 18 and 9.5 Ma (Di Celma et al., 2017; Bosio et al., in press). Note, however, that DeVries and Jud (2018) previously proposed an early–middle Miocene age for the lower portion of the Pisco Formation (including P0), based on a series of diatom samples analyzed by H. Schrader in the 1980s. The P0 strata of the Pisco Formation reach their maximum thickness (about 40 m) at the locality of Cerro Submarino (14°34'38"S; 75°39'51"W) (Fig. 3D). This unit is mostly composed of fine- to very coarse-grained, cross-stratified, fossil-rich sandstones, suggesting a very nearshore environment. The fossil invertebrate assemblage of P0 is dominated by mollusks, with barnacles being less common. Mollusks are mostly represented by bivalves and primarily include *Chionopsis* spp. and *Dosinia ponderosa*, but *Miltha* cf. *vidali* and ostreids are also present (Di Celma et al., 2017). Vertebrates are abundant, but less studied than those from the underlying Chilcatay strata. Baleen whales (Cetacea: Mysticeti) dominate the assemblage and are represented by i) a large-sized stem balaenopteroid, *Pelocetus* sp. (Bianucci et al., 2019); ii) the archaic cetotheriid *Tiucetus rosae*, previously described from the southeastern locality of Santa Rosa (Marx et al., 2017b); and iii) a third form, previously identified as a cetotheriid (Di Celma et al., 2017), but here more conservatively reinterpreted as an indeterminate plicogulan (i.e., a crown mysticete more closely related to extant rorquals than to extant right whales). Toothed whales include at least one physeteroid and two kentriodontid-like delphinoids. In addition, there are fragmentary fossils of chelonioid turtles and longirostrine crocodilians, and a single bird specimen representing an indeterminate pelagornithid (Di Celma et al., 2017). Shark and ray teeth are relatively common and include, among others, *Carcharocles megalodon*, *Cosmopolitodus hastalis*, *Isurus oxyrinchus*, and myliobatids (eagle rays) (Di Celma et al., 2017).

3. Material and Methods

3.1. Study area and sample collection

The study area is settled in the Ica Desert, at the localities of Roca Negra (14°39'04"S; 75°38'54"W) and Ullujaya (14°34'59"S; 75°38'27"W), where the Chilcatay Formation crops out (Di Celma et al., 2018b, 2019; Bianucci et al., 2018b), and Cerro Submarino (14°34'38"S; 75°39'51"W), where P0 displays its maximum thickness (40 m) (Fig. 2, 3).

From 2015 and during five successive field campaigns, the Chilcatay and Pisco outcrops at the localities of Roca Negra, Ullujaya, and Cerro Submarino were investigated in detail for fossil vertebrates and macro-invertebrates by means of systematic surface prospecting. Preliminary identifications of the surveyed fossil specimens were made in the field. A few highly significant specimens, including representatives of the thermophilic taxa reported herein, were collected and

deposited in the Museo de Historia Natural de la Universidad Nacional Mayor de San Marcos (MUSM) in Lima for preparation and study.

Samples collected for applying the SIS were carbonates (invertebrate shells, diagenetic cements) and phosphates (shark teeth). For the Sr isotope analyses, shark tooth fluorapatite and low-Mg calcite shells were preferred, as low-Mg calcite is more stable, more resistant to diagenesis and less soluble than other carbonates (Brand, 1991; Steuber, 2003). Mollusks (i.e., ostreids and pectinids) and barnacles, shark teeth, and bulk and cement samples were collected along the measured section of the well-dated Chilcatay Formation at Roca Negra and Ullujaya, as well as from the undated P0 deposits at Cerro Submarino (Fig. 2, 3). Different horizons were selected at each locality (Fig. 3, C) and, for each level, several carbonate shells were sampled, usually from different organisms (e.g., oysters, pectinids, and barnacles). Some bulk and cement samples were also analyzed for better understanding the diagenetic path and its effects on $^{87}Sr/^{86}Sr$ values.

3.2. Optical microscopy (OM), scanning electron microscopy (SEM), and cathodoluminescence

Diagenetic processes or weathering could modify the pristine $^{87}Sr/^{86}Sr$ ratio of carbonate and phosphate marine minerals, providing wrong ages of mineral formation (Scasso et al., 2001; Brand et al., 2012; Ullmann and Korte, 2015). Therefore, their identification is critical for evaluating the reliability of the $^{87}Sr/^{86}Sr$ ratio, which is suitable for SIS only when post-depositional alterations can be excluded. With the aim of evaluating the impacts of diagenesis and weathering on shells, mollusk and barnacle samples from both the Chilcatay and Pisco formations have been prepared for realizing thin sections. Petrographic analyses of polished thin sections were carried out through Leica and Olympus optical microscopes. A SEM-EDS Tescan VEGA TS Univac 5136XM was used at the Department of Earth and Environmental Sciences of the Università degli Studi di Milano-Bicocca for petrographic and morphological observations, as well as for checking evidence of dissolution in the structure of the selected shells. Cathodoluminescence was performed using a CITL Optical Cathodoluminoscope at the Department of Earth Science "Ardito Desio" of the Università degli Studi di Milano, operated at about 14kV accelerating voltage and 0.5 mA gun current intensity. Cathodoluminescence observations permitted the identification of different carbonate generations, as well as of recrystallized portions of the shells (Barbin, 2013; Ullman and Korte, 2015).

Pristine shells were chosen for the age calculation, whereas shells showing a recrystallized and/or dissolved texture under both the optical and the electron microscope and a high luminescence under the cathodoluminoscope were discarded. Some recrystallized materials were also selected for $^{87}Sr/^{86}Sr$ analyses, for understanding how diagenesis changed the Sr ratio (i.e.,

diagenesis path) (Steuber, 2003). Regarding shark teeth, samples were taken from the outer enameloid layer (Enax et al., 2014), avoiding dentine, which is generally more porous and susceptible to diagenetic alteration than enameloid (Becker et al., 2008). Sr isotope measurement on the enameloid portion of shark teeth has proven to provide reliable age estimates (Becker et al., 2008; Harrell et al., 2016).

After the preliminary analyses, fifteen samples were selected from the Ct1 sequence of the Chilcatay Formation, whereas eight samples were selected from the P0 sequence of the Pisco Formation (Table 1).

3.3. Compositional and strontium isotope analyses

After microscopic investigations, the selected pristine mollusk shells and well-preserved barnacle sheaths were carefully cleaned through an ultrasonic bath in distilled water. They were bored with a Dremel micro-drill to obtain shell powder from the diagenetically unaltered portion. After an ultrasonic bath in distilled water, the selected shark teeth were also scraped as powder of their unaltered, non-porous enameloid was collected with the micro-drill. About 10-50 mg of each sample were collected for inductively coupled plasma-optical emission spectroscopy (ICP-OES) and $^{87}Sr/^{86}Sr$ analyses, taking care to avoid contaminations.

ICP-OES and $^{87}Sr/^{86}Sr$ analyses were made at the Institute für Geologie, Mineralogie und Geophysik of the Ruhr-Universität of Bochum. ICP-OES analyses were made with a Thermo Fisher Scientific iCAP 6500 DUO spectrometer for analyzing the concentrations of Sr, Ca (calcium), Mg (magnesium), Fe (iron), and Mn (manganese).

$^{87}Sr/^{86}Sr$ ratios were determined with a TIMS Finnigan MAT 262 solid source mass spectrometer with seven collectors, using a dynamic (peak-hopping) mode of measurement. The cut-off limit for a strontium run was an error of $\pm 2\sigma \leqslant 5 \times 10^{-6}$ for the $^{87}Sr/^{86}Sr$ ratio, with 100–200 ratios per run (typical duration: 110 ratios, lasting 2 h 15 min, plus the filament heating time). Two standards – NIST NBS 987 and USGS EN-1 – were used to calibrate the analysis. NIST NBS 987 was loaded directly onto the filament, to confirm the stability of the mass spectrometer. USGS EN-1, a powder derived from a modern *Tridacna* shell, underwent the same procedure as the other carbonate samples, to ensure the reproducibility of the entire analytical process. Thirty-four analyses of NIST NBS 987 resulted in a mean of 0.710240 ± 0.000023 (2σ), while 24 analyses of USGS EN-1 yielded 0.709153 ± 0.000019 (2σ). No Rb (rubidium) correction was applied, but Rb was nonetheless monitored during the entire run. When Rb levels exceeded the detection limit, the result was discarded and the measurement repeated.

The results of the $^{87}Sr/^{86}Sr$ analyses were corrected for the difference between the USGS EN-1 value used for the compilation of the reference curve (McArthur et al., 2012) and the USGS EN-1 Bochum mean value, and then converted into ages using the LOWESS Table 5, which is tied to the GTS2012 timescale (McArthur et al., 2012). The latter is still incomplete, therefore the results were rounded off to the nearest value. Following the method described by Frijia et al. (2015), when more than one sample was analyzed for a single layer, a mean $^{87}Sr/^{86}Sr$ value was calculated. The uncertainty for each stratigraphic level was calculated as 2 s.e. (i.e., standard error) from the standard deviation of the mean.

4. Results

4.1. Diagenesis evaluation for SIS analyses

The Chilcatay and Pisco formations present notably divergent patterns of carbonate preservation. In the Chilcatay Formation, fossil invertebrates are well-preserved and retain their pristine calcitic shells. In contrast, in the Pisco Formation, calcitic shells are only present in the P0 sequence, where examples of calcite recrystallization also occur, but not in P1 and P2, where mollusks are only preserved as casts (Di Celma et al., 2017). For these reasons, SIS could only be applied in the Ct1 and P0 sequences, once some caveats have been taken into account, such as in-depth petrographic and chemical analyses of the samples.

