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ORIGINAL ARTICLE

Why Did *Red Ereño* Limestone Go Red? Linking Scientific Knowledge and Geoheritage Story-Telling (Basque Country, Spain)

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Abstract
Red Ereño is a red-stained ornamental and construction limestone with characteristic white fossil shells. Although exploited since Roman times, marketed worldwide and that the rock itself and its outcrop areas have been included in geological heritage inventories, the origin of its characteristic reddish colour remained unresolved. The aim of this work is to deepen the scientific knowledge of *Red Ereño* as a basis for understanding the characteristics of this stone and to make this information available for geoconservation actions. The mineralogical and petrological study, mainly based on optical and electron microscopy, X-ray diffraction, and rock magnetism and paleomagnetic techniques, concluded that the red-staining mineral is pigmentary hematite. Moreover, the analysis stated that hematite precipitated after sedimentation but prior to burial diagenesis and before alpine inversion. Based on palaeomagnetic studies, it can be stated that mineralisation occurred during the Late Cretaceous. This work illustrates how scientific research on this potential heritage stone provides key information for geoconservation.

Keywords *Red Ereño* · Colour · Rock magnetism · Paleomagnetism · Cultural heritage · Geoheritage story-telling

Introduction

Ornamental and construction stones are rocks extracted from their natural outcrops, which have been worked and are part of emblematic buildings, fountains, or other urban elements and are considered as cultural heritage of geological interest (Díaz-Martínez and Díez-Herrero 2011; Brocx and Semeniuk 2019). Studies to identify the origin and characteristics (mechanical, petrological, etc.) of these stones are essential for the conservation and/or restoration of emblematic buildings and ornamental elements, as well as for the generation of geotourism experiences. Besides, the attribution of unique identities to these rocks, particularly, to the ones considered as heritage stone, is a priority for the Commission on Geoheritage of the International Union of Geological Sciences (IUGS-ICG) (Pereira and Marker 2016a).

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One of the most outstanding aspects of ornamental and construction stones is the colour. Numerous intrinsic factors, as well as external processes, determine this physical property. Some of them are the nature of the sculpting, its external finishing, and various weathering or polluting processes (Damas Mollá et al. 2018). In fact, there are several studies in the literature about colour changes of ornamental stones due to accelerated ageing processes (Grossi and Benavente 2016). Nevertheless, studies focused on the origin of stone colour (i.e. on the intrinsic factors) are still scarce.

Since ancient times, intense coloured stones, especially varieties with reddish tones, have had a great relevance in terms of their human use. An example of this importance is the significance of red porphyry associated with the imperial supremacy in the Roman times (Abu El-Enen et al. 2018). Currently, red porphyry is still a highly valued material, as illustrated by the use of Iranian red porphyry in the Basilica of *La Sagrada Familia* in Barcelona (Spain), declared as UNESCO World Heritage Site (Sagrada Familia 2022). Furthermore, it can be easily identified the use of numerous varieties of red rocks all over the world, e.g. *Carmen Red* and *Eagle Red* granites (Finland) (Härmä et al. 2015); *Red Ogwell* limestone (UK) (Walkden 2016); *Royal Byzantiné*, *Griotte coquiller* (Dusar et al. 2009), and *Red Marble of Baelen* (Dreesen et al. 2013) limestones (Belgium); *Portasanta Marble* limestone (Greece) (Cantisani et al. 2014); *Rojo Baztán* (Damas Mollá et al. 2013), and *Rojo Alicante* (IGME 1976) limestones (Spain); *Vindhyan Sandstone* (India) (Kaur et al. 2019); or *Rojo Tlayúa* limestone (Mexico) (Pantoja-Alor 1992).

Red Ereño is a limestone quarried for ornamental and construction purposes in the Basque Country (Spain) since Roman times (first century A.D.) until the end of the twentieth century. Despite the existence of other red-stained rocks in the Basque Country (i.e. Triassic red sandstones, Upper Cretaceous and Lower Paleocene red marly limestones), the presence of abundant white fossils (mainly rudists) embedded in an intense red (pigmented) matrix gives this rock its distinctive character. Due to this appreciated characteristic, this stone has been widely recognised in the local, national, and international market; therefore, it is possible to find it in important buildings across the Iberian Peninsula and in many others of world renown, both for religious and civil use (Damas Mollá et al. 2021a).

Hematite is usually responsible for red staining in sedimentary rocks (Hargraves and Fischer 1959; Hu et al. 2005; Tong et al. 2017; Abrejevitch et al. 2018). Nonetheless, in the case of some red limestones, such as the Cretaceous *Red Scaglia Rossa* limestone (Italy), the presence of hematite is not detectable by X-ray diffraction (XRD), which has led to numerous debates about the origin of the staining (Cai et al. 2009). The variety of hematite that has a high staining capacity is extremely fine-grained, also known as pigmentary hematite (Hargraves and Ficscher 1959). It generally occurs in very low concentrations and its distribution is random in the rock (Cai et al. 2009). The origin of this hematite variety can be syngenetic (detritic) or epigenetic (diagenetic). In this second case, the precipitation of authigenic hematite is likely associated with reactive fluids, both deep hydrothermal (Park 1997) and meteoric.

Hematite is an antiferromagnetic iron oxide (Fe_2O_3) that behaves as a ferromagnetic mineral (sensu lato). This type of mineral is scarce but very important when studying palaeomagnetism, since when applying on it a certain magnetic field, it is preferentially oriented, and when this field disappears, it has the capacity to maintain a remanent magnetisation. This property in rocks is known as the natural remanent magnetisation (NRM). NRM is resistant to partial heating and can remain chemically unaltered under oxidizing conditions at Earth's surface (Dunlop and Ozdemir 1997). In the case of rocks that show reddish staining and where the concentration of hematite is so low as to be undetectable by XRD, its presence can be determined by different rock magnetic experiments. The rock magnetic analysis and the palaeomagnetic study of NRM carrier minerals can help identify specific mineralisations (Hargraves and Fischer 1959). In addition, the combination of these experiments allows relative dating, that is, constraining mineralisation emplacement timing within sedimentary rocks such as limestones (Hurley and Van der Voo 1987), fine-grained detritic rocks (Abrajevitch et al. 2014, 2018; Tong et al. 2017), metamorphic and igneous rocks (Brown and McEnroe 2012), and besides, mineral deposits (Symons et al. 2017).

From another point of view, scientific research on construction and ornamental stones provides key information that is useful in four items:

(1) Geosites. Scientific data provide knowledge concerning geosites and help in their assessment to be part of an inventory. In many cases, some of the stones coming from the quarries as moveable geoheritage, and often, ornamental or construction stone outcrops themselves are part of geosites inventories (De Wever et al. 2017; Todaro 2019; Monge-Ganuzas 2021).

(2) Cultural heritage: Characterisation and/or restoration. The petrological study of construction and ornamental stones, especially those used in heritage buildings, is essential in order to define an identity for that lithology and to determine their provenance. Worldwide, there are numerous examples of descriptions of these stones that are part of the cultural heritage (Cooper 2015; Schouenborg et al. 2015; Navarro et al. 2019; Kaur et al. 2020; Del Lama and Costa 2022). Besides, a clear characterisation of the stones is essential in order to establish the most appropriate restoration criteria of heritage buildings (Pozo-Antonio et al. 2017). Furthermore, the use of stone has varied throughout the history and it is observable in current buildings; therefore, the characterisation of the lithology used in each period complements the historical and architectural study of those buildings (Pereira and Marker 2016b; Lezzerini et al. 2019; Damas Mollá et al. 2021b).

(3) Story-telling for geotourism activities. Information for public consumption related to construction and ornamental stones as well geosites must be based on scientific evidence and must modulate depending on the type of public and the nature of the tourist activity. In this way, scientific knowledge must be translated into different registers, from those aimed at scholars and teachers, general public, and to those aimed at experts (Carcavilla 2007; Hilario 2018). The environments in which this geotourism takes place are also important, although the materials and resources to be used on a guided route or in an interpretation centre are not always the same (Ham 2013). Science must be accessible and explainable for the transmission to society. There are many examples of stories related to tourists describing the ornamental stones of cities or relating these rocks as building material to their original outcrops (Borghi et al. 2014; Palacio-Prieto 2015; Del Lama 2019; Kubalíková et al. 2020). Geourban routes based on ornamental stones are a frequent resource (Freire-Lista et al. 2022; Santi et al. 2021; Damas Mollá et al. 2021c). Without the appropriate scientific knowledge, interpreters or tour guides will hardly be able to offer a rigorous and at the same time attractive account about, for example, the striking colour of *Red Ereño*.

