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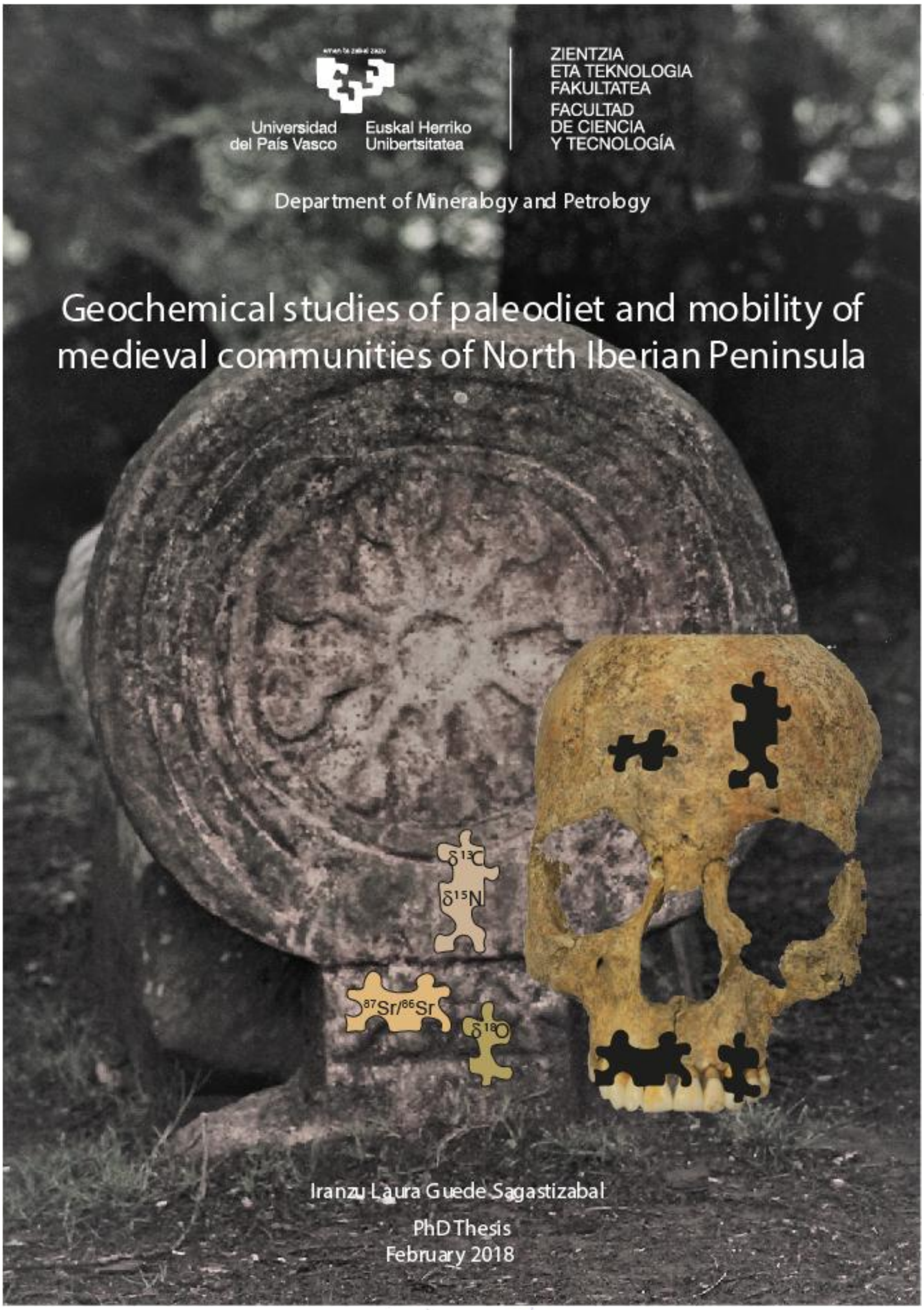
Department of Mineralogy and Petrology

Geochemical studies of paleodiet and mobility of medieval communities of North Iberian Peninsula



Iranzu Laura Guede Sagastizabal

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Mila esker nire zuzendariei, Luis Angel Ortega doktoreari eta bereziki, Maria Cruz Zuluaga doktoreari, geologiaren beste alderdi batzuk ezagutzeko aukera emateagatik. Nork esango zidan geologia, karreran zehar ikusi eta ikasitako arrokak, mineralak eta fosilak baino gehiago zela.