At the macroscale, all of the bivalves selected for Sr analyses from both the units appear well-preserved and composed of calcite. The oysters (Fig. 4A) are usually incided by attachment traces by sponges and/or drill holes due to the predatory action of carnivorous gastropods; in several cases, they preserve layers of unaltered nacre. The pectinids (Fig. 4B) are also well-preserved, with small borings and some barnacle attachment traces (*Anellusichnus*; Santos et al., 2005). By contrast, the barnacles (Fig. 4C) generally display obvious signs of abrasion and lack their opercula; the best-preserved and less porous portion of the shell is often constituted by the sheath, the thickened upper part of the inner wall (see Fig. 4C). Shark teeth selected for Sr analyses are moderately- to well-preserved; although two shark teeth show a breakage surface in their root, they all have an intact thin layer of enameloid that surrounds the pulp cavity and covers the surface of the tooth crown (Fig. 4D).

Microscopically, the best-preserved pectinids and ostreids are characterized by foliated and prismatic calcite, respectively (Fig. 5A, C) (Cox et al., 1971). Both also have a low, homogeneous luminescence, suggesting little or no diagenesis (Fig. 5B, D). Nevertheless, some of the analyzed ostreids also have layers that are luminescent, documenting diagenetic modifications. Microborings filled by luminescent calcite or terrigenous sediments, can be observed (Fig. 5C, D). These infills

must be avoided during sampling for Sr isotope analyses, because they could have non-pristine Sr ratio. As reported also by Crippa et al. (2016), fossil ostreids occasionally exhibit layers of sparry calcite replacing the pristine aragonitic layers (i.e., myostracum) or filling the chambers once permeated by organic matter (Fig. 5E, F). All those specimens that present similar features were discarded from the Sr analyses. Several oysters from the Ct1 and P0 sequences that exhibit a generally low luminescence have discrete luminescent layers that appear pristine and prismatic. For explaining this feature, which has also been documented in modern oysters, Barbin (1991, 2013) argued that luminescence may not be always a convincing indicator of diagenetic alteration for oysters, because the absorption of Mn^{2+} by benthic organisms depends on various factors, such as growth rate, ontogeny, bathymetry, salinity, and redox conditions. To minimize the risk of diagenetic bias, we conservatively discarded all the ostreid samples with diffuse microborings, sparry calcite, or displaying high and/or heterogeneous luminescence. By contrast, the best-preserved barnacles are characterized by low luminescence throughout the less porous portion of the wall, but high luminescence charaterized the parietal tubes, which are normally filled with secondary calcite (Fig. 5G, H). We carefully avoided to contact the parietal tubes during sampling of the barnacle shells for the strontium analysis.

Secondary electron (SE) images confirmed the presence of alternating prismatic and sparry calcite layers in some ostreid specimens (Fig. 6A, B) that were hence discarded. Other specimens either retain a pristine prismatic layer (Fig. 6C) or show clear signs of alteration such as obliteration of the pristine prismatic texture (very irregular or dissolved) and widespread, pervasive holes (Fig. 6D). Again, specimens exhibiting obvious evidence of diagenesis were discarded.

ICP-OES analysis is useful for measuring the Fe, Mn, Mg, and Sr concentrations, which in turn can be informative in terms of diagenetic imprint on carbonates. Usually, the Fe and Mn contents of carbonates increase during the diagenetic processes; on the contrary, the Mg and Sr contents generally decrease during diagenesis (Brand and Veizer, 1980; McArthur, 1994; Steuber, 1999). Fixed or static limits for Fe, Mn and Sr content have been proposed by several authors to discriminate between pristine and altered biotic low-Mg calcite of fossil shells (see Ullmann and Korte, 2015 for a recent review). However, this approach totally ignores natural variation due to spatial and temporal differences (Brand et al., 2011; Ullmann and Korte, 2015). For this reason, following the approach of Brand et al. (2012), we evaluated separately each stratigraphic horizon, comparing the chemical composition of different coeval components (i.e. optically well preserved shells, recrystallyzed shells, cemented bulk sediment enclosing or encrusting the shells) to select the best preserved material. In Figure 7A, the low Sr content of cement and bulk samples indicates a decrease in Sr during diagenesis, as reported by Frijia and Parente (2008) for diagenetically altered

marine carbonates, coupled with a decrease of the $^{87}Sr/^{86}Sr$ value. The latter trend is opposite to what is normal observed in diagenetically altered carbonates, when the interaction of the diagenetic fluids with siliciclastic rocks generally results in more radiogenic Sr isotope values (McArthur, 1994). The decrease of the Sr ratio in the diagenetically altered calcite of the studied successions can be due to the pre-Miocene seawater-derived brine circulating within the sedimentary sequence, as reported by Gioncada et al. (2018a). This decrease of the Sr isotope ratio from pristine to diagenetic calcite confers older Sr ages to the diagenetically altered samples. The abundance of Fe and Mn in the diagenetic samples (see Fig. 7B, C, D) conforms to the expected pattern of increasing Fe and Mn concentration during diagenesis. The above reconstruction of the diagenetic paths helps in assessing the preservation of each sample. Indeed, samples displaying the same degree of morphological preservation but higher concentrations of Sr and lower concentrations of Fe and Mn might be considered as retaining the pristine Sr isotope ratio of seawater.

4.2. Strontium Isotope Stratigraphy of the Ct1 sequence

After discarding all those shells that may have been affected by post-depositional modifications, fifteen samples from the Ct1 sequence of the Chilcatay Formation (Ullujaya and Roca Negra) were selected for $^{87}Sr/^{86}Sr$ analysis (Table 1). From each locality, Sr isotope analyses of different well-preserved shells yielded similar Sr isotope values. The internal consistency of the Sr isotope ratios of different samples from the same stratigraphic level can be regarded as a strong argument supporting the preservation of the original seawater $^{87}Sr/^{86}Sr$ value (McArthur et al.,1994; Steuber, 2003).

At Roca Negra, we sampled three well-preserved oysters (PN-OST, PN-GIO1, PN-GIO2) from the *Ct1c* facies association. The three specimens came from a single bed (PN Oyster bed; Fig. 3C) close to the base of the formation, ca. seven meters above an ash layer (PN-T2) dated to 19.25 ± 0.05 Ma by Bosio et al. (in press). Sr isotope analyses give similar $^{87}Sr/^{86}Sr$ results (Table 1) and the mean value gives a preferred age of 18.50 with a range of uncertainty spanning between 18.85 and 18.15 Ma (Table 2).

At Ullujaya, we sampled 12 specimens from the *Ct1a* facies association, between a basal ash layer (UJA-T35) dated to 19.00 ± 0.28 Ma by Bosio et al. (in press) and a second ash layer in the overlying *Ct2b* facies association (SOT-T3; Fig. 3C) dated to 18.02 ± 0.07 Ma by Di Celma et al. (2018b).

Three samples – a barnacle sheath (UL-LIVa), composite sample of two pectinids (UL-LIVb), and a further, single pectinid (UL-LIVc) – were chosen from a layer 9 m above the base of the measured section (hereafter: abs), named "mollusk- and barnacle-rich horizon" by Collareta et al.

(2019) (Fig. 3C). The mean value of the $^{87}Sr/^{86}Sr$ values gives again a preferred age of 18.50 Ma and an uncertainty that ranges from 18.60 to 18.40 Ma for this horizon (Table 2).

At 17 m abs, in occurrence of the key bed C (KbC of Di Celma et al., 2018b) (Fig. 3C), nacre layers of two well-preserved oysters were selected (UJA-LIVC1, UJA-LIVC3) together with a barnacle sheath (UJA-LIVC4) (Table 1). In addition, a sample of the cemented bulk sediment from the outer part of an oyster shell was also analyzed (UJA-LIVC2). Considering only the ages from the pristine shells, we obtained similar $^{87}Sr/^{86}Sr$ values and Burdigalian ages (see Tables 1, 2). The bulk sample UJA-LIVC2 shows a lower value of $^{87}Sr/^{86}Sr$ ratio and gives older ages (i.e., 21.45–21.00 Ma), suggesting an ageing effect caused by diagenesis, as discussed above. The age range of the key bed C is thus comprised between 18.50 and 18.10 Ma, with a preferred age of 18.30 Ma (Table 2). The SIS age of this bed is apparently younger than that of the bed below (i.e., the mollusk- and barnacle-rich horizon), dated at ca. 18.50 Ma, although the two obtained age ranges are still largely overlapping.

Three samples were selected for the UL-D4 bed, a carbonate-rich horizon located 25 m abs (see Fig. 3C): UL-D4a, from prismatic layers of an oyster; UL-D4b, from a barnacle sheath; UL-D4c, the bulk sediment cemented within the same barnacle shell (Table 1). The bulk sample, UL-D4c, exhibits a relatively low $^{87}Sr/^{86}Sr$ value and, consequently, a relatively older age value (i.e., 19.60–19.20 Ma), indicating that diagenesis had an ageing effect on the shells also in this bed. The shell samples from the UL-D4 bed did not return consistent values of $^{87}Sr/^{86}Sr$ (see Supplementary Material) and they were discarded from the age determination.

Finally, at 30 m abs, we took two samples (UJA-2a and UJA-2b) from different layers of a single oyster shell, collected from key bed B of Di Celma et al. (2018b) (Fig. 3C). The mean $^{87}Sr/^{86}Sr$ value provides a preferred age of 18.30 Ma for the key bed B, with a maximum age of 18.60 Ma and a minimum age of 18.00 Ma, in agreement with the levels dated below (Table 2).