(4) Story-tellings for educational or interpretative. Scientific research should serve as a basis for the generation of didactic and educational content both for scholars and undergraduates and for the development of workshops and dynamic tools to use in this field (Valentino et al. 2020). In this sense, ornamental stones are very accessible for use as an educational resource. A simple walk through any city will provide an insight into the geological elements of the environment and will give us the basis to structure an interpretative story based on solid scientific knowledge produced by geoscientists. It is becoming more and more common to take students out into the streets to study natural sciences using the meaningful learning method, in which the prior ideas of the students are used as a starting point for experiential work. In this sense, working with heritage stones can be very easy and effective, and for this, it is essential to have the best possible scientific knowledge.

The aim of this work is to deepen the scientific knowledge of *Red Ereño* as a basis for understanding the characteristics of this ornamental and building stone. Firstly, we will present the techniques used for mineralogical determination that allow identification of the red-staining mineral, staining/mineralising processes, and estimated staining period. Secondly, we will discuss why this research should be considered as an example that illustrates how scientific research provides valuable information that should be used as a resource to deepen the knowledge of a geosite and even used to elaborate story-telling for geotourism consumption and/or educational resources.

***Red Ereño*: Geographical and Geological Framework**

Red Ereño has been exploited from small- to medium-sized quarries surrounding the municipalities of Ereño and Gautegiz-Arteaga (Biscay, Spain) (Fig. 1a). The most extensive quarry developed on the *Red Ereño* is named as *Cantera Gorria* ("Red Quarry"), and it is located in Gautegiz-Arteaga (Fig. 1b). The outcrop of this red limestone was inventoried at local (Urdaibai BR geosite inventory, Mendia et al. 2011), regional (Basque Autonomous Community geosite inventory, Gobierno Vasco 2015), and national scale (Spanish geosite inventory, IELIG 2022) (Damas Mollá et al. 2022). This stone has been widely marketed and is found in many important buildings of the Iberian Peninsula and in many other world-renowned buildings (Damas Mollá et al. 2021a).

Ereño limestone outcrops are located in the northern central part of the Basque-Cantabrian Basin (BCB), an inverted Mesozoic rift basin, to the east of the NNW-SSE Gernika anticline and to the north of the NW–SE Aulesti anticline (Agirrezabala 1996). The evolution of this basin is linked to the opening of the North Atlantic region during the Mesozoic, which resulted in the opening of the Bay of Biscay and the creation of the Pyrenean rift basins under extensional deformation (i.e. Choukroune and Mattauer 1978; Montadert et al. 1979; García-Mondéjar et al. 1996; Roca et al. 2011). The BCB underwent subsequent rifting phases since the Late Triassic, but the main rift phase took place during the Aptian-Albian interval, when synsedimentary faulting compartmentalised the basin and caused coeval shallow-marine to deep-water sedimentation, allowing deposition of thick sedimentary piles (García-Mondéjar et al. 2004a, b). Recent studies suggest that this extensional event led to an extremely thinned crust (hyper-extension) in the BCB, resulting in the emplacement of crust/mantle rocks at structurally shallow depths, or even exhumation at the seafloor or at the base of the sedimentary pile (Roca et al. 2011; Tugend et al. 2014; DeFelipe et al. 2017). Subsequently, a generalised transgression occurred from latest Albian to Santonian resulting in the deepening of the basin and sedimentation of thick hemipelagic successions. The convergence between Iberia and Eurasia from Late Cretaceous to Miocene resulted in basin inversion due to shortening and uplift of the former Mesozoic rift basin (Alpine orogeny; e.g. Choukroune 1976; Srivastava et al. 1990; Muñoz 1992; Vergés et al. 1995).

The study area includes an Aptian-Albian stratigraphic succession, which records multiple deformation events: a syndepositional extensional (rift-related) phase and a compressive (inversion) phase related to the Alpine orogeny. The lowermost part of the studied succession is composed of alternating 5–20 cm tabular sandstones and irregularly laminated siltstones of deltaic to shallow-marine siliciclastic

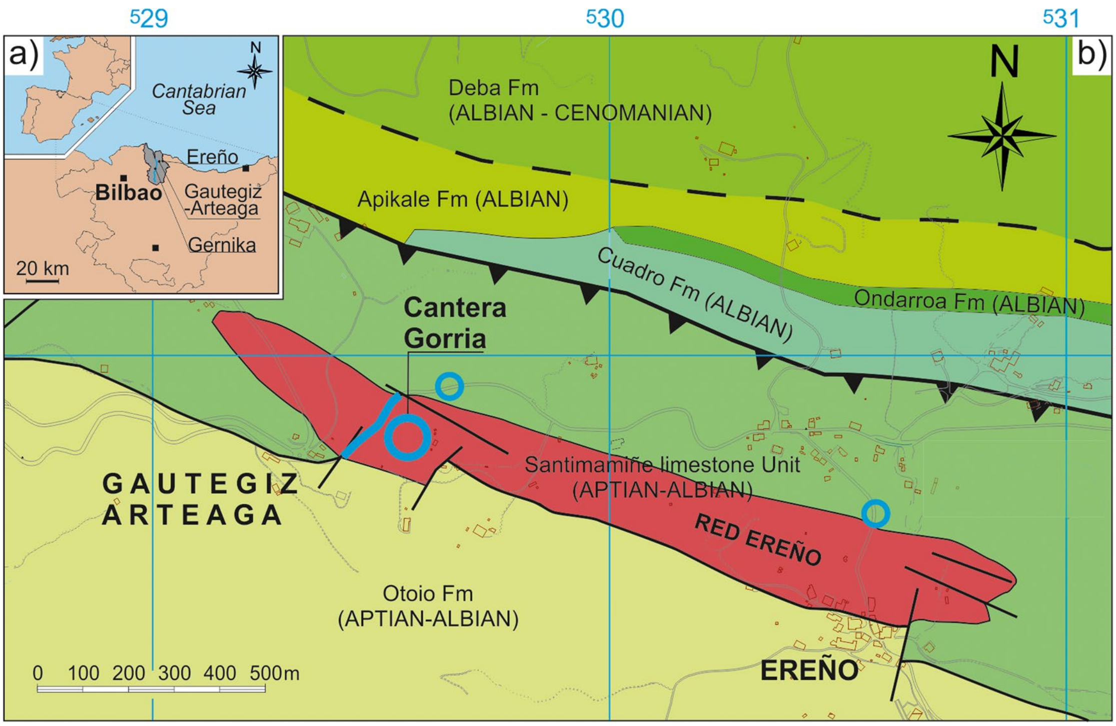

Fig. 1 **a** Geographical location. **b** Geological map of the area showing the sector where the reddish limestone crops out. The blue line to the west of *Cantera Gorria* indicates where the samples were taken for mineralogical analysis by X-ray (Blue circles indicate the sampling locations for palaeomagnetic experiments)

shelf deposits of early Aptian to early Albian age (Otoio Fm.; IGME 2009). This unit is diachronically overlain by the Santimamiñe unit (Agirrezabala 1996), where Ereño red limestone appears, a 500–600 m thick succession of lower Albian stage limestones (100.5–113 Ma). This subtropical shallow-water marine, ramp type, carbonate-platform micritic limestone body, shows a general N110ºE strike and a dip of 85º towards the NE. The common colour of the matrix of the limestone unit is grey, except for an elongated band, of approximately 1500 m, at the base of the unit. This band is subparallel to the stratification, shows reddish tones and corresponds to *Red Ereño* (Fig. 1b) (Damas Mollá et al. 2021a). The Santimamiñe unit is overlain by deep-water deposits: megabreccias, marls, and calcarenites of the Cuadro Fm.; turbiditic conglomerates, sandstones, and lutites Ondarroa Fm.; sandstones and bioturbated calcareous lutites of the Apikale Fm.; and turbiditic lutites and sandstones of Deba Fm (IGME 2009).

The limestone preserves an abundant fossil content in which bivalves are particularly significant, especially polyconitid and requienids rudists. Polyconitid rudists are found in thickets, bioconstructions of hundreds of individuals (Fig. 2a and b). The requienid rudists, likewise, appear in groups of dozens of individuals (Fig. 2c and d) (Damas Mollá et al. 2021a, 2022). The most striking feature of *Red Ereño* is that only its matrix is red, whereas fossils are not. Fossils remain white, indicating that the process that produced this colouration did not affect its internal microstructure. The shells of the rudists preserve a distinct two-layered microstructure: the outer layer consists of prismatic calcite (prismatic calcite layer, PCL) (Skelton 1974), and the inner layer is composed of aragonite (AL). PCL in the polyconitid is light grey (Fig. 2a and b) while in the case of the requienids is deep black (Fig. 2c and d). The inner layer, originally aragonitic (AL), is usually more affected by diagenetic fluids (Al-Asm and Veizer 1982, 1986), and calcitic cements might locally replace it (Damas Mollá et al. 2004; Damas Mollá 2011). In the rudists of Ereño limestones, this inner layer is white (Fig. 2).