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Amaitzeko garrantzitsuena utzi dut. Tesia idazten bukatzen nengoela Jazz joan zitzaigun. 14 urtez nire ondoan egon ondoren leku berezia gorde nahi izan diot memoria honetan. Ondorengo estrofek (Gontzal mila esker hitz eder hauengatik) ondo baino hobeto laburbiltzen dute gure historioa.

“Hainbeste urtez itxaron ondoren
ustekabean agertu zinen
babesik gabe eta maitasun eske
Edonork animaliarik abandonatuko luke?
Zorionak zorion, aurkitu zintuzten
etorri berri zinela korrika hasi zinen egiten
argazki, marrazki, koadro eta guzti
irudi guztietan bezain azti.
Denborak, hala ere, ez du barkatzen
pixkanaka hasi zinen itzaltzen
istorio guztien antzera
hasierak ere badu bere amaiera.
Horrela joan zinen, lagun
zure musikaren memoriak ez utziriz oso urrun.”

(Gontzal Guede)

Abstract

Stable isotopes ($\delta^{13}C$, $\delta^{15}N$, $\delta^{18}O$) and a radiogenic isotope ($^{87}Sr/^{86}Sr$) in human bone and tooth remains have been used to reconstruct residential mobility and dietary patterns. The isotope study has been conducted for several medieval archaeological sites in different geographical areas and time periods, in the North Iberian Peninsula from the 6th to 12th centuries. The archaeological sites that have been studied are three Christian graveyards (Alegria-Dulantzi, Alava; Las Gobas, Burgos and San Juan de Momoitio, Biscay) and one Muslim cemetery (Tauste, Zaragoza) dated between the 6th and 12th centuries.

Carbon and nitrogen isotope measurements have been performed in bone collagen extracted from human individuals and from faunal bone samples. Additionally, teeth and bone samples have analyzed for strontium and oxygen isotope studies. Complementary with the isotope studies of the Muslim population of Tauste, trace elements have been analyzed in tooth samples by means of the LA-ICP-MS technique to investigate health and palaeodietary patterns.

Strontium isotope analysis of human bone remains corresponding to 33 individuals from San Martín de Dulantzi (Alegría-Dulantzi, Álava, Spain) graveyard has revealed that most individuals exhibit values similar to the domestic fauna isotope composition, indicating local origin or long residence time in the region. When bone data are compared with tooth enamel values, two groups of non-local individuals from distinctive geological environments have been established. However, the non-local individuals are distributed through

the studied period of time, which suggests that migration movements were limited in number. Thus, the Dulantzi population was mainly a local society with some influxes of foreigners.

Strontium, carbon and nitrogen isotopes of human bone and tooth remains of 40 individuals have been analyzed at Las Gobas (Burgos). Strontium values have indicated that most non-local individuals were women and infants and that residential mobility took place in the 10th and 11th centuries coinciding with the formation of Laño village.

Carbon and nitrogen isotope ratios of bone collagen indicate a homogenous omnivorous diet based on terrestrial plant resources, with few animal-derived proteins from livestock. Millet consumption has been established although it was restricted to an earlier period of time (7th-9th centuries) and replaced in later periods (10th-11th centuries) by wheat and barley. In general, within the Laño population, no dietary differences are seen between individuals according to sex or age.

Strontium, oxygen, carbon and nitrogen isotopes in bones and teeth from a total of 93 individuals have been analyzed at San Juan de Momoitio (Bizcay). Oxygen and strontium isotopes indicate that most individuals were from the immediate vicinity of Momoitio and only five individuals were non-local and would have come from Atlantic Ocean coastal areas (probably from south-western France).

Carbon and nitrogen values suggest similar foodstuff resources and diet to the Momoitio populations, based on cereals (both C4 and C3 types), vegetables and pulses, and animal protein derived from husbandry and scarce marine resources. The observed variations in dietary habits do not reflect gender inequality but illustrate rural social organization and changes in medieval rural society. Contrary to the Las Gobas site, millet consumption increased during the later period coinciding with the gradual abandonment of the graveyard.