The $^{87}Sr/^{86}Sr$ ages calculated for each bed give a Burdigalian age for the Ct1 sequence of the Chilcatay Formation. The time resolution of the SIS method is not sufficient to discriminate the age of these closely spaced horizons. However, the preferred ages of the sampled levels tend to be younger according to the stratigraphy.

4.3. Strontium Isotope Stratigraphy of the P0 sequence

Carbonates from the P0 sequence are poorly preserved, resulting in just five samples from a ~10 m-thick stratigraphic section located north of Cerro Submarino (Fig. 3D). Given the scarcity of suitable carbonate samples, we also collected samples from well-preserved shark teeth (phosphates). They show $^{87}Sr/^{86}Sr$ values that are consistent with those of the pristine carbonate

samples (see Fig. 7, Table 1). Taking also into account the limited thickness of the P0 deposits exposed at Cerro Submarino, we considered all the collected samples as belonging to a single level and we calculated a unique $^{87}Sr/^{86}Sr$ age starting from five different samples.

Three shark teeth were chosen from the lower portion of the P0 sequence, a few meters above the PE0.0 unconformity, in the plain northward of Cerro Submarino, here informally called "Tooth plain" (see Fig. 3D). Of the analyzed teeth, Tooth 1 is a lower anterolateral tooth of *Cosmopolitodus hastalis*, Tooth 2 is an upper tooth of the same species (Fig. 4D), and Tooth 3 is a lower tooth of *Isurus oxyrinchus* (see Table 1). The three resulting $^{87}Sr/^{86}Sr$ values are slightly different from each other, but there are no morphological or chemical reasons to prefer one tooth over another and all these samples were selected for the final age calculation.

A few meters above the PE0.0 unconformity, a calcite nodule from within a baleen whale mandible (SUB-2, Table 1) was first selected following the hypothesis of precipitation during early diagenesis, similarly to what hypothesized for the dolomite nodules found inside bone cavities in the Pisco Formation (Gariboldi et al., 2015; Gioncada et al., 2016). However, SUB-2 has a very low $^{87}Sr/^{86}Sr$ value similar to the late diagenetic cements, suggesting a late diagenetic imprint of this nodule, and therefore it was discarded. Close to this sample, an oyster was collected (SUB-5, Fig. 3D), and the thin nacre layers were drilled, giving an $^{87}Sr/^{86}Sr$ value similar to those of the shark teeth. Finally, a few meters above, three samples were selected from level SUB-8bis (see Fig. 3D): one from the presumably pristinely preserved sheath of a barnacle (SUB-8bis1), one from the bulk sediment cemented inside the same barnacle shell (SUB-8bis2), and the last one from a completely recrystallized shell of a bivalve (SUB-8bis3) (Table 1). The samples from the bulk and the recrystallized bivalve indicate that, as in the Ct1 sequence, diagenesis resulted in a substantial reduction of the $^{87}Sr/^{86}Sr$ value and a consequent increase of the calculated ages. In turn, the pristine calcite of the barnacle sheath shows an $^{87}Sr/^{86}Sr$ value similar to the other pristine samples and was considered for the final age calculation.

Discarding samples affected by diagenesis, a mean $^{87}Sr/^{86}Sr$ value can be calculated starting from 5 samples collected from the lower part of the P0 sequence. Together, the three shark teeth, SUB-5, and SUB8bis1 result in a mean age of 13.45 Ma, with an uncertainty time range spanning between 14.80 and 12.45 Ma. Consequently, by means of SIS, the P0 sequence can be placed in the Langhian–Serravallian (Table 2). The age of the P0 sequence is now resolved, but poorly constrained because of the scarcity of well-preserved specimens (allowing for just a few dated samples) and the intrinsic limits of the SIS method.

4.4. Thermophilic invertebrates and vertebrates from the P0 sequence

The thermophilic fossils identified from the P0 strata exposed in the vicinities of Cerro Submarino include members of both the invertebrate and vertebrate assemblages (Fig. 8).

Among the invertebrates, three cypraeid specimens could be identified as belonging to *Muracypraea ormenoi*, an extinct species that has been interpreted as a thermophilic taxon that shared the environmental and climatic preferences of extant *Muracypraea mus* (DeVries et al., 2006). Although the holotype of *M. ormenoi* was collected from Chilcatay beds, another specimen was reported by DeVries et al. (2006) from undifferentiated strata of the Pisco Formation in the vicinities of Cerro Submarino, where the P0 strata are mostly exposed (Di Celma et al., 2017, 2018a). Unfortunately, the poor preservation state of the inner mold observed by us does not allow an unambiguous identification of these cipraeid specimens at the genus and species levels (see Fig. 8A–C). A specimen of the gastropod *Ficus* cf. *distans* was also found in these strata (personal communication by T.J. DeVries, in Di Celma et al., 2017); according to DeVries and Frassinetti (2003) and Nielsen and Glodny (2009), *Ficus distans* should be regarded as a thermophilic member of a generally warm-water genus. Seven specimens belonging to the family Architectonicidae were also found (see Fig. 8H, I, J). Architectonicids were considered by Nielsen and Frassinetti (2007) as tropical and subtropical gastropods. Finally, the P0 invertebrate assemblage from the Cerro Submarino area includes the only known coral occurrence from the East Pisco Basin, reported in this paper for the first time. The single examined specimen consists of several tiny corallites (up to 4.5 mm in calicular diameter) settled on two large barnacle shells (Fig. 8F, G). The distal part of the corallites is badly preserved, however their size, shape, septal number, septal arrangement, and stolon-like connections allow us to ascribe them to a colony of the tropical to warm temperate family Rhizangidae (Cairns et al., 2005). High affinities with the genus *Culicia* have been noted, but further (better preserved) specimens must be analyzed to confirm the genus-level identification.

With respect to the vertebrates, teeth and dermal spines belonging to various taxa of more or less thermophilic cartilaginous fishes have been collected from the studied P0 exposures. The elasmobranch assemblage includes abundant teeth of the requiem shark *Carcharhinus*, among which *Carcharhinus brachyurus* and *Carcharhinus* cf. *leucas* stand out. Most extant species of *Carcharhinus* are confined today to warm-temperate to tropical waters, and *C. leucas* is especially known as a tropical to sub-tropical species (Simpfendorfer and Burgess, 2009); in turn, *C. brachyurus* is a more temperate species that thrives in relatively cooler waters (Duffy & Gordon, 2003). Another carcharhinid genus, *Galeocerdo*, is known from the P0 by several teeth of the extinct species *Galeocerdo aduncus*, whose closest living relative (i.e., the tiger shark *Galeocerdo cuvier*) is known as a tropical/subtropical fish (Ferreira and Simpfendorfer, 2019); similar considerations also apply to the sole extant species of *Anoxypristis* (i.e., the knifetooth sawfish

Anoxypristis cuspidata; D'Anastasi et al., 2013), a genus of rays that is represented in the lower Pisco strata by several rostral spines that could not be determined at the species level. A predilection for tropical and warm-temperate waters is also regarded as characteristic of the extinct sharks *Carcharocles megalodon* (Purdy, 1996; Aguilera and Aguilera, 2004; but see also Pimiento et al., 2013, for a different interpretation) and *Hemipristis serra*; in particular, the extant snaggletooth shark *Hemipristis elongata* – the only living species of *Hemipristis* – is a tropical coastal shark that inhabits inshore and offshore waters up to 30 m depth (Compagno, 1984; Pimiento et al., 2013).

5. Discussion

5.1. SIS age of the P0 strata

Strontium data confirm a Burdigalian age for the Ct1 sequence of the Chilcatay Formation, in line with the ^{39}Ar–^{40}Ar dating on tephra and with the biostratigraphic results given by diatoms and silicoflagellates from the localities of Roca Negra and Ullujaya (Belia and Nick, 2016; Di Celma et al., 2018b; Bosio et al., in press). This SIS ages are in agreement with the older age of 19.25 ± 0.05 Ma of the PN-T2 ash layer dated by Bosio et al. (in press) at the base of the Chilcatay Formation at Roca Negra, and with the ages of 19.00 ± 0.28 Ma and 18.02 ± 0.07 Ma obtained from ash layers at the base and at the top of the Ullujaya succession (UJA-T35 and SOT-T3, respectively, of Bosio et al., in press; Di Celma et al., 2018b). In addition, they are consistent in themselves, with stratigraphically lower beds yielding older or at least matching ages, although this observation is limited by the broadly overlapping error ranges for each layer. Overall, the consistency of the strontium dates from the Ct1 sequence confirms the suitability of the method for dating sediments in the East Pisco Basin.

Our results also provide the first direct evidence for the age of the P0 sequence of the Pisco Formation in the study area, placing this unit in the middle Miocene (Langhian–Serravallian). This estimate is again consistent with the broad age constraints (ca. 9.5–18.0 Ma) imposed by previous radiometric dating and biostratigraphy (Di Celma et al., 2018b; Bosio et al., in press).