However, the reddish staining of the matrix of the Santimamiñe limestone is not homogeneous. Thus, the sections of *Cantera Gorria* show variations in the reddish tone of the rock (Fig. 3a–c). This is sometimes associated with fault planes (Fig. 3d). The red tones are more intense among stylolite planes, surfaces formed by pressure-dissolution processes during diagenesis (Fig. 3e and f).

Red Ereño Colour in Constructions

Since Roman times, *Red Ereño* has been used as an ornamental stone (De Areitio 1906). Its red colour, the hallmark of this lithology, differs according to the sector of the original quarry from which it comes. The finish and the degree of alteration also control the intensity of the colour. For example, the raw rock has lighter tones than the carved ones, as can be seen in

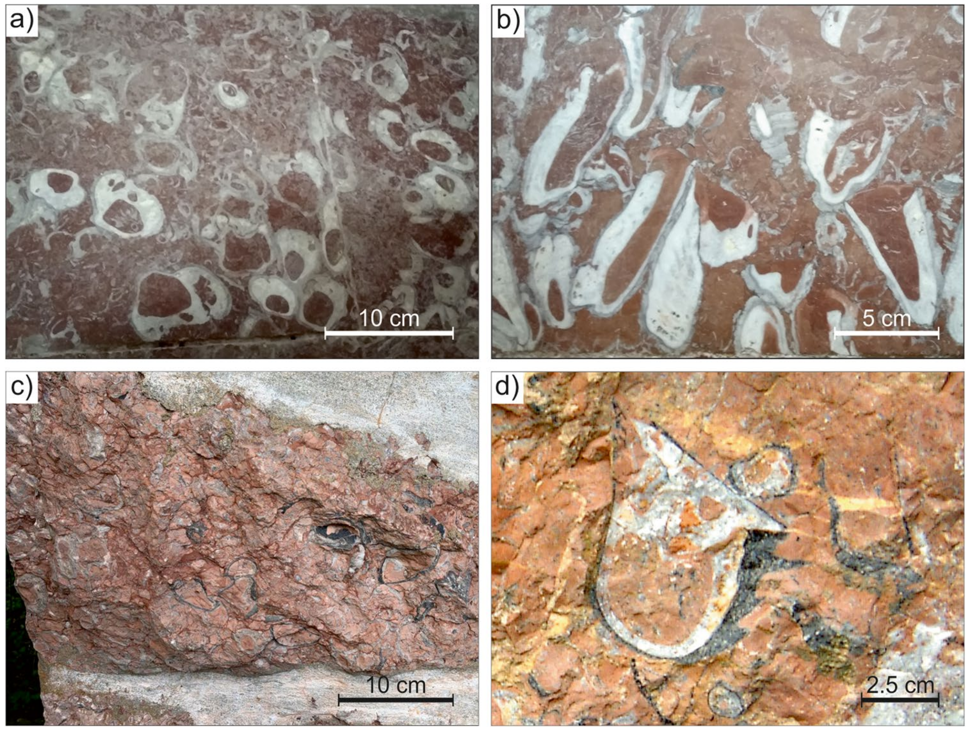

Fig. 2 **a** Transversal section of polyconitid rudists on the flooring inside *Andra Mari* Church (Gautegiz Arteaga). **b** Longitudinal section of polyconitid rudists on the flooring inside *Andra Mari* Church (Gautegiz Arteaga) (grey: PCL; white AL). **c** *Red Ereño* masonry with abundant fossils of requienid rudists used in a building located in the upper part of *Cantera Gorria*; **d** Oblique section of requienid rudists from the *Cantera Gorria* showing the two layers of their shell (black, PCL; white, AL)

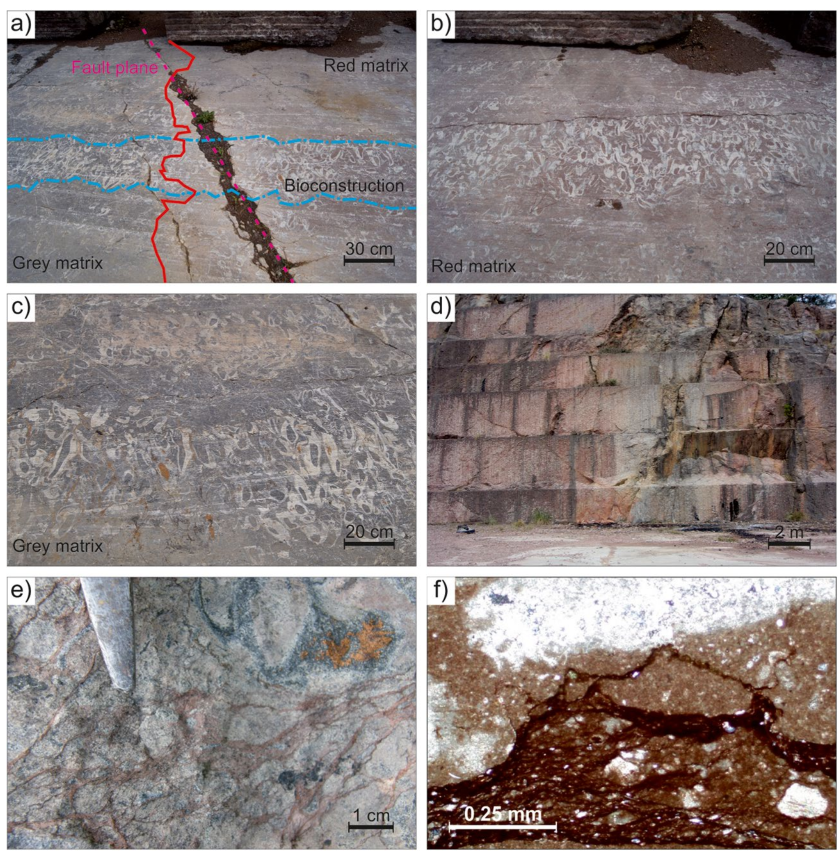

Fig. 3 **a** Changes in the colour of the micritic matrix observed in a section of the *Cantera Gorria* (*the red line defines the irregular limit between red and grey matrix bodies*). Detail images of polyconitid rudist thickets within the red (**b**) and grey (**c**) micritic matrix. **d** Fault plane at *Cantera Gorria* front with clear changes in the colour of the stone matrix. **e** Stylolites with an intense red colour in *Cantera Gorria*. **f** Detail microphotograph of stylolite in limestone matrix (cross-polarised light)

Fig. 4 Sculpture of Count Don Tello in Gernika-Lumo (Biscay, Spain). The base is constructed in *Red Ereño*

the base of the sculpture of *Count Don Tello* in Gernika-Lumo (Biscay, Spain) (Fig. 4).

Churches are the most abundant type of buildings of the Basque Country containing carved *Red Ereño*. Remarkably, the churches closest to *Red Ereño* quarries are the ones that show most significant colour variability in its constructions (Fig. 5). It is the case of the fifteenth century *San Miguel* Church (Fig. 5a), in Ereño village, which shows a greyish and reddish mosaic of well-carved metric blocks (Fig. 5c). Another example is the *Andra Mari* Church (Fig. 5b), in Gautegiz-Arteaga (the village where the main *Red Ereño* quarry, *Cantera Gorria*, is located). This church dates from the thirteenth century and has two pilasters made of *Red Ereño* ashlars at the main entrance that stand out from the rest of the construction, which is made of grey limestone (Fig. 5d and e) also from the Santimamiñe unit. Both constructions are listed in the Basque cultural heritage inventory as sites of archaeological presumption (Gobierno Vasco 2022a, 2022b).

Nevertheless, when the rock polished, the matrix takes on an intense dark red colour (Fig. 6a–c). Additionally, some constructions show clear signs of differential erosion and discolouration of the matrix, such as the columns at the entrance to the back courtyard of the *Victor Chavarri* Palace in Bilbao (Basque Country, Spain) (Aranburu et al. 2009) (Fig. 6d).