Finally, 31 individuals from the Islamic necropolis of Tauste (Saragossa, Spain) have been analyzed to determine their $\delta^{15}N$, $\delta^{13}C$, $\delta^{18}O$ and $^{87}Sr/^{86}Sr$ composition. The combination of strontium and oxygen isotopes indicates that most individuals were of local origin although three females and two males were foreigners. The origin of the two males would be from a warmer zone whereas two of the females would be from a more mountainous geographical region and the third from a geologically-different area. Tauste stands out among the studied archaeological sites because of the extremely high $\delta^{15}N$ baseline that has been determined and which is due to the bedrock composition (gypsum and salt). The individual high $\delta^{15}N$ values have been related to the manuring effect and consumption of fish. The isotope data also show adult males consumed more animal proteins than females and young males were considered the most privileged members of society in the medieval Muslim world.

Additionally, the chemical composition of 23 tooth enamel and dentine samples has been determined for the Muslim population in Tauste. The analytical results indicate different food intake between adult males, on one hand, and young males and females on the other. Thus, the chemical composition and isotope data reflect a similar palaeodietary pattern. Besides, chemical composition also allows the state of health of individuals to be investigated, since five individuals show lead intoxication suggesting occupational exposure to anthropogenic lead.

The multi-isotope study enhances the understanding of medieval Iberian lifeways. The strontium and oxygen isotope compositions suggest limited residential mobility because only a few individuals were of non-local origin. Determining the outsiders' provenance area is difficult but isotope data suggest nearby areas and only few individuals' origin has been impossible to determine. Palaeodietary patterns indicate similar foodstuff in medieval rural populations but with dietary differences among Muslim individuals according to sex and age, thus illustrating different dynamics of social life between Christian and Muslim populations.

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Introduction

Archaeologists mainly use different archaeological materials to reconstruct the lifestyle of ancient populations, including faunal and macrobotanical remains and pollen, as well as pottery residues, coprolites, and indirect sources such as skeletal pathology, or dental wear patterns (Reinhard, 2000; Gretchen and Stanley, 2001; Buzhilova, 2016, Jervis, 2014)). In recent decades, dietary and mobility research developed by means of the isotope and chemical composition of human bone and tooth.

Dietary habits and their change over time provide insight into social and economic structures and aspects such as sex, age or wealth within the different communities. Most dietary reconstruction is based on carbon and nitrogen isotope values in human skeletal tissues. However, alternative analyses based on the chemical composition of human teeth have recently been applied to determine palaeodietary patterns. The use of laser ablation-inductively coupled plasma-mass spectrometry (LA-ICP-MS) has enabled direct analysis of solid samples using only a small amount of material and minimal sample destruction (Kang et al., 2004; Speakman and Neff, 2005). The principle of the method is based on the chemical signature of skeletal tissues reflecting the signature of food consumed, because the composition of human bone and tooth enamel depends on types of food consumed (Ambrose and Katzenberg 2000).

On the other hand, residential mobility patterns were carried out by the study of grave goods buried with exhumed individuals (Brogiolo and Chavarria Arnau, 2008). Strontium and oxygen isotopes are two independent isotopic systems that in combination provide information about an individual's possible area of origin and thus mobility patterns (e.g. Bentley and Knipper, 2005; Evans et al., 2006a; Evans et al., 2006b).

In the present PhD thesis, the isotopic composition of human bone and tooth were analysed to explore mobility and diet of several Middle age populations. Additionally, tooth elemental composition was used as a complementary methodology to the stable isotopes of carbon and nitrogen to establish dietary patterns.