Current knowledge about both invertebrate and vertebrate assemblages of the P0 sequence is poorly informative in terms of chronostratigraphy; that said, a middle Miocene age matches the general aspect of the P0 oryctocoenosis. Indeed, mollusks suggest similarities with the lower–middle Miocene Navidad Formation of central Chile (DeVries and Frassinetti, 2003). Likewise, the baleen whale assemblage appears remarkably more archaic than that of the overlying P1 and P2 sequences (Di Celma et al., 2017; Marx et al., 2017b), with the presence of *Pelocetus* sp. (Bianucci et al., 2019) matching other early–middle Miocene occurrences of this genus from Japan (Kimura et

al., 2007) and eastern North America (Kellogg, 1965). The occurrence of the archaic cetotheriid *Tiucetus rosae*, the earliest branching member of a mysticete family (i.e., Cetotheriidae) that flourished in late Miocene times (Marx et al., 2019b), also characterizes the P0 oryctocoenosis, possibly hinting at a middle Miocene age. Also notable is the abrupt increase in baleen whale abundance relative to the underlying Chilcatay Formation, which appears to reflect a global early–middle Miocene pattern (Bianucci et al., 2018b; Marx et al., 2019a). Sharks and rays are of limited utility, as all the taxa recognized so far exhibit a stratigraphic range which spans for most of the Miocene at least. As a consequence, all the elasmobranch taxa recognized from the P0 sequence but *Carcharocles megalodon* and *Hemipristis serra,* are known from both the lower Miocene Chilcatay strata exposed in the Ullujaya-Zamaca areas (Bianucci et al., 2018b; Di Celma et al, 2018b, 2019; Landini et al., 2019) and the upper Miocene Pisco strata (P1 and P2 sequences) exposed at Cerro Colorado (Di Celma et al., 2017; Landini et al., 2017a, b). *Carcharocles megalodon*, whose fossil record starts with the Burdigalian (Carrillo-Briceño et al., 2019, but see also Perez et al., 2019, at this regard), is well-known from the P1 and P2 beds but absent from the Chilcatay Formation, where the genus *Carcharocles* is represented by the more archaic species *C. chubutensis*. Conversely, *Hemipristis serra* is present in the Burdigalian deposits of the Chilcatay Formation, whereas it is absent from the younger P1 and P2 sequences, although it is known from outside the East Pisco Basin in deposits as young as the early Pleistocene (Ebersole et al., 2017).

5.2. Paleoclimatic significance of the P0 fossil assemblage

Previous investigations on the invertebrate assemblages from the P0 and P1 sequences revealed a marked faunal change coincident with the PE0.1 unconformity (Di Celma et al., 2017) (Fig. 3D). Specifically, species such as *Ficus distans* and *Miltha* cf. *vidali*, which also occur in the Chilcatay Formation, associate P0 with the Navidad Formation of Chile, rather than the Panamic fauna (DeVries, 2002, 2007; DeVries and Frassinetti, 2003). According to Nielsen and Glodny (2009), an interpretation of the mollusk assemblage from the Navidad Formation in the light of the ecological preference of extant genera suggests water temperatures of at least 20°C for the Navidad area (34°S) during the early-middle Miocene. Indeed, the presence in the P0 sequence of warm-water or tropical taxa such as Architectonicidae and Cypraeidae (DeVries and Frassinetti, 2003; Nielsen and Frassinetti, 2007) (see Fig. 8A–C, H–J), as well as the occurrence of *Ficus*, indicates a warm-water, tropical paleoenvironment for this unit. This is also supported by the finding of the only coral colony ever collected from the East Pisco Basin, belonging to the family Rhizangiidae (Fig. 8F, G). Rhizangiid scleractinians are the most common corals in shallow-water tropical and warm temperate environments of the eastern Pacific; in particular, the P0 specimens appear as

morphologically close to the Miocene to Holocene genus *Culicia*, which currently lives in the tropical waters of the Indo-Pacific region (Cairns et al., 2005). On the whole, these observations support the paleoclimatic reconstruction proposed by DeVries and Frassinetti (2003), who hypothesized warm-water conditions for the southern Peruvian coast during the early and middle Miocene, in contrast with the cooler conditions during the late Miocene and early Pliocene.

Our data on the composition of the vertebrate oryctocoenosis from P0 also indicate tropical to subtropical thermal affinities for the paleoenvironment. In particular, among the recognized taxa, the knifetooth sawfish *Anoxypristis* is currently known as a nectobenthic organism that inhabits coastal and estuarine warm-water environments (e.g., D'Anastasi, 2013). Similarly, the extinct snaggletooth shark *Hemipristis serra* (Fig. 8D, E) is one of the most common chondrichthyan taxa in low-latitude neritic deposits of the Neogene, and its closest extant relative (*Hemipristis elongata*) is known as a tropical nearshore shark that inhabits coastal waters not deeper than about 30 m (e.g., Compagno, 1984). Interestingly, there are no late Miocene occurrences of *H. serra* from the East Pisco Basin, even though this species persisted till the early Pleistocene in many tropical/subtropical chondrichthyan assemblages worldwide, and has even been recorded from the late Miocene strata of the Miramar Formation of northern Peru (Apolín et al., 2004).

It should be noted that the deposition of the P0 strata likely took place during the last phases of the Middle Miocene Climatic Optimum (= MMCO, the last major warming interval of the Cenozoic, occurred between 17 and 14 Ma; Loughney et al., 2019, and references therein) or the onset of the Middle Miocene Climatic Transition (= MMCT, the subsequent interval of gradual change towards cooler climatic conditions), i.e., during a period of globally high temperatures relative to the modern. As reported above, the warm, tropical paleoenvironment here reconstructed for the P0 sequence sets it apart from the remainder of the Pisco Formation, which is thought to reflect a cooler setting (e.g., Dunbar et al., 1990; DeVries and Frassinetti, 2003; Amiot et al., 2008; Di Celma et al., 2017). In particular, oxygen-isotope analyses on phosphatic remains of marine vertebrates from upper Miocene horizons of the Pisco Formation exposed in the Ica desert (i.e., the CLB vertebrate level of Muizon and DeVries, 1985; referred to the late Miocene according to Di Celma et al., 2017) and the Sacaco area (i.e., the ELJ, AGL, SAS and SAO vertebrate levels of Muizon and DeVries, 1985; late Miocene in age according to Ehret et al., 2012) have revealed marine paleotemperatures that, on the whole, match those observed today off the coasts of Peru (Amiot et al., 2008). Overall, this pattern is suggestive of relatively cool conditions along the Peruvian coast during the late Miocene, which could be regarded as reflecting the post-MMCO trend of global cooling that culminated with ocean temperatures dipping to near-modern values around 7 and 5.4 Ma (Herbert et al., 2016). Furthermore, since the upper Miocene deposits of the

Pisco Formation contain abundant diatomaceous siltstones that indicate the existence of high primary productivity resulting from strong and persistent coastal upwelling conditions (Dunbar et al., 1990; DeVries and Frassinetti, 2003; Di Celma et al., 2017), it is reasonable to hypothesize that the above pattern is also representative of a major regional oceanographic change – i.e., a middle or early late Miocene strengthening of the Humboldt Current (see also conclusion in Amiot et al., 2008). This change might also explain the local disappearance of *H. serra*, whose range likely contracted northwards as colder conditions took hold along the coasts of southern Peru.

6. Conclusions

Along the western side of the Ica River (Peru), the age of deposition of the P0 sequence, the lowest stratigraphic unit of the Pisco Formation, was an uncertain and debated issue. In the present study, Strontium Isotope Stratigraphy was applied on carbonates and phosphates of both the Chilcatay and Pisco formations for resolving the age of this paleontologically significant unit.

In this work, we provide new Burdigalian ages (18.8–18.0 Ma) for the Ct1 sequence of the Chilcatay Formation and propose a Langhian–Serravallian age (14.8–12.4 Ma) for the P0 sequence of the Pisco Formation (see Fig. 9). This estimate is consistent with the relatively archaic aspect of the fossil assemblage from the lower Pisco strata, and provides a time constraint for a marked faunal change occurring across the PE0.1 unconformity. With respect to the Chilcatay Formation, the obtained Sr ages perfectly agree with previous ^{39}Ar–^{40}Ar ages on tephra layers and biostratigraphic results, confirming the feasibility of SIS in the studied deposits. This gives further reliability to the middle Miocene ages obtained by means of Sr isotope analyses for the P0 sequence.

Not least, both the invertebrate and vertebrate fossil assemblages indicate that the P0 sequence was deposited in a warm-water environment resembling that of the underlying Chilcatay Formation, but contrasting with the cooler and more productive setting of the overlying P1 and P2 sequences. We suggest that the marked cooling distinguishing P0 from the remainder of the Pisco Formation may reflect both the late Miocene trend of global cooling and a middle–late Miocene strengthening of the Humboldt Current.

Acknowledgments

The authors are grateful to T.J. DeVries for information and helpful discussions on the age and paleoenvironment of the Pisco Formation. Special thanks to W. Landini, O. Lambert and C. de Muizon for interesting discussions on the vertebrate fauna, and to D. Basso and G. Coletti for helpful discussions on mollusk systematics and strontium dating. The authors would like to thank

also R. Varas-Malca and W. Aguirre for assistance in the field, and D. Buhl for running the ICP-OES and Sr isotope analyses at Bochum University. The authors wish to thank the journal reviewers F. Núñez for his valuable contribution and J. McArthur for the helpful criticism and for sharing the LOWESS 5 Table.

Funding

This study was supported by grants from the Italian Ministero dell'Istruzione dell'Università e della Ricerca (PRIN Project 2012YJSBMK) to G.Bi.; from the Università degli Studi di Milano-Bicocca to E.M. (2017-ATE-0466); from the Università di Pisa to G.Bi. (PRA_2017_0032); and an EU Marie Skłodowska-Curie Global Postdoctoral fellowship (656010/ MYSTICETI) and an Australian Research Council DECRA fellowship (DE190101052) to F.G.M.