Fig. 5 **a** San Miguel Church in Ereño (Basque Country, Spain). **b** *Andra Mari* Church in Gautegiz-Arteaga (Basque Country, Spain). **c** Detail of the dimensions of stones of different tones of *San Miguel* Church (ashlar width varies between 0.5 and 1 m). **d** Pilasters from the main door of *Andra Mari* Church. **e** Detail of the ashlar blocks containing polyconitid rudists (block width is 1 m)

Fig. 6 **a** Recently restored and newly polished covering slab of the *Sota* building in Bilbao. Longitudinal section of polyconitid rudists. **b**, **c** *Red Ereño* in the Sacristy of Toledo Cathedral (Spain). They are behind the painting "The Despoilment of Christ" by El Greco. The front columns are from the *Black Markina* ornamental limestone (Bizkaia, Spain). **d** Column discoloured by differential erosion processes from the rear door of the garden of the *Victor Chávarri* Palace in Bilbao (Basque Country, Spain)

Materials and Methods

In order to identify, characterise, and quantify the mineralogy of the Ereño limestones, 17 samples were selected, from base to top of the reddish band of the Santimamiñe unit (Fig. 1b). We analysed the samples by X-ray diffraction (XRD) in two rock fractions: the total rock and the fraction below 2 µm. To this end, we used the diffractometer Philips PW 1710 with Cu + 2 anticatod. In addition, we developed further studies with a Jeol JSM–6400 (SEM) microscope, and microanalyses were carried out with an Oxford INCA ENERGY 350 energy dispersive X-ray equipment (EDS), in the general services (SGIker) facilities of the UPV/EHU.

Four different types of palaeomagnetic analysis on Ereño limestones were performed in order to obtain data to clarify the magnetic mineralogy represented. Thus, it is to known which mineral carries the natural remanent magnetisation of the rock and when this magnetisation was acquired, i.e. whether it is primary or secondary. Palaeomagnetic analyses were conducted at the Palaeomagnetic Laboratory of the *Universidad de Burgos* (Spain). We selected two types of samples for the magnetic experiments. Firstly, 62 oriented sample cores were obtained in *Cantera Gorria*. Secondly, the set of subsamples described in Table 1 was selected.

Three magnetic experiments were performed based on the properties of the (s.l.) ferromagnetic minerals. In these minerals, induced magnetisation (J) increases when an external magnetic field (H) is applied to them up to value known as saturation field (Hs) reaching the so-called saturation magnetisation (Js). When the external field is no longer applied, the ferromagnetic minerals do not cancel

Table 1 Selected samples of oriented cores obtained in the Ereño limestones for magnetic experiments (*AF*, demagnetisation by decreasing alternating fields; *IRM*, isothermal remanent magnetisation; NMR, natural remanent magnetisation)

Sample type	AF	IRM	NMR
Red matrix	AT-A – 5.A	AT-A – 5.C	AT-A – 5.B
Red matrix in stilolytes	AT-B – 3.A	AT-B – 3.C	AT-B – 3.B
Red matrix in internal cavity of polyconitid rudist	AT-C – 5.A	AT-C – 5.C	AT-C – 5.B
Polyconitid rudist shell and red matrix	AT-D – 7	AT-D – 5.A	AT-D – 6
Chondrodonta sp. shell	–	–	AT-F – 2
Requienid rudist shell	–	–	AT-F – 4
Ferrous calcite in the inner cavity of a polyconitid rudist	AT-G – 7.B	AT-G – 7.A	AT-G – 1
Ferrous calcite in fracture	AT-G – 3.B	AT-G – 3.A	AT-G – 2.A
Ferrous calcite in fracture	AT-G – 4.A	AT-G – 4.B	AT-G – 4.C
Ferrous calcite in rudist aragonitic layer	–	–	AT-G – 5
Black crystal in ferrous calcite fracture	–	AT-G – 8.B	AT-G – 8.A
Grey matrix	AT-H – 1.A	AT-H – 1.B	AT-H – 1.C
	–	–	AT-H – 4.B
Rose matrix	AT-I – 3.A	AT-I – 3.B	AT-I – 3.C
Ereño limestone with grey matrix outside the red ones	–	–	KER.2 – 5.A
	KER.2 – 7.A	–	KER.2 – 7.B
	KER.2 – 9.C	KER.2 – 9.A	KER.2 – 9.C

their magnetisation, but retain a magnetisation that is called remanent magnetisation. This irreversible behaviour is known as hysteresis. The magnetic field applied in the opposite direction necessary to cancel the induced magnetisation is the intrinsic coercive field (Hc). When the value of this intrinsic H is applied in the opposite direction to the first, the reverse cycle takes place, creating what is known as the hysteresis loop, which is characteristic of each ferromagnetic material (s.l.). In addition, ferromagnetic minerals lose their magnetic properties at a certain temperature, known as the Curie temperature (or Néel temperature in antiferromagnetic minerals), which is used to identify this type of mineral (Butler 1998; Tauxe 2010).

In carbonate rocks, the main ferromagnetic minerals recognised are magnetite and hematite. Magnetite and hematite are both iron oxides, but differ in the cation incorporated in each. Magnetite (Fe_3O_4) is a mineral characterised by its low coercivity and a Curie temperature of 580 °C. Hematite (Fe_2O_3), on the other hand, is of high coercivity with a Néel temperature of 670 ºC (Butler 1998; Tauxe 2010). There are two types of hematite: specular hematite, black in colour, and pigmentary hematite with a finer grain size, which gives a characteristic red colour to the materials in which it is found. We recognise the latter by a progressive decrease in the hard phase of the three-component IRM thermal demagnetisation curves (Lowrie 1990).

The magnetisation during the palaeomagnetic and most of the rock magnetic analysis has been measured using a 2G-755 cryogenic magnetometer at the Laboratory of Palaeomagnetism of the *Universidad de Burgos* (Spain). The common point of the first three techniques is based on progressive demagnetisation according to different properties of the minerals: (i) alternating field demagnetisation (AF) of NRM; (ii) thermal demagnetisation of the NRM; and (iii) progressive acquisition of isothermal remanent magnetisation (IRM) and subsequent thermal demagnetisation of three orthogonal IRM components.

Alternating field demagnetisation (AF) of NRM consists of applying decreasing alternating magnetic fields on the sample in a series of pre-established steps. Minerals with a coercivity equal to or less than the maximum peak field applied will be demagnetised, thus obtaining a detailed reading of minerals with a coercivity lower than the highest field emitted. This experiment has been developed by a device in line with the 2G magnetometer.

Thermal demagnetisation of the NRM has been carried out by progressive heating and cooling periods to room temperature, once the initial remanent magnetisation (NRM) has been measured. The heating and cooling has been carried out in a furnace with μ-metal shielding that isolates the samples from external magnetic field. The model used is the TD-48–SC (ASC Scientific). Each group of magnetic grains has a so-called unlocking temperature, which is lower than the Curie temperature, where they are unlocked for a short period of time. During cooling at zero field, they randomly orient their magnetic moment and become demagnetised. After each temperature step, the remaining magnetisation has been measured.

The last demagnetisation process considers progressive acquisition of isothermal remanent magnetisation (IRM) and subsequent thermal demagnetisation of three orthogonal IRM components following the protocol of Lowrie (1990). For the first part, each sample is exposed to an increasing magnetic field in a given direction for a short time period at constant temperature. This magnetic field was generated in a model M2T-1 impulse magnetiser. The intensity of the magnetic fields has been progressively increased in 13 steps from 0 to 2 T. After the application of each magnetic field, a measurement of the acquired magnetisation is performed. The second part starts with the exposure of each sample to three magnetic fields of different strength and orthogonal orientation in a given order. In this case, a strong field of 2 T has been applied in the direction of the Z-axis of the sample; a medium field of 0.40 T in the direction of the Y-axis of the sample; and a weak field of 0.12 T in the direction of the X field of the sample. This is done in order to differentiate minerals of different coercivity, since those of high coercivity will be arranged according to the strong field direction, those of medium coercivity according to the medium field, and those of low coercivity according to the 120 mT field. The identification is carried out after subjecting the samples to a thermal demagnetisation process analogous to that carried out on the samples to demagnetise the NRM (Lowrie 1990), which allows coercivity and unlocking temperatures to be associated.

Four more rock magnetic experiments were carried out on the variable magnetic field translation balance (MMVFTB, magnetic measurements) of the Palaeomagnetic Laboratory of the *Universidad de Burgos* (Spain). For this purpose, we selected 15 powder samples to allow the determination of the magnetic mineralogy present in samples that are more specific. Samples of grey, pink, red matrix, in fractures with ferrous calcite and in the shells of the main bivalves, i.e. polyconitids and requienids rudist and shells of *Chondrodonta* sp., have been selected. The translational balance generates four main types of data: (1) progressive acquisition curves of the isothermal remanent magnetisation (IRM), already described above; (2) hysteresis loops (± 1 T) that allow characterisation of the mineral and grain size in some cases; (3) backfield curves that indicate the coercivity of the remanence of a material; and (4) thermomagnetic curves that show the Curie (or Néel) temperature as well as the formation of new minerals during heating.