Residential mobility

In recent decades, strontium isotopes have been successfully used to identify nonlocal individuals and residential mobility in ancient burials all over the world. Out of all the published research, some of the most relevant studies will be referred to. Strontium isotope ratios and strontium concentrations in bone and tooth enamel have been used to investigate patterns of residential mobility and migration of prehistoric populations: in the United States (Price et al. 1994; Ezzo et al. 1997), in Europe (Grupe et al. 1997; Bentley 2001; Price et al. 2001; Bentley et al. 2002) and in Mesoamerica (Price et al. 2000). Cox and Sealy (1997) used strontium isotope analysis to obtain information about the possible origin of shipwrecked slaves. By means of strontium isotopes Sillen et al. (1995, 1998) reconstructed hominid habitat utilization and Hoogewerff et al. (2001) determined the last domicile of Europe's oldest known natural human mummy: the Tyrolean Iceman “Ötzi” found on the border between Austria and Italy.

Likewise, oxygen isotope signatures in archaeological human tooth enamel and bone are used to examine residential mobility, based on drinking water reflecting place of residence. Authors such as White et al., (1998, 2000, 2002, 2004a, 2004b, 2007) and Prowse et al., (2007) had used oxygen-isotope ratios of enamel phosphate of ancient populations to differentiate local and foreign individuals. Additionally, contemporary human populations have been studied using hair and urine samples (O’Brien and Wooller, 2007; Ehleringer et al., 2008). Similarly, scholars from a variety of disciplines have used oxygen-isotope in various species to reconstruct palaeoclimate (e.g. Reinhard et al., 1996; Stuart-Williams and Schwarcz, 1997; Shahack-Gross et al., 2003; Zazzo et al., 2006), animal migration and herding patterns (Killingley, 1980; Killingley and Lutcavage, 1983; Balasse et al., 2006) and the provenance of plants and animals (Kelly et al., 2005; Williams et al., 2005a; Benson et al., 2006; Dufour et al., 2007).

Strontium isotope

Naturally occurring strontium is composed of four stable isotopes : ^{84}Sr, ^{86}Sr, ^{87}Sr and ^{88}Sr. The isotopic composition of strontium atoms in the Earth's atmosphere is ^{84}Sr (0.56%), ^{86}Sr (9.86%), ^{87}Sr (7.0%) and ^{88}Sr (82.58%). Only 87Sr is radiogenic and is formed by the radioactive decay of rubidium. The Rb-Sr decay system has been widely used in geochronology and remains one of the most useful geochemical tracers. The amount of rubidium in a rock and its age determines the amount of ^{87}Sr in the bedrock, and therefore the $^{87}Sr/^{86}Sr$ isotope ratio (Faure and Powell, 1972). Rb is a highly soluble and incompatible element, while Sr is also relatively soluble but not quite as incompatible, which is to say that Sr has a smaller ionic radius than Rb, being more compatible in silica-rich igneous systems. Very old rocks (>100 mya) with high Rb/Sr have $^{87}Sr/^{86}Sr$ ratios generally above 0.710, while rocks formed recently (<1–10 mya) with low Rb/Sr ratios have low $^{87}Sr/^{86}Sr$ ratios generally less than 0.704. The Earth's mantle has a relatively uniform and low $^{87}Sr/^{86}Sr$ ratio, about 0.702–0.704 in basalts erupted along mid oceanic ridges, or oceanic islands such as the Hawaiian chain (White and Hofmann, 1982). In oceanic island arcs (e.g. Aleutian Islands, Japan, Vanuatu), formed by subduction-related magmatism of mantle/crust mixtures, $^{87}Sr/^{86}Sr$ ratios range from about 0.7035 to 0.707 (Dickin, 1995). Phanerozoic marine limestone and dolomite have intermediate $^{87}Sr/^{86}Sr$ ratios of about 0.707–0.709, reflecting the composition of the ocean during their deposition. $^{87}Sr/^{86}Sr$ ratios in rocks of the continental crust vary between 0.702 and 0.750, including older granites, with $^{87}Sr/^{86}Sr$ ratios typically above 0.710 and as high as 0.740, to younger basalts, with lower $^{87}Sr/^{86}Sr$ ratios around 0.703–0.704. These variations are large relative to the instrumental error of modern mass spectrometry measurements (typically ±0.00001 or better).

Strontium is present in variable concentrations in the bedrock, groundwater, soil, plants, and in the animals of a given ecosystem. Although the strontium concentration in organisms varies in accordance with their trophic level, $^{87}Sr/^{86}Sr$ isotope ratio in plants, animals and groundwater of a given geological region, and also in humans that consume these resources, will reflect the bedrock $^{87}Sr/^{86}Sr$ values (Price et al., 2002; Bentley, 2006; Malainey, 2011) because is not fractionated by biological processes (Graustein, 1989; Capo et al., 1998; Blum et al., 2000) (Fig. 1).