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Figure captions

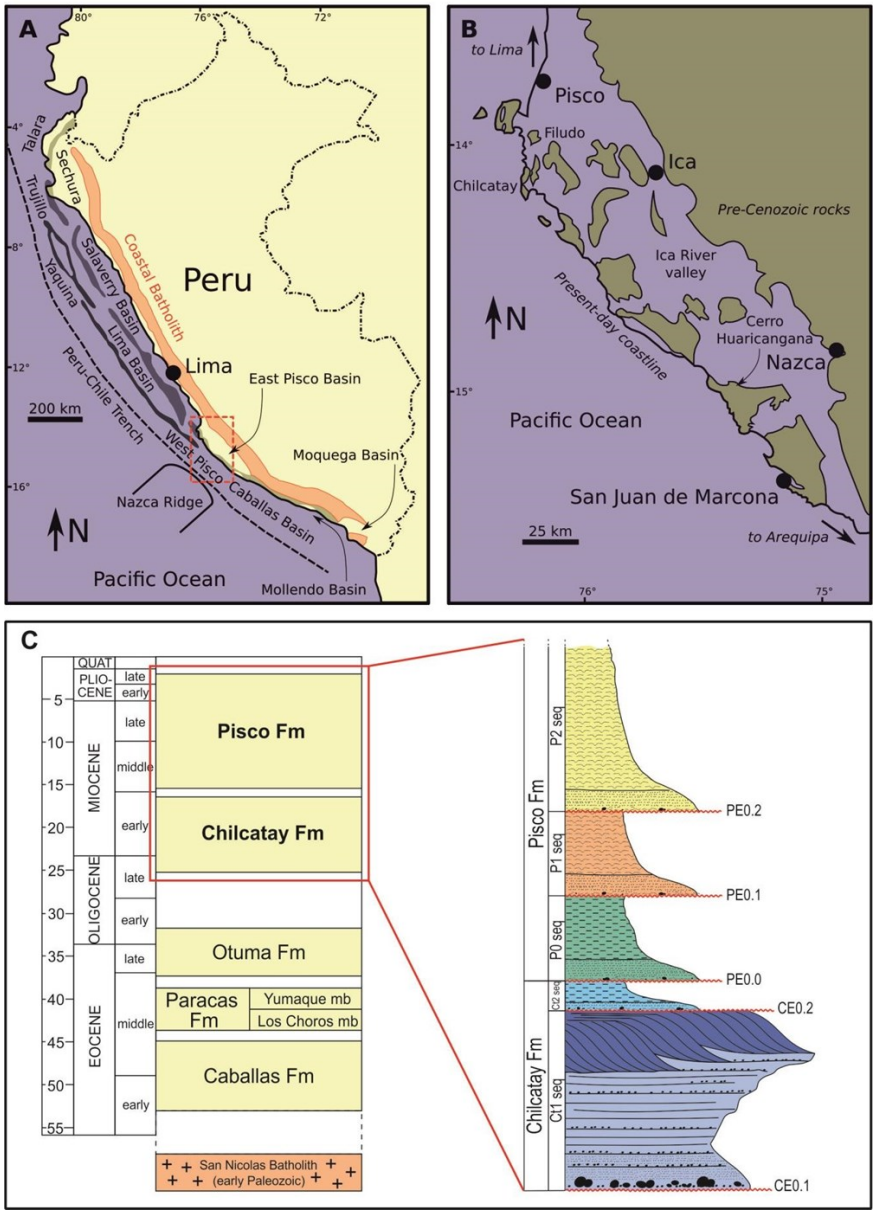

Figure 1. A. Map of the major Cenozoic sedimentary basins along the Peruvian coast. Major structural highs are the Coastal Batholith, the Outer Shelf High and the Upper Slope Ridge. Redrawn and modified from Travis et al. (1976) and Thornburg and Kulm (1981). **B.** Paleoenvironmental reconstruction of the Peruvian coast during the Miocene. Islands are hypothesized on the basis of the distribution of the pre-Cenozoic rocks. Redrawn and modified from DeVries and Schrader (1997). **C.** Schematic stratigraphic column of the East Pisco Basin (from Eocene to Recent, ages are in Ma) with a close-up of the Chilcatay and Pisco formations and their internal subdivisions in sequences.

Figure 2. Satellite image and position of the localities of Cerro Submarino (), Ullujaya (), and Roca Negra () along the western side of the Ica River (Ica desert). Based on a Google Earth image (© 2019 Maxar Technologies).

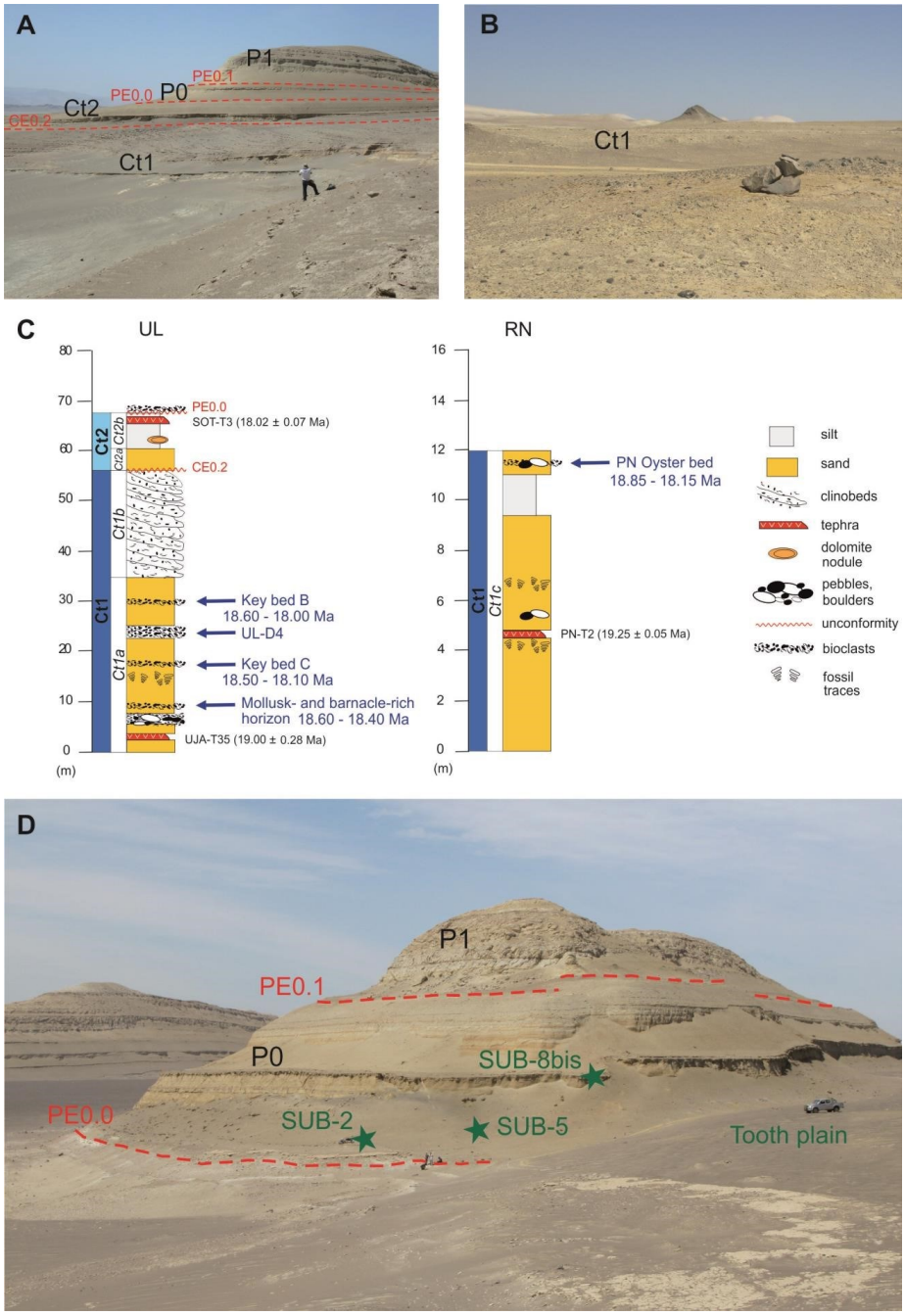

Figure 3. A. Field photo of the Chilcatay and Pisco strata at the locality of Ullujaya, **B.** Field photo of the Chilcatay basal strata at the locality of Roca Negra. **C.** Schematic stratigraphic sections measured at the Chilcatay outcrops of Ullujaya (UL) and Roca Negra (RN) that show the stratigraphic position of i) the investigated beds and their $^{87}Sr/^{86}Sr$ ages and ii) the ash layers with the $^{39}Ar–^{40}Ar$ ages obtained by Bosio et al. (in press). **D.** Field photo at Cerro Submarino, with the location of the sampled beds (green stars) collected from the P0 sequence. The PE0.0 and PE0.1 unconformities are traced in red.