Research Results and Discussion

Mineralogy by XRD

The total rock XRD analysis shows that calcite is the main mineral present (94%) in the reddish and greyish limestone of the Santimamiñe unit (Fig. 7). Furthermore, although all the red samples are intensely coloured, the whole-rock XRD analysis could only detect hematite in few samples with an average content of < 1% (Fig. 7b). Characteristically, those samples stand out for having intense red stylolites in which hematite and clay minerals are concentrated (Fig. 3e and f). Quartz, potassium feldspar, and plagioclase are also present in the total rock fraction. In addition to petrographic observations, we consider a detrital origin for these minerals. The ferrous calcite is concentrated in late fractures (Fig. 8a) and inside the cavities and in the shell microstructure of some rudists (Fig. 8b). This mineral phase is clearly authigenetic and stands out in the field for its intense orange colour. Illite is the main mineral in the < 2 µm fraction (up to 70%) (Fig. 7c) and has a foliated appearance. It adapts to the micrite in the matrix, which indicates a detrital origin (Fig. 8c). It is noteworthy that hematite has been identified in the composition of the fine fraction of some samples where it is undetectable in the whole-rock fraction. Authigenic chlorite is also present in the most intense red samples. Scanning electron microscope (SEM) observations and semi-quantitative EDS microanalysis on these stylolitic zones have allowed us to recognise crystals with star-shaped morphologies, rich in iron and oxygen, attributed to pigmentary hematite crystals (Fig. 8d and e).

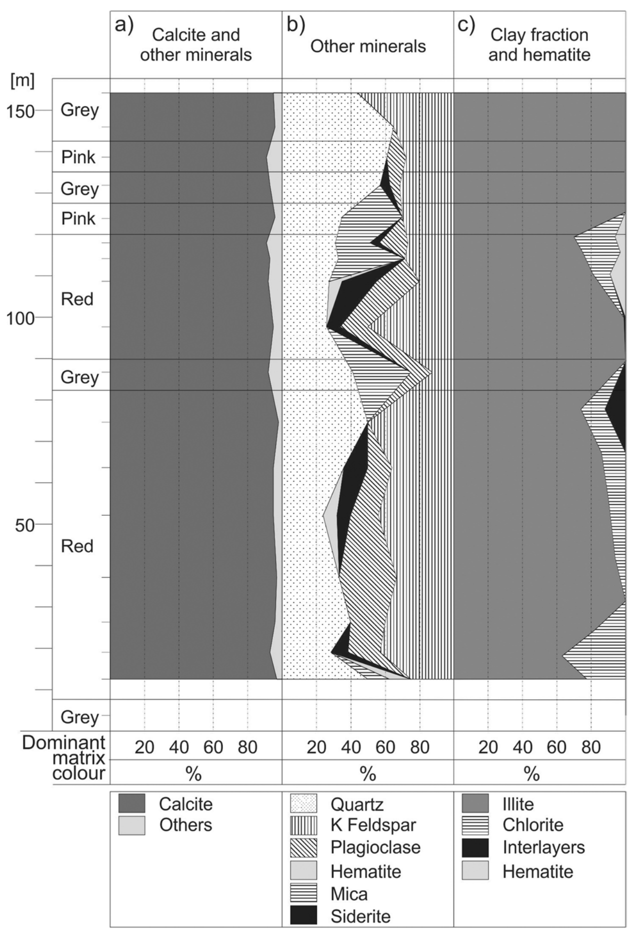

Fig. 7 X-ray diffraction mineralogy of samples of Ereño limestones according to the shade of their matrix: **a** results in total rock; **b** percentages of the rest of the mineral without taking calcite into account; **c** results of the phase below 2 µm ([m] refers to sampling along the sweep marked in Fig. 1b with a blue line (SW to NE))

Magnetic Mineralogy

On the one hand, the acquisition of isothermal remanent magnetisation (IRM) for red matrix samples indicates that the dominant magnetic carriers have high coercivity, which do not saturate when reaching 2 T fields. The thermal demagnetisation of three IRM components shows a dominant high coercivity fraction (2 T) with a dramatic drop at unblocking temperatures of about 670 °C, suggesting that hematite is the main ferromagnetic (s.l.) mineral. The morphology of the high coercivity phase thermomagnetic curve shows a progressive drop indicative of different grain size suggesting that the finest grains are pigmentary hematite (Dunlop and Ozdemir 1997) (Fig. 9a and b). On the other hand, the acquisition of IRM for grey matrix samples indicates the saturation of magnetisation between the 0.3 and 0.4 T fields. Another not saturated minor phase of high coercivity has been observed. The thermomagnetic curves show a dominant low coercivity component with maximum unblocking temperatures of about 550 °C, probably owing to the presence of magnetite. The scarce presence of the high-coercivity phase suggests the absence of hematite in the grey matrix samples (Fig. 9c and d).

The other rock magnetic experiments developed in the variable magnetic field translation balance show similar results to those obtained in the previous experiment. The red limestones show good records of pure hematite, which can be identified by several aspects (Fig. 10a): (i) the IRM does not saturate before 1 T; (ii) the backfield curve shows very high remanent coercivity of 500 mT; (iii) the hysteresis loop does not close the lines at lower values of 1 T showing typical shape of hematite; and (iv) the thermomagnetic heating curve shows only a clear sharp drop at 680 °C indicative of hematite. Both heating and cooling curves are irreversible. The later show an inflexion at 550 °C indicating the neoformation of magnetite at high temperatures.

The matrix of the grey limestones, meanwhile, shows evidences of magnetite dominance related to the following

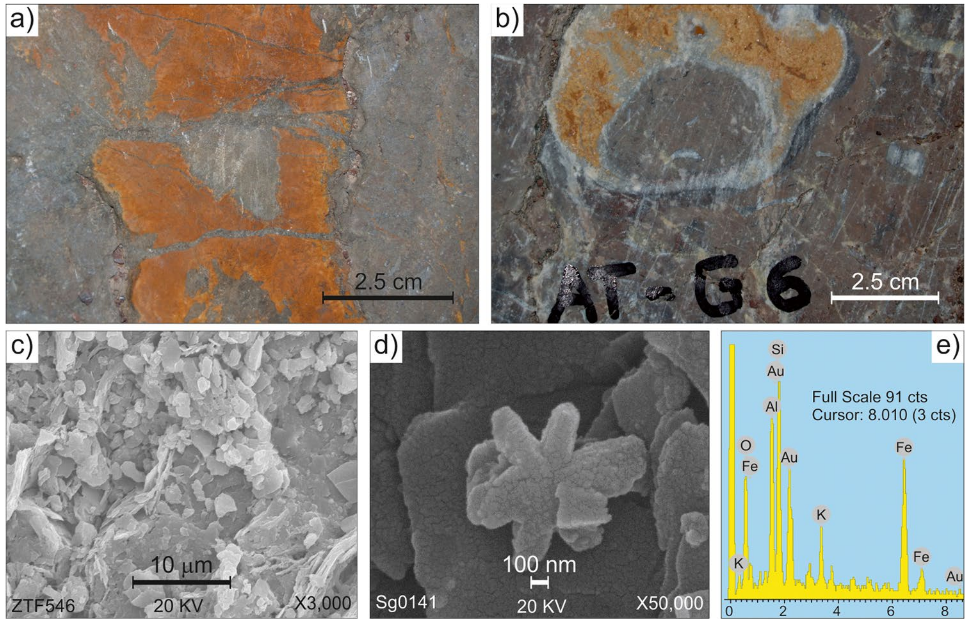

Fig. 8 **a** Late fracture in *Cantera Gorria* filled with ferrous calcite of intense orange tones. **b** Crystals of ferrous calcite in the aragonitic layer of a requienid rudist (cross section) in *Cantera Gorria*. **c** SEM microphotography of foliated illite in the matrix of the red limestones of Ereño. **d** Star-shaped iron oxide in the matrix of the Ereño limestones observed by SEM. **e** Semi-quantitative analysis by EDS obtained in the sample image above