Strontium substitutes for calcium in the hydroxyapatite of bones and teeth. The isotope ratios of strontium in teeth and human bones reflect the $^{87}Sr/^{86}Sr$ isotope ratio in the food and water ingested during the individual's lifespan (Ericson, 1985, 1989; Price et al., 1994, 1994a). Individuals who have consumed local foods and have lived in a specific geological region will have $^{87}Sr/^{86}Sr$ isotope ratios in their teeth and skeleton reflecting their living area. In addition, dental enamel, in contrast to bones, does not incorporate other elements after its formation in infancy (Hillson, 1986, 1996). Thus, the $^{87}Sr/^{86}Sr$ ratios in tooth enamel reflect the composition of the place of residence during childhood. In contrast, bones continually regenerate and incorporate strontium (Parfitt, 1983; Smith, 1991) so the $^{87}Sr/^{86}Sr$ ratios reflect the composition of the place of residence for the last few years of life.

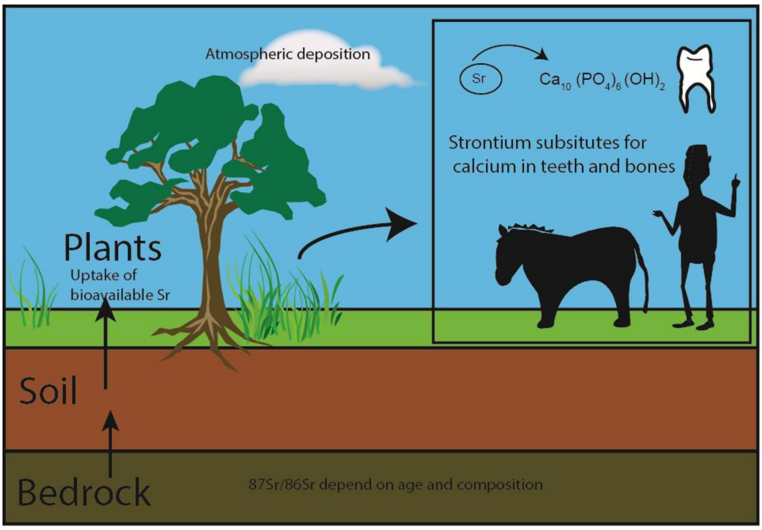

Figure 1. The strontium isotope cycle.

It is crucial to determine the local $^{87}Sr/^{86}Sr$ baseline to be able to identify non-local individuals (Bentley et al. 2004; Price et al. 2002, Tütken et al. 2011). There are several methods to establish the local baseline of the isotope signature by analyzing environmental samples, including freshwater, soil leachates, ancient fauna and present-day small wild animals (Price et al., 2002; Bentley et al., 2004; Evans et al., 2010; Tütken et al., 2011). However diagenetic processes could modify the strontium isotope ratios of archaeological materials and anthropogenic activities such as the use of fertilizers could modify the strontium isotope ratios of modern ecosystems (Böhlke and Horan, 2000; West et al., 2009; Tichomirowa et al., 2010; Christian et al., 2011). So these parameters have to be taken into consideration when performing the mobility studies.

Oxygen isotopes

Oxygen has four stable isotopes naturally occurring: ^{16}O, ^{17}O, and ^{18}O. The isotopic composition of oxygen atoms in the Earth's atmosphere is 99.759% ^{16}O, 0.037% ^{17}O and 0.204% ^{18}O. The relative amounts of ^{16}O and ^{18}O isotopes in a sample of water, ice, rock, plant, human, etc. is a function of climate/environment. The relative amounts are expressed as either $^{18}O/^{16}O$ or $\delta^{18}O$; the calculation formula is as follows:

$\delta^{18}O$ (in ‰) = [($^{18}O/^{16}O$) sample – ($^{18}O/^{16}O$) standard]×1000 / ($^{18}O/^{16}O$) standard.