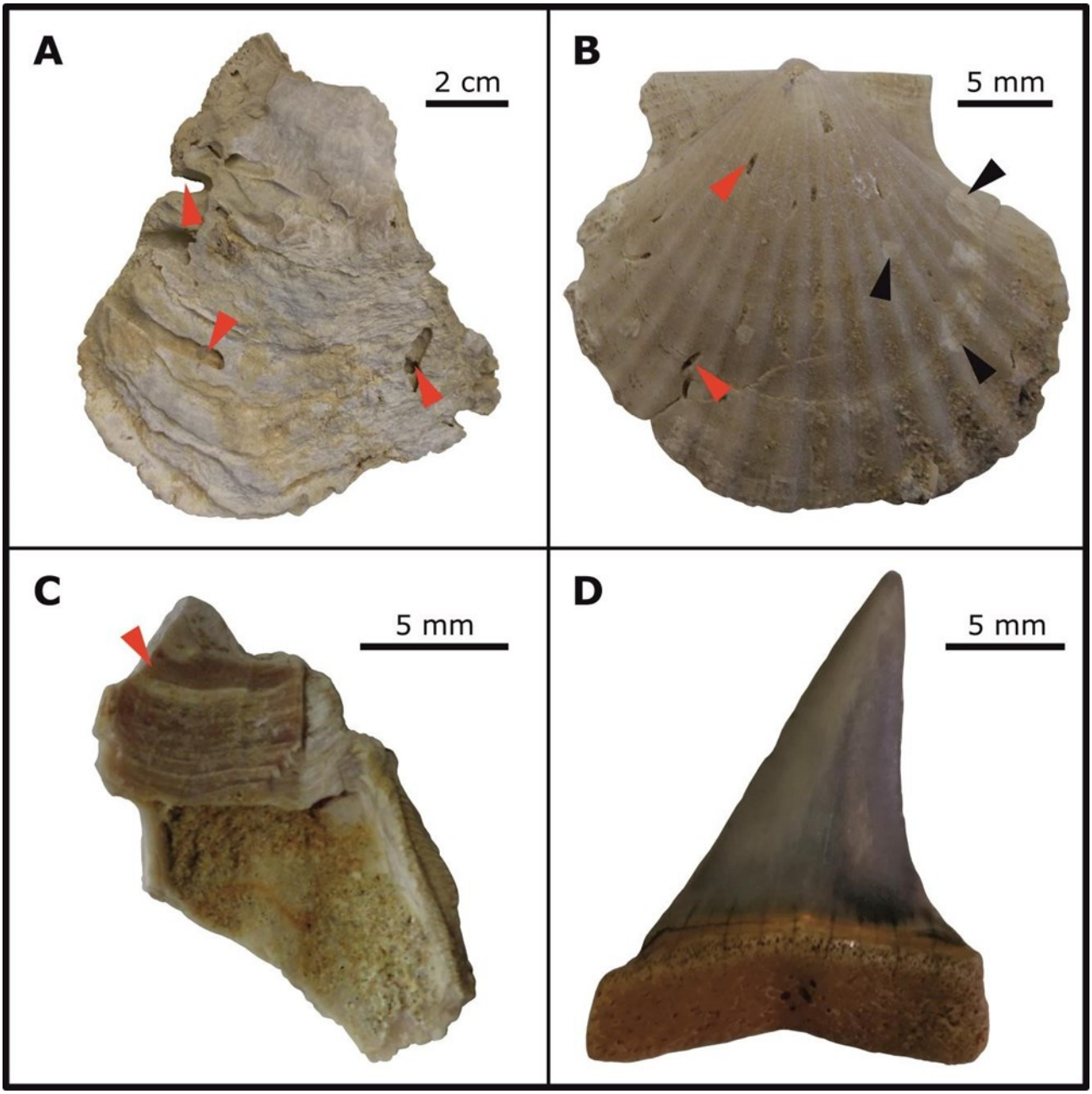

Figure 4. A. Oyster shell from the PN Oyster level in the Ct1 sequence at Roca Negra. Note the predation holes on the outer part of the shell pointed out by red arrows. **B.** Pectinid specimen from the mollusk- and barnacle-rich horizon in the Ct1 sequence at Ullujaya. Note the small drill holes (red arrows) and the barnacle attachment scars (i.e., *Anellusichnus*) (black arrows). **C.** Barnacle plate from the Key bed C in the Ct1 sequence at Ullujaya. Note the lamellar sheath (i.e., the thickened upper part of the inner wall pointed out by the red arrow) from which the SIS samples were taken. **D.** Tooth of *Cosmopolitodus hastalis* from the P0 sequence at Cerro Submarino.

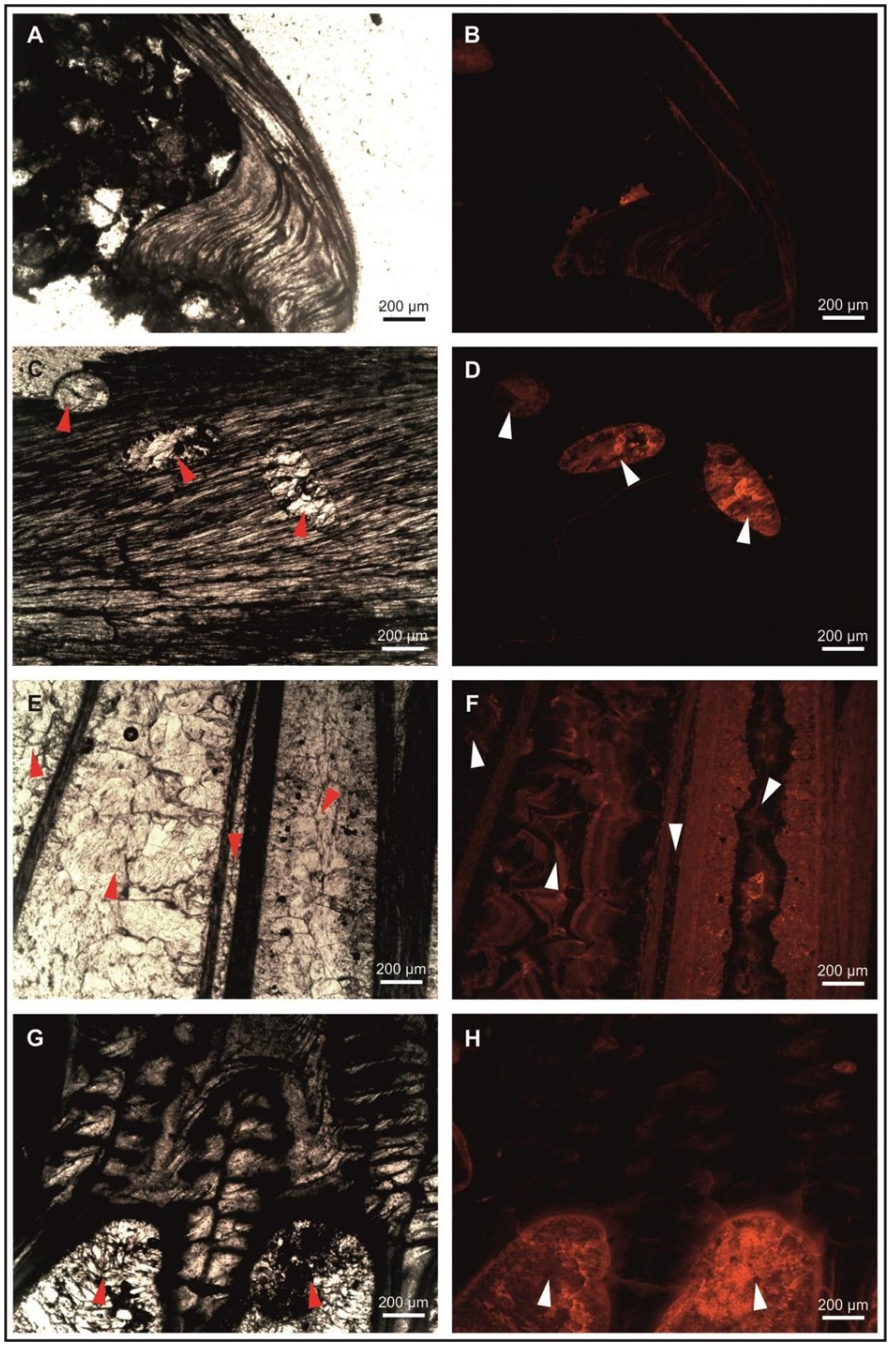

Figure 5. Transmitted light and cathodoluminescence microscopical photos. **A, B.** Pectinid shell with a homogenous luminescence. **C, D.** Oyster prismatic layer with a low and homogeneous luminescence, punctuated by microborings with a high luminescence (red and white arrows). **E, F.** A poorly preserved oyster shell characterized by an alternation of high luminescent layers of sparry calcite (red and white arrows). **G, H.** Transverse thin section of a barnacle shell showing tree-like interlaminate figures and the parietal tubes filled by diagenetic calcite with a high luminescence pointed out by red and white arrows.