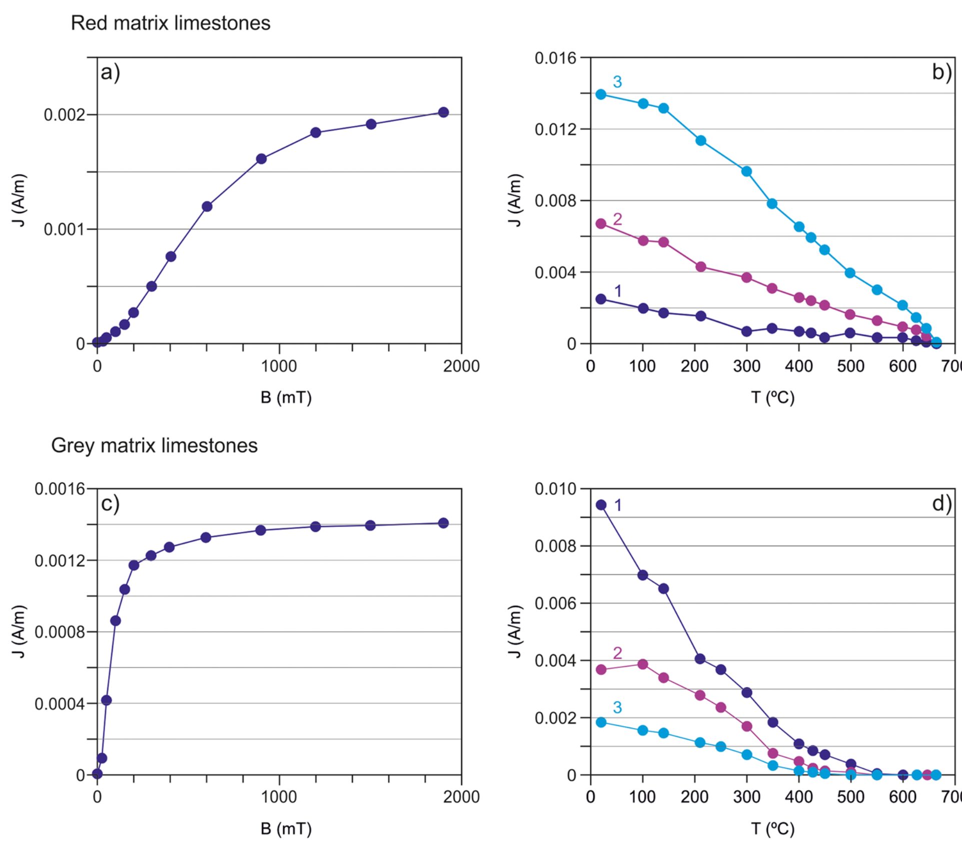

Fig. 9 Isothermal remanent magnetisation (IRM) acquisition (left column) and thermal demagnetisation curves of three IRM components (right column) from: **a**, **b** red matrix sample; **c**, **d** grey matrix sample.(J, remanent magnetisation (A/m); B, applied magnetic field (mT)) (line 1 (purple): high coercivity fraction (0.12 T in the X-axis); line 2 (pink): medium coercivity fraction (0.4 T in the Y-axis); line 3 (light blue): high coercivity fraction (2 T in the Z-axis))

characteristics (Fig. 10b): (i) low coercivity IRM; (ii) the back-field curve shows a low remanent coercivity of 60 mT; (iii) the hysteresis loop shows low coercivity dominance with a wasp waisted shape indicating mixing of coercivities, due to probably different magnetite size grain, typical of chemical remagnetised carbonates with authigenic magnetite (Jackson 1990; Channell and McCabe 1994); iv) the thermomagnetic curve is clearly irreversible due to the neoformation of ferromagnetic minerals during the experiment; however, the heating curve marks the presence of magnetite with a Curie temperature of about 580 °C.

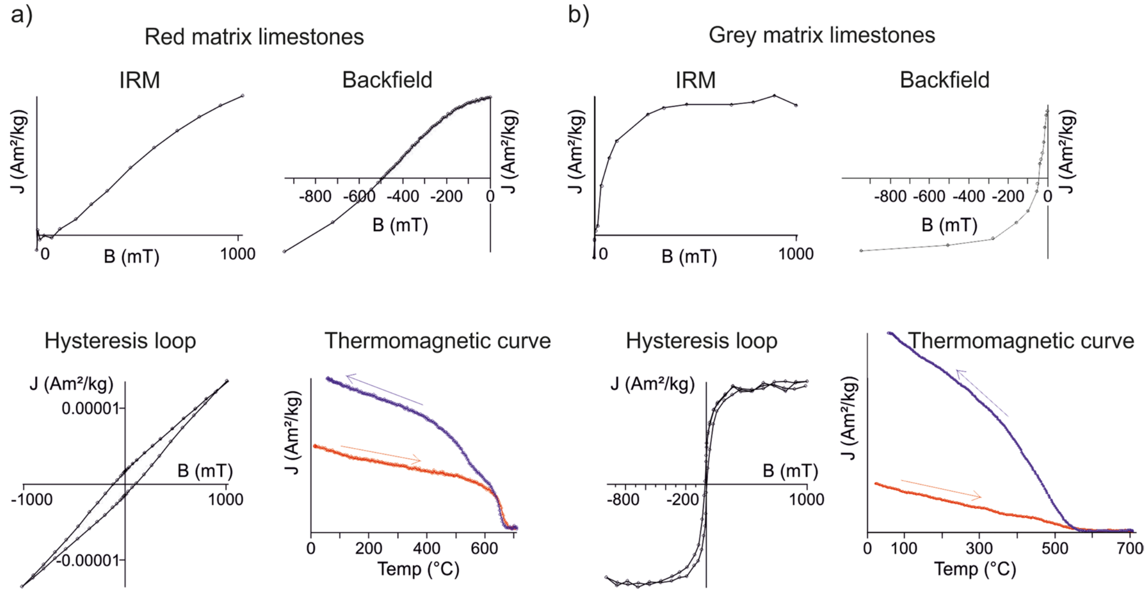

Fig. 10 Results obtained in the translation balance. **a** Red matrix sample. **b** Grey matrix sample. Experiments from left to right and from top to bottom: IRM acquisition curve, backfield experiment, hysteresis loop (dia/paramagnetic slop corrected in **b**), induced magnetisation thermomagnetic curve. (heating (red) and cooling (blue) (B, Magnetic field (mT); J, Magnetisation (Am2/Kg); Temp., temperature (°C))

The rock magnetic experiments performed in the shells of the different families of rudists show a very low concentration of ferromagnetic (s.l.) minerals, so that the thermomagnetic curves, IRM acquisition curves, and backfield are very noisy. Figure 11a shows the hysteresis cycle, obtained for the outer layer of the shell (PCL) of a requienid rudist (Fig. 11b), typical of an almost pure diamagnetic behaviour. The absence of ferromagnetic minerals does not allow the recording of magnetic remanence. This result indicates that, when the iron input occurred, the diagenetic system of the shells was closed.

Palaeomagnetism

Thermal demagnetisation of red matrix samples shows a stable component, although in many samples, the creation of ferromagnetic minerals during heating has masked the behaviour at high temperatures. However, in some cases (Fig. 12a), it has been possible to establish maximum unblocking temperatures at about 665 °C. On the other hand, regarding the results of alternating field (AF) demagnetisation, this paleomagnetic component cannot be demagnetised at peak fields of 100 mT, indicating that the main carrier of the NRM shows high coercivity (Fig. 12b). This result confirms the presence of hematite observed in the rock magnetic experiments and that this mineral is the carrier of the NRM in red limestones.

In the case of limestones with a grey matrix, thermal demagnetisation shows, after removing the typical viscous phases at low temperatures, a stable paleomagnetic component with maximum unblocking temperatures of about 550 °C (Fig. 12c). AF demagnetisation evidences that most part of the NRM can be destroyed at AF peak fields of 100 mT (Fig. 12d), indicating that the magnetic carrier has low coercivity. This result points to magnetite as the carrier of

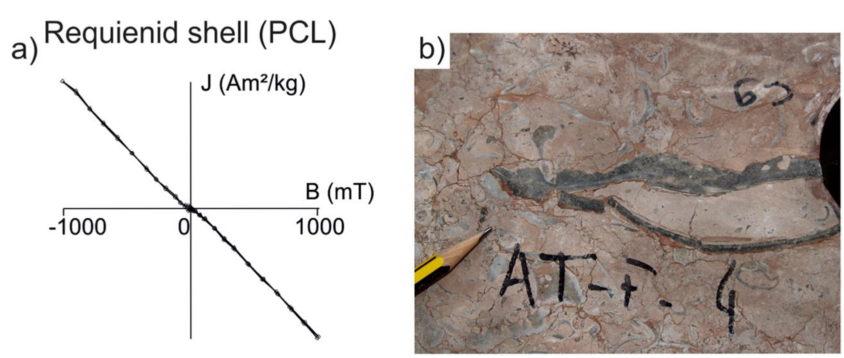

Fig. 11 **a** Hysteresis loop cycle corresponding to an almost pure diamagnetic behaviour obtained from a PCL sample of a requienid rudist shell. The magnetisation per unit mass (J) versus magnetic field (B) is presented. **b** Requienid rudist in oblique section at *Cantera Gorria* where the sample has been taken