The lighter ^{16}O evaporates more easily while the heavier 18O is easier to condense out in isotope fractionation (Fig. 2). The oxygen isotope reflects the isotopic composition of ingested water that is derived from meteoric water.

The $\delta^{18}O$ in precipitation varies regionally according to temperature and other climatic parameters, such as distance from the coastline, altitude and latitude (Longinelli, 1984; White et al., 1998;

Darling et al., 2006; Daux et al., 2008). Oxygen isotopes in the body are subject to several steps of metabolic fractionation. The fractionation mechanisms are relatively well known, allowing the calculation of approximate drinking water ($\delta^{18}O_w$) values from the $\delta^{18}O_p$ of biogenic phosphate by means of conversion equations (Longinelli, 1984; Daux et al., 2008; Luz et al., 1984; Luz and Kolodny, 1985; Levinson et al., 1987; Bryant and Froelich, 1995; Kohn, 1996; Iacumin and Venturelli, 2017). The oxygen isotope composition of human remains allows the identification of palaeomobility patterns despite difficulty in the calculation of meteoric water isotope composition in the past.

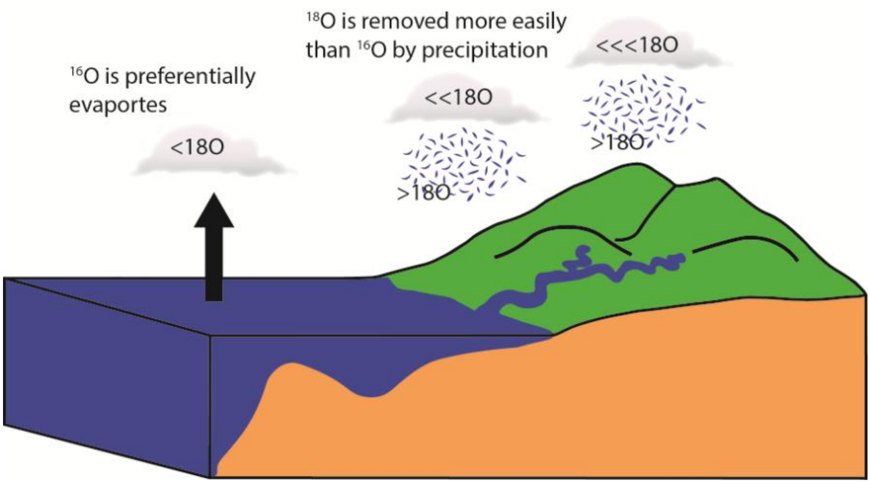

Figure 2. Rainout effect on δ^{18}O values (based on Hoefs 1997 and Coplen et al. 2000).

In a similar way to strontium, oxygen is fixed in teeth and bones through ingested water. Standard practice for investigating mobility of past people with isotopes is to measure the isotopic composition in the body tissues (for example $^{18}O/^{16}O$) and compare the composition with the distribution of the same isotopes in the local and wider environment.

In most archaeological studies, the skeleton is the material chosen since is the tissue most likely to survive in the burial environment. Oxygen isotopes of bioapatite phosphate and carbonate ($\delta^{18}O_p$ or $\delta^{18}O_c$) are measured in teeth or bones and then the values are converted to their equivalent water $\delta^{18}O_w$ values by means of specific conversion equations. The equations available in the literature take into account the physiological effects of the different isotopes during the integration into the tissues. The calculated values are subsequently compared to variation patterns of rainwater or groundwater, either obtained from the literature or extrapolated from the general distributions of isotope ratios in precipitation. Thus, non-local individuals are identified by comparing the difference between skeletal $\delta^{18}O_w$ values and the drinking water. However, the isotope composition in the environment and in living individuals does not vary in exactly the same way and the relationship between the measured value and the value expected of drinking water is not straightforward. Hence, the biological values recorded in local populations may be consistent or not with the predicted values. Moreover, the oxygen isotope values recorded in people living in a specific area may be influenced by other factors. Short-term climate conditions (warmer/colder, wetter/drier periods) occurring during teeth formation in childhood can affect the isotope composition leading to atypical $\delta^{18}O$ values consisting of ^{18}O-enrichment or ^{18}O-depletion. Mean annual water values used for comparison correspond to the average of a period of time, normally 10 to 30 years, that is a period longer than required to mineralise the tooth. Besides, sourcing drinking water from reservoirs other than the local groundwater such as from rivers flowing from higher latitudes or from lakes or ponds, may also contribute to alter the individuals' expected skeletal $\delta^{18}O$ values. The water used for food cooking can also contribute to offset skeletal $\delta^{18}O$ values from the drinking water values. Boiling, brewing and other cooking practices cause shifts in the isotope values of fresh food and drinking water, often tending to produce enrichment in 18O. Finally, analytical errors associated with the mathematical conversion from $\delta^{18}O_p$ to $\delta^{18}O_w$ may lead to additional modifications of the expected water values. Despite complexities in the calculation of meteoric water isotope composition in the past, the oxygen isotope composition of human remains allows the identification of palaeomobility patterns.