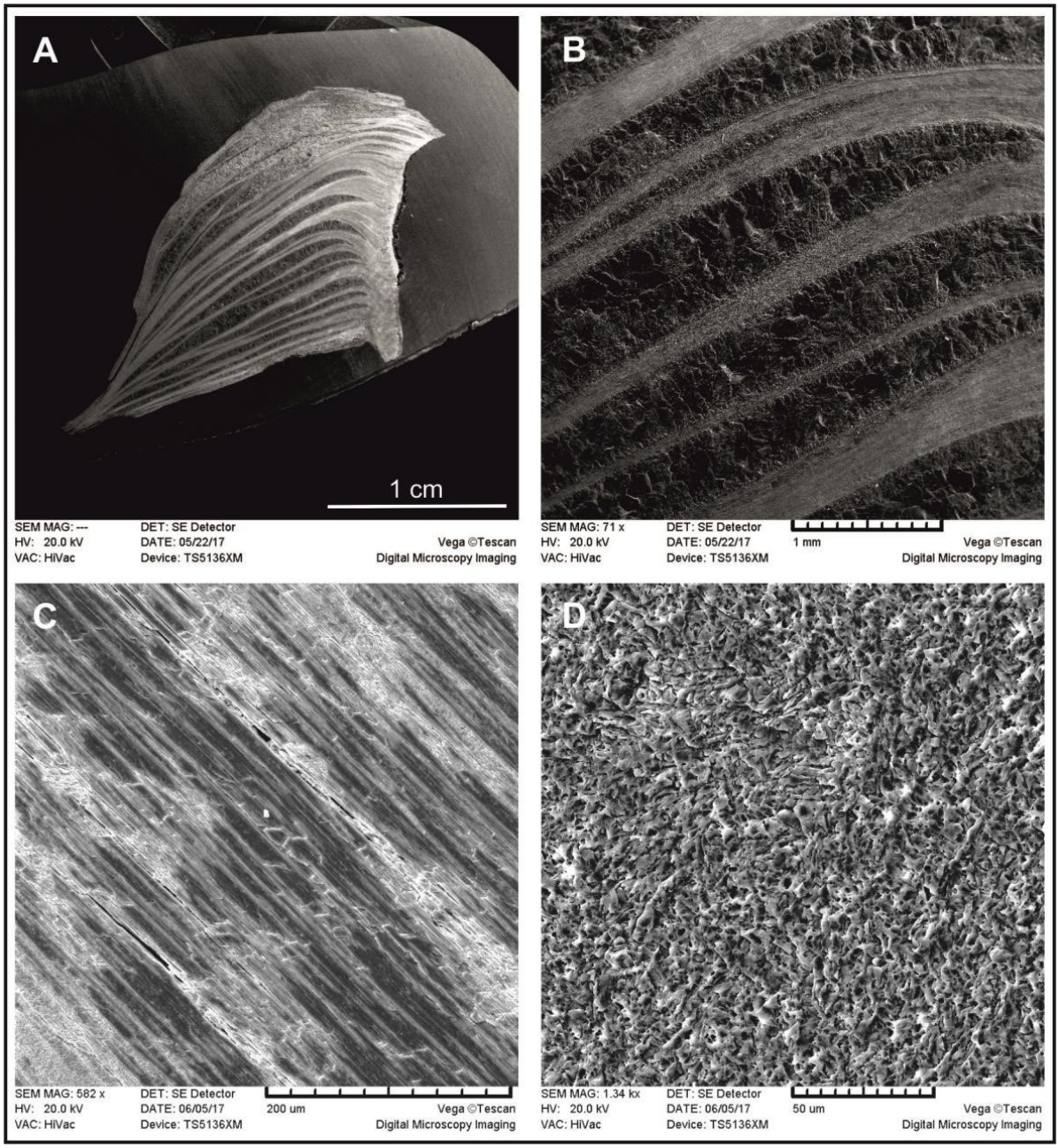

Figure 6. A. SE (Secondary Electron) image of an oyster cross-section. Note the alternation of different layers. **B.** Close-up of the oyster shell depicted in panel A. Note the alternation of prismatic layers (light grey) and sparry calcite layers (dark grey). **C.** SE image of a well-preserved prismatic layer of an oyster. **D.** SE image of a poorly preserved oyster shell exhibiting dissolution features as obliteration of the pristine prismatic texture and widespread, pervasive holes.

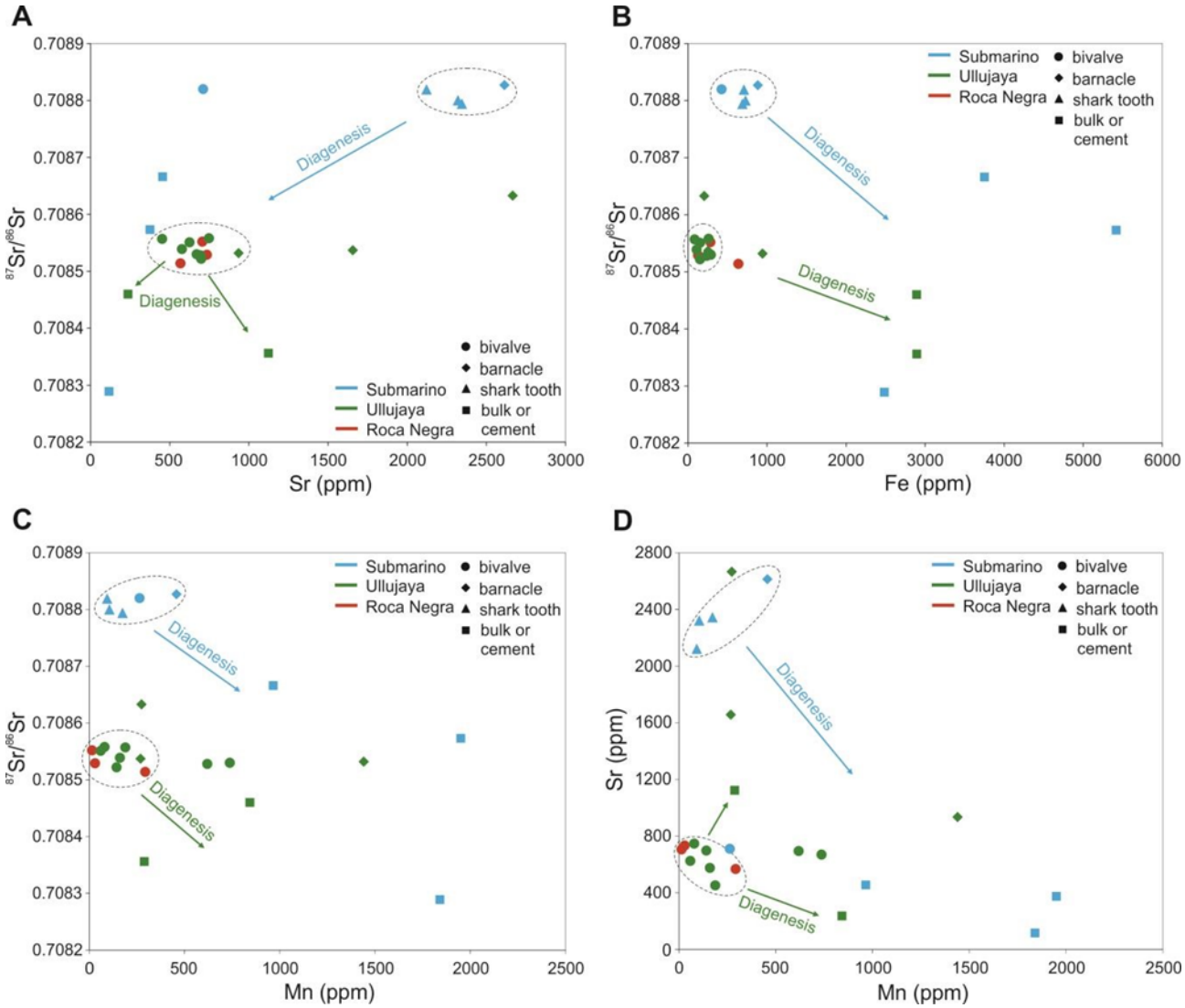

Figure 7. Diagenetic path diagrams, showing the major trends of diagenetic alteration in multi-component plots. **A.** $^{87}Sr/^{86}Sr$ ratio vs Fe concentration. **B.** $^{87}Sr/^{86}Sr$ ratio vs Mn concentration. Ellipses show well preserved (continuous line) and altered (dotted line) samples. Arrows indicate trends of diagenetic alteration.