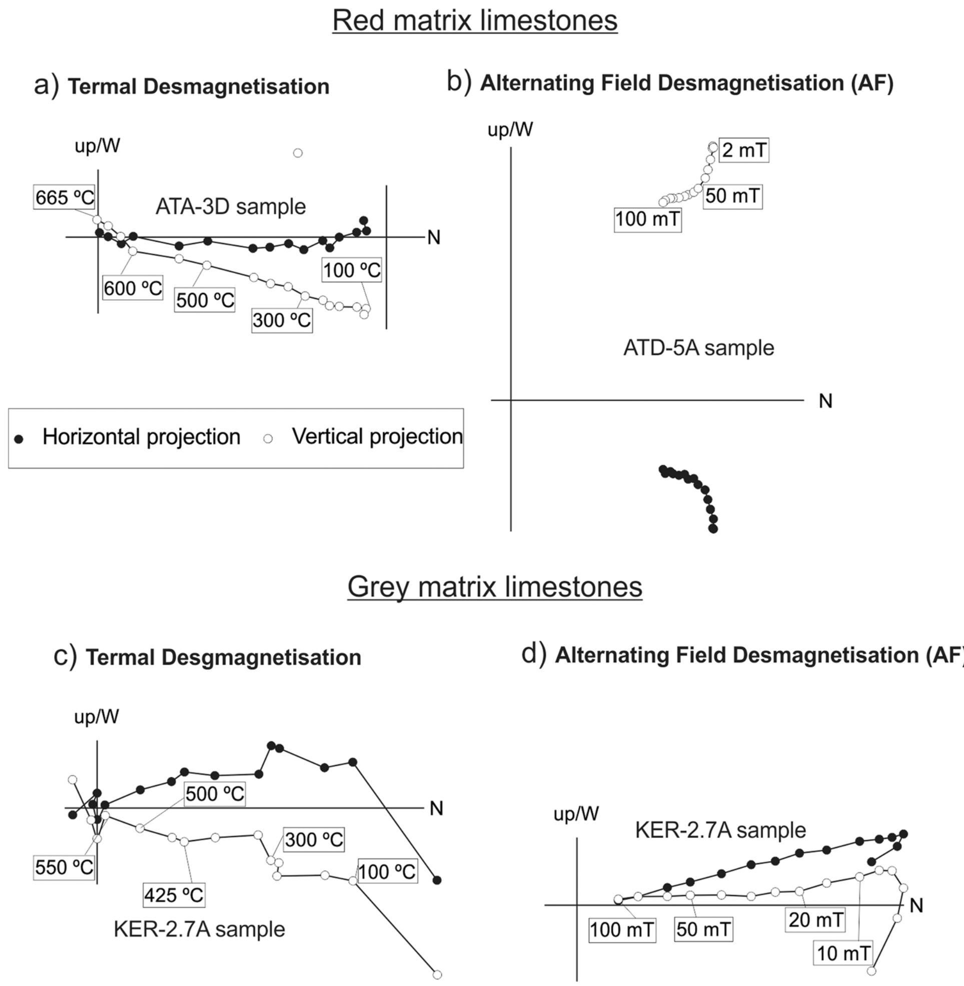

Fig. 12 Zijderveld plots after bedding correction showing the NRM demagnetisation of representative samples. **a** Thermal demagnetisation of red matrix limestones; **b** alternating field (AF) demagnetisation of red matrix limestones; **c** thermal demagnetisation of grey matrix limestones; **d** alternating field (AF) demagnetisation of red matrix limestones. Solid/open circles in orthogonal plots represent the projections of vector endpoints onto the horizontal/vertical and N-S planes

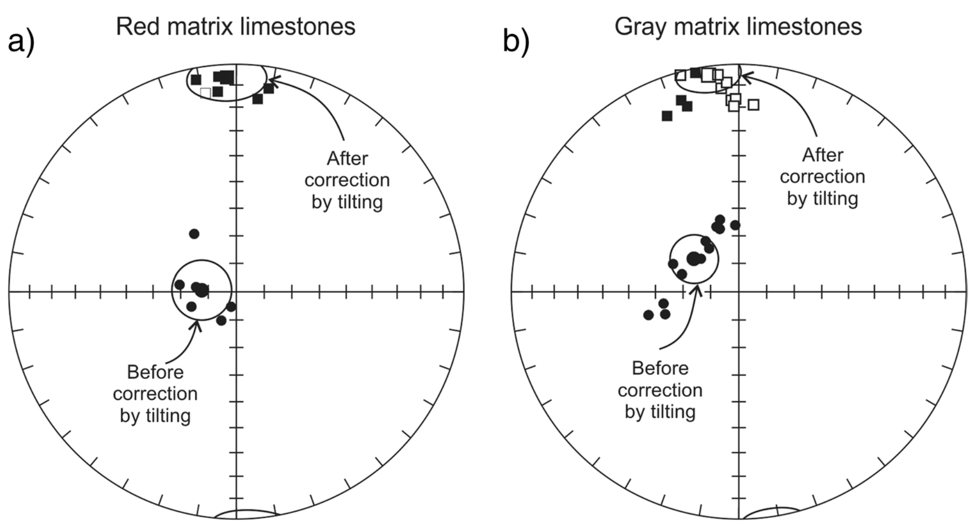

Fig. 13 Stereographic projection of palaeomagnetic directions obtained before and after tilting correction for: **a** Ereño limestones with red matrix; **b** Ereño limestones with grey matrix. Solid circles are projections in the lower hemisphere and white circles are projections in the upper hemisphere. Mean direction and 95% confidence circle are also shown

the characteristic remanent magnetisation (ChRM) in agreement with the observed in the rock magnetic experiments.

The ChRM direction observed in both red and grey limestones has been calculated from the results of thermal and AF demagnetisation in grey samples and only thermal in red ones. Plotting the results obtained on red and grey matrix limestones (Fig. 13a and b), it can be seen that the palaeomagnetic directions obtained from the hematite and the magnetite are similar, but not identical. However, none of them, in both pre-tilting and post-tilting configurations, is compatible with the palaeomagnetic direction estimated for the age of these rocks being far away from the expected direction for Aptian-Albian for the Iberian or European plates (Villalaín et al. 2003; Neres et al. 2012; Torsvik et al. 2012).

In both cases, the magnetisation direction differs from that established for the estimated formation age for these limestones. Magnetisation shows a consistent direction with cretaceous if about a 50% of the present-day tilt is restored, indicating that the ChRM in both lithologies is an interfolding remagnetisation. Therefore, it can be concluded that (at least part of) the remanent magnetisation has been acquired before current tilting of the limestone unit that is prior to the convergent stress attributed to the Alpine orogeny. Furthermore, hematite is concentrated in the insoluble residue of the stylolites, suggesting its precipitation prior to stylolite formation. Therefore, mineralisation occurred after the closure of the fossil shell microstructures inhibiting its staining (Damas Mollá 2011), but before chemical compaction, thus during mid-diagenesis (before burial). This analysis suggests that the pigmentary hematite, responsible for the characteristic colour of these limestones, is epigenetic. These results constrain the mineralisation period between the (upper?) Albian (post-sedimentation; Agirrezabala 1996) and the start of the Alpine convergence (Santonian–Campanian; e.g. Macchiavelli et al. 2017).

Epigenetic mineralisations are usually attributed to reactive fluids (e.g. Park 1997), which have been usually linked to active tectonics and/or volcanic episodes in the Basque-Cantabrian Basin (e.g. Aranburu et al. 2002; López-Horgue et al. 2010; López-Horgue and Bodego 2012; Iriarte et al. 2012; Ladron de Guevara et al. 2020). One of the most active tectonic episodes in the Basque-Cantabrian Basin occurred during Upper Albian-Cenomanian times due to extensional synsedimentary tectonics. In literature, several examples show the interaction between Aptian-Albian rocks and Upper Albian-Cenomanian volcanism and hydrothermalism in the centre of the basin (Fernández 1994; Aranburu et al. 2002; Fernández-Mendiola and García-Mondéjar 2003; López-Horgue et al. 2010; López-Horgue and Bodego 2012). Although further investigations would be needed in order to constrain the mineralisation time interval and the nature of the mineralising processes, the upper Albian-Cenomanian tectonic event may be a candidate for the red-staining mineralisation.

Colour Research on *Red Ereño*: an Example of Scientific Research as a Key Tool for Geoconservation

The research carried out and presented in this work provides data that enrich the knowledge of the studied ornamental stone. Although it is known that the use of *Red Ereño* through the history and its importance has arisen the outcrop of this stone as a geosite, no scientific information was published before about why it is red, how this rock became red, and when did it occur. This information is necessary in order to enhance its scientific/intrinsic value (e.g. Forno et al. 2022).

The produced data focused on the origin of the red colour of Ereño limestone enables us to generate adequate material for the different items presented in the "Introduction" section (Fig. 14).

(1) Geosite. The performed research on the study of the red colour of the Ereño limestones completes the knowledge about its geological and diagenetic history. In this work, one of the most relevant questions about *Red Ereño* limestones is answered: why is red? Mineralogical studies indicate the presence of iron oxides in both the red and grey limestones. However, these oxides present different oxidation forms in red and grey limestones, hematite, and magnetite crystals, respectively. So, only the hematite form gives its characteristic red colour to *Red Ereño*. This aspect would need further study to understand why the hematitic oxidation form is only present in a limited band of the Santimamiñe limestone unit. Moreover, the applied magnetic studies confirm the micron size of the hematite, which can be interpreted as pigmentary hematite. This explains why although present in very scarce amounts (usually under 1%) and not detectable under ordinary mineralogical techniques (e.g. XRD), the hematite present in the matrix of the limestone has so much staining power.

A deep petrological analysis resulted in constraining how the process of hematite introduction and/or precipitation occurred. The analysis concludes that the micron-sized hematite crystals are only present in the matrix of the limestones, even concentrated in the stylolitic surfaces, but not in the interior of fossil shells. These data indicate that the hematite crystallised within the matrix, before burial diagenesis.

Finally, the palaeomagnetic analysis has allowed constraining the mineralising time interval. The results indicate that hematite was formed during the extensional phase and prior to the inversion of the Basque-Cantabrian Basin due to the Alpine convergence. The presence of other hydrothermal mineralisations and volcanism during the upper Albian to Cenomanian time interval in the central part of the basin (where the study area is located) suggests that this time interval could be an appropriate candidate for the red-staining mineralisation of Ereño limestones. Although less probable, later Late Cretaceous times (Cenomanian, Turonian, and Santonian) cannot be discarded, since there are also volcanic episodes registered for that period in the basin (Fernandez-Carrasco et al. 1991, 1992; Castañares et al. 1997).

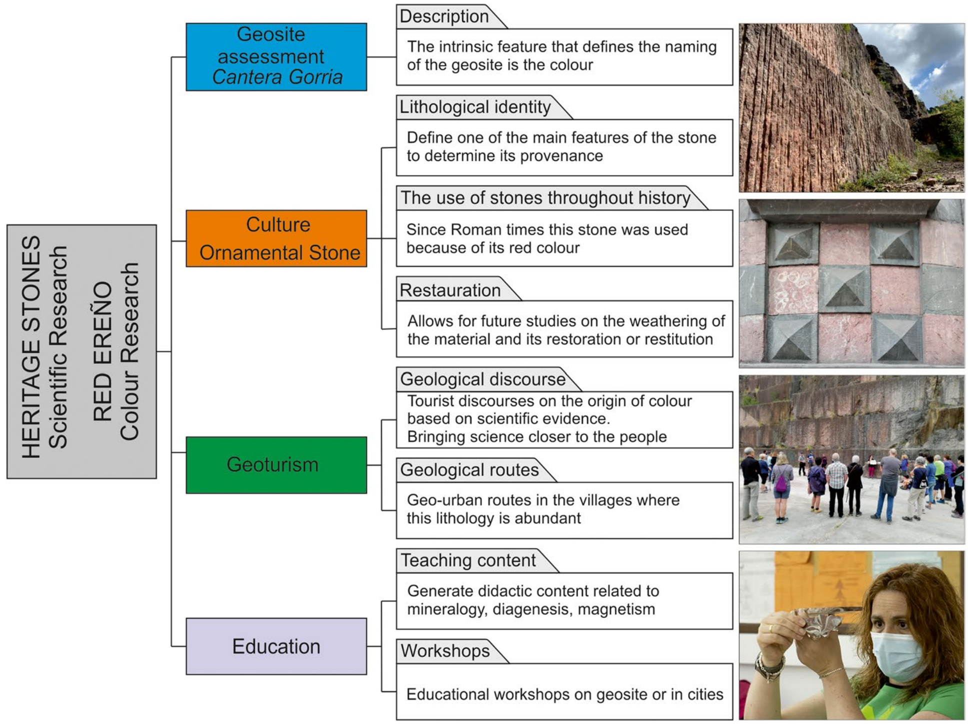

Fig. 14 Diagram of the potential of scientific research on ornamental stones. Example of the application in the study of the colour of *Red Ereño* (from top to bottom pictures correspond to: main quarry front of *Cantera Gorria*; detail of the *Victor Chávarri* Palace frontage (Bilbao); guided visit to *Cantera Gorria*; practical activity with high-school students in the University of the Basque Country)

(2) Cultural heritage. The results obtained from this investigation complement the information related to the ornamental character or *Red Ereño*. The study of the red colouring and how is the colour distributed within the matrix allows its discrimination from other similar limestones such as *Red Baztán* (Damas Mollá et al. 2013) which is commonly used for its replacement. Related to this, the *HGI research group* (*Hidro-Geo-Ingurumena/Hidro-Geo-Environment*) is currently developing an inventory to establish the extent of the use of the *Red Ereño* through the recent history, not only highlighting the construction and ornamental elements but also their dating. In the near future, public participation will be promoted in order to obtain more data that will be finally be open access, in collaboration with official entities such as *Basque Energy Agency* (EVE) and the provincial administration *Department of Culture of the Provincial Council of Biscay* (BFA); hence, it is a first step in the conservation of this rock from the indiscriminate replacement during building refurbishments. Since local stones confer identity to urban scenery (Damas Mollá et al. 2022), this collaboration will improve the awareness concerning the need to preserve original stones to maintain the heritage-identity of communities and thus differentiate themselves from cliché cities. The preservation of heritage is necessary although aesthetic preferences of the society change over time.

(3) Story-telling for geotourism activities. Dissemination is making scientific knowledge accessible to the society. For that purpose, speech has to be simple but maintaining scientific rigour. The main attraction of the tourism and dissemination activities concerning *Red Ereño* is its red colour, and that is why, this investigation and its results are relevant. The singularity of the colour of this rock permits bringing closer geological knowledge to the society in different ways: touristic activities visiting outcrop quarries (*Cantera Gorria*), urban tours visiting emblematic buildings, generating dissemination literature, and other activities (Geolodía 2023).

(4) Story-tellings for educational or interpretative. In the case of educational content, the red colour is the main feature that makes it singular and easy to identity. This gives the possibility to do many educational activities from the lowest levels (elementary school) to the highest (university/academy) (Fig. 14). For instance, activities and workshops are being carried out with school students from Gautegiz Arteaga (one of the villages where *Red Ereño* was extracted).

Conclusions

This study documents that the presence of pigmentary hematite gives its characteristic reddish colour to the Ereño limestone. The hematite crystals precipitated after the start of diagenesis but before the burial diagenetic stage, based on the shielding of the fossil shells that maintained their original white colour and the concentration of hematite in stylolitic surfaces due to chemical compaction during burial. The use of magnetic experiments in order to determine the presence of particular minerals in low concentrations has been validated. Furthermore, palaeomagnetic techniques allowed constraining the time interval for the mineralisation during the Late Cretaceous. Moreover, based on the geological context, the Albian-Cenomanian interval could be the most appropriate candidate for the occurrence of this process.

The scientific analysis developed on *Red Ereño* limestones not only enhances this stone knowledge but also generates information for other purposes. It has increased the knowledge about the geosite of the biggest quarry of *Red Ereño* (*Cantera Gorria*). Therefore, taking into account the use of this rock as an ornamental stone, studies on the minerals that give its colour are a priority when defining the aspects that make this lithological variety unique. These data provide additional information in order to its identification in historical monuments and their correct restoration. Concerning geotourism, this investigation answers the most repeated question in geourban tours related to *Red Ereño*, which is "why is it red?". Thus, this work provides information for scientifically rigorous geostory-tellings. Finally, regarding educational resources, the singular red colour of this rock permits developing pedagogical activities in several educational levels.

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Author Contribution All authors contributed to the study conception and design. Material preparation, data collection, and analysis were performed by Laura Damas Mollá, Arantza Aranburu Artano, Juan José Villalain, and Francisco García-Garmilla. The first draft of the manuscript was written by Laura Damas Mollá, Arantxa Bodego, and Arantza Aranburu, and all authors commented on previous versions of the manuscript. All authors read and approved the final manuscript.

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Data Availability Not applicable.

Declarations

Competing Interests The authors declare no competing interests.

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