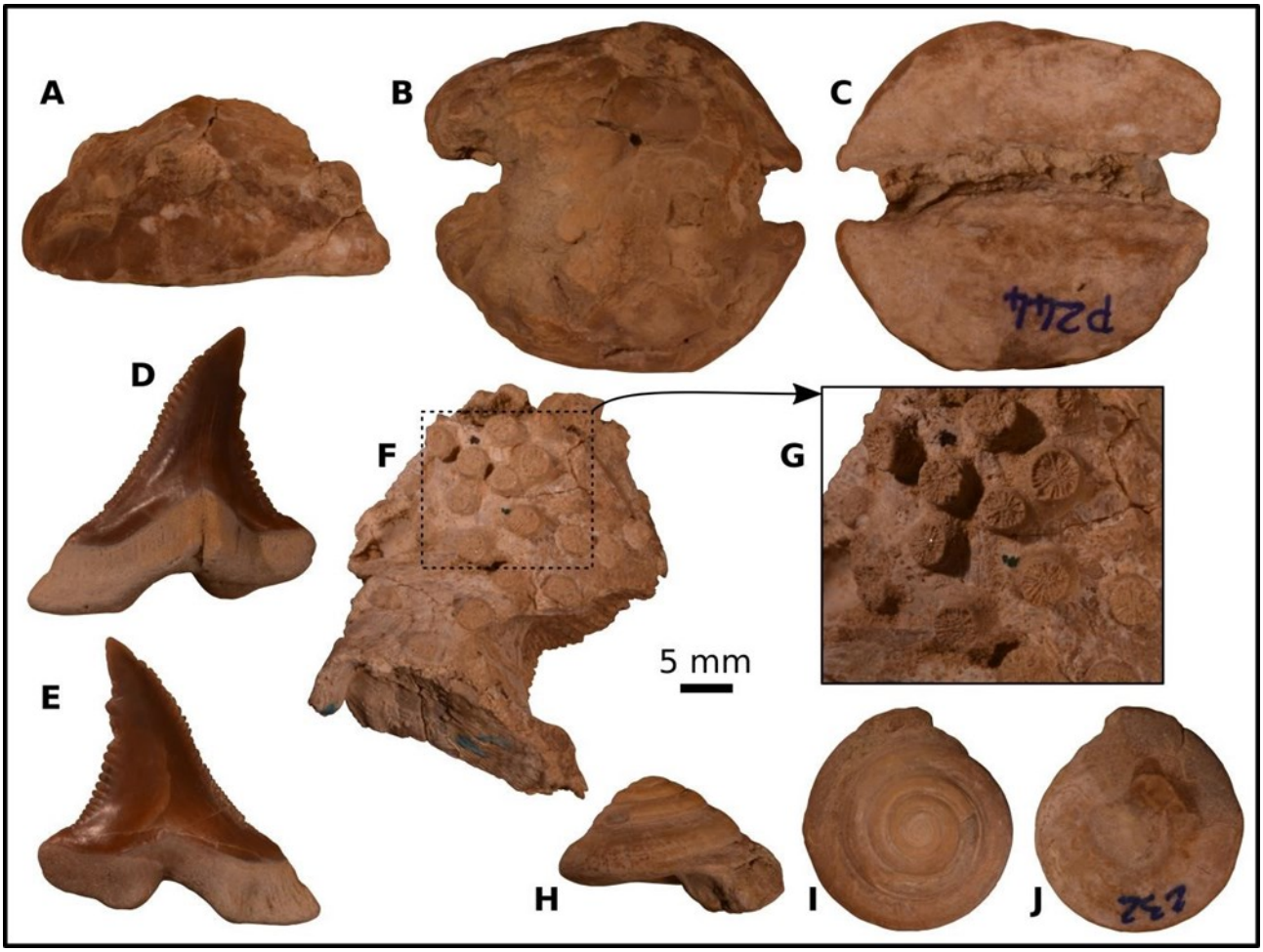

Figure 8. Fossils of warm-water taxa from the P0 sequence. **A, B, C.** Cypraeid specimen in lateral (A), dorsal (B), and apertural (C) views. **D, E.** Tooth of *Hemipristis serra* in lingual (A) and labial (B) views. **F.** Rhizangiid corallites encrusting a cluster of barnacle shells. **G.** Close-up of the corallites depicted in panel F. **H, I, J.** Architectonicid specimen in lateral (H), dorsal (I), and ventral (J) views.

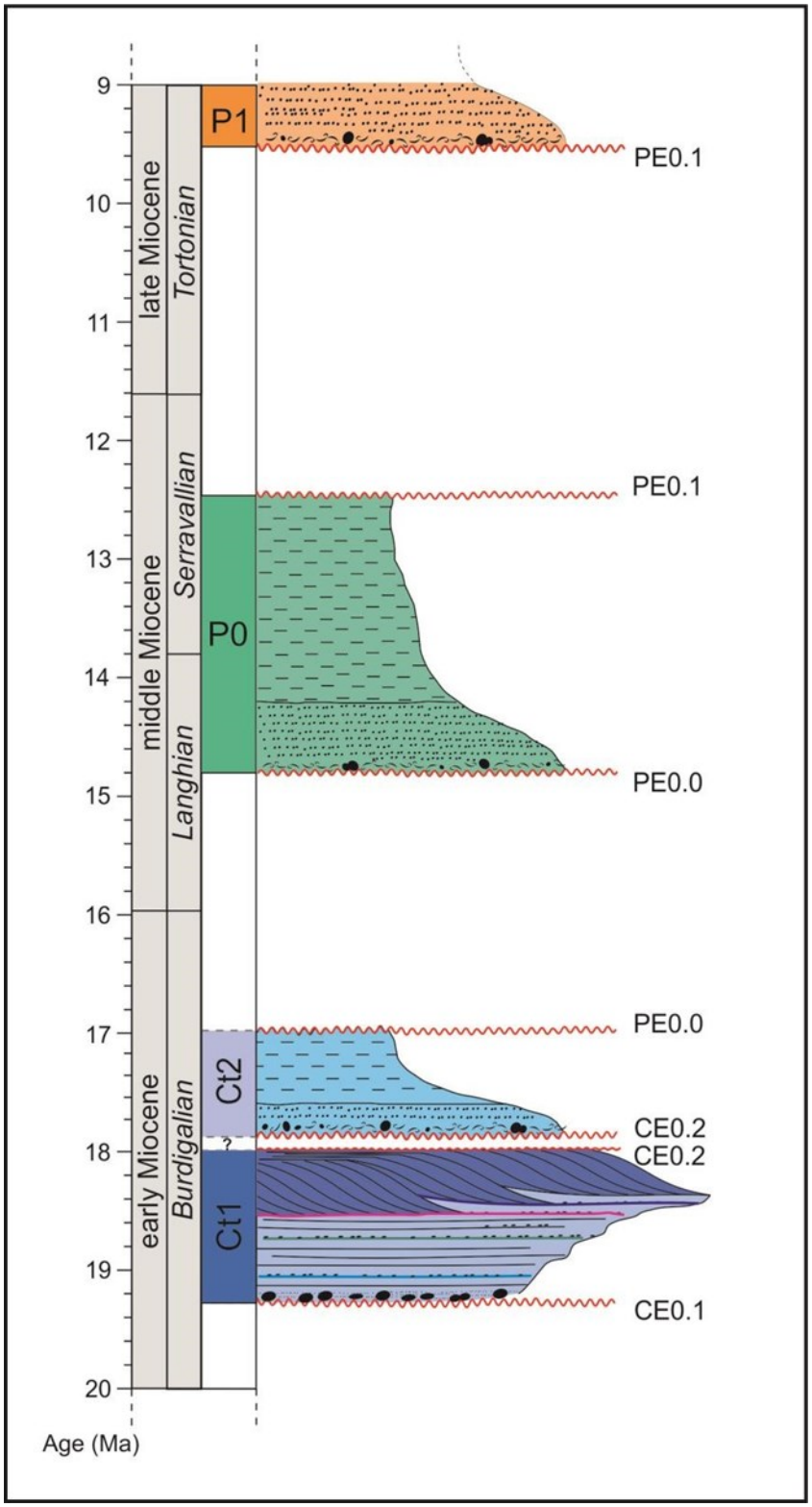

Figure 9. Schematic chronostratigraphic section of the Chilcatay (Ct1 and Ct2) and Pisco (P0 and P1) sequences according to Strontium Isotope Stratigraphy.

Table captions

Table 1

Formation	Sequence	Locality	Stratigraphic level	Samples	Description	Measured $^{87}Sr/^{86}Sr$	± 2s $_{mean}$	Corrected $^{87}Sr/^{86}Sr$
Pisco Fm	P0	Cerro Submarino	SUB-8bis	SUB-8bis1	Barnacle sheath	0.708811	0.000005	0.708827
				SUB-8bis2	Bulk cemented sediment	0.708273	0.000005	0.708289
				SUB-8bis3	Recrystallized bivalve shell	0.708650	0.000005	0.708666
			SUB-5	SUB-5	Ostreidae specimen	0.708804	0.000005	0.708820
			SUB-2	SUB-2	Calcite nodule	0.708557	0.000005	0.708573
			Tooth plain	Tooth 1	Shark tooth: *Cosmopolitodus hastalis*	0.708803	0.000005	0.708819
				Tooth 2	Shark tooth: *Cosmopolitodus hastalis*	0.708778	0.000005	0.708794
				Tooth 3	Shark tooth: *Isurus oxyrinchus*	0.708784	0.000005	0.708800
Chilcatay Fm	Ct1	Ullujaya	Key bed B	UJA-2a	Ostreidae specimen	0.708541	0.000005	0.708557
				UJA-2b	Ostreidae specimen	0.708523	0.000005	0.708539
			UL-D4	UL-D4a	Ostreidae specimen	0.708506	0.000004	0.708522
				UL-D4b	Barnacle sheath	0.708617	0.000005	0.708633
				UL-D4c	Bulk cemented sediment	0.708444	0.000005	0.708460
			Key bed C	UJA-LIVC1	Ostreidae specimen	0.708535	0.000005	0.708551
				UJA-LIVC2	Bulk cemented sediment	0.708340	0.000005	0.708356
				UJA-LIVC3	Ostreidae specimen	0.708542	0.000005	0.708558
				UJA-LIVC4	Barnacle sheath	0.708521	0.000005	0.708537
			Mollusc- and barnacle-rich	UL-LIVa	Barnacle sheath	0.708516	0.000005	0.708532
			horizon	UL-LIVb	Pectinidae specimens	0.708512	0.000005	0.708528
				UL-LIVd	Pectinidae specimen	0.708514	0.000005	0.708530
		Roca Negra	PN Oyster bed	PN-OST	Ostreidae specimen	0.708498	0.000005	0.708514
				PN-GIO1	Ostreidae specimen	0.708513	0.000007	0.708529
				PN-GIO2	Ostreidae specimen	0.708536	0.000005	0.708552

Table 1. Sample list and description, with locality and stratigraphic data, reporting $^{87}Sr/^{86}Sr$ measured values and standard deviation, with $^{87}Sr/^{86}Sr$ values corrected for the difference between the USGS EN-1 value used for the compilation of the reference curve (McArthur et al., 2012) and the USGS EN-1 Bochum mean value.

	Stratigraphic level	*minus 2 s.e.*	$^{87}Sr/^{86}Sr$ mean value	*plus 2 s.e.*	Maximum age (Ma)	Preferred age (Ma)	Minimum age (Ma)
P0	Lower P0	0.708799	0.708812	0.708825	14.80	13.45	12.45
Ct1	Key bed B	0.708530	0.708548	0.708566	18.60	18.30	18.00
	Key bed C	0.708536	0.708549	0.708561	18.50	18.30	18.10
	Mollusk- and barnacle-rich horizon	0.708528	0.708530	0.708532	18.60	18.50	18.40
	PN Oyster bed	0.708510	0.708532	0.708554	18.85	18.50	18.15

Table 2. $^{87}Sr/^{86}Sr$ ages for the Ct1 and P0 sequences, calculated from the LOWESS Table 5 (McArthur et al., 2012).