However, oxygen and strontium isotope compositions are not perfectly predictable, because of variability in available food over time, and because they reflect an average value of the geological and geographical composition of the provenance of food ingested during childhood. Therefore, isotope values are used to predict the most likely geographic links between tissue and location.

Dietary reconstruction

The use of stable carbon and nitrogen studies to reconstruct the diet of a past population is a well-established method in archaeology. Besides in human investigations, stable carbon and nitrogen are used to determine diet and ecology for extinct and actual species. The method is based on the principle that the isotope composition of a consumer's collagen reflects the isotope signature of the intake. Nevertheless, the isotope composition does not identify particular foods or diet (Richards and Hedges 1999) but allows the main sources of protein in the diet to be recognised (Ambrose and Norr 1993)(Fig.3).

Until recently, application of the stable carbon and nitrogen methods has been limited to archaeological sites and usually to ages not older than a few thousand years (e.g. Van der Merwe, 1982; DeNiro, 1987, Bocherens et al., 1991). Exceptionally well-preserved soft tissues of mummified mammals from Alaska have been investigated for their carbon isotopic abundances (Bombin and Muehlenbachs, 1985). Also carbon and nitrogen isotopic composition in fossil collagen has been used for palaeodiet determinations of Pleistocene mammals, including Neanderthal man (Bocherens et al., 1991; Richards et al., 2008), cave bear (Bocherens et al., 1994a, 1990) and mammoth (Tutken et al 2007; Bocherens et al., 1994b). Palaeodietary investigations using isotopic biogeochemistry have also been attempted on late Cretaceous dinosaurs (Ostrom et al., 1990; Bocherens, 1992). Carbon isotopic composition in enamel has been used to infer the palaeodiet of fossil baboons (Lee-Thorp et al., 1989), australopithecines (Lee-Thorp 1989; Van der Merwe et al., 2003) and extinct ungulate species (Lee-Thorp, 1989, Codron et al., 2007) as a tool for tracking palaeoenviromental changes (Drucker et al., 2003) or breastfeeding and weaning patterns in archaeological populations (Katzenberg and Pfeiffer, 1995; Schurr, 1997, 1998; Herring et al., 1998; Wright and Schwarcz, 1998, 1999; Dupras et al., 2001; Mays et al., 2002; Schurr and Powell, 2005; Fuller et al., 2006).

In the mineralized tissues, carbon is present in the organic phase i.e. in the collagen that constitutes 90% of bone and dentine and in the inorganic phase (Lee-Thorp, 1989). In contrast, nitrogen is a major component of proteins, i.e. in collagen, which is the predominant protein in bone and dentine.

In order to infer palaeoecological information in fossils, the preservation of the isotope signature is absolutely necessary. With regard to organic matter, it has been established that (1) carbon and nitrogen isotopic abundances are not altered by diagenesis if the amino acid composition of extracted organic matter is similar to that of collagen, and (2) the quality of preservation seems independent of the amount of preserved organic matter (DeNiro and Weiner, 1988; Bocherens et al., 1991a, 1993a; Fizet, 1992). The best way to check the quality of extracted organic matter is by determination of amino acid composition and a simple method is by determining the C/N ratio. Ratios of about 3.0 in collagen indicate good quality of the collagen (DeNiro, 1985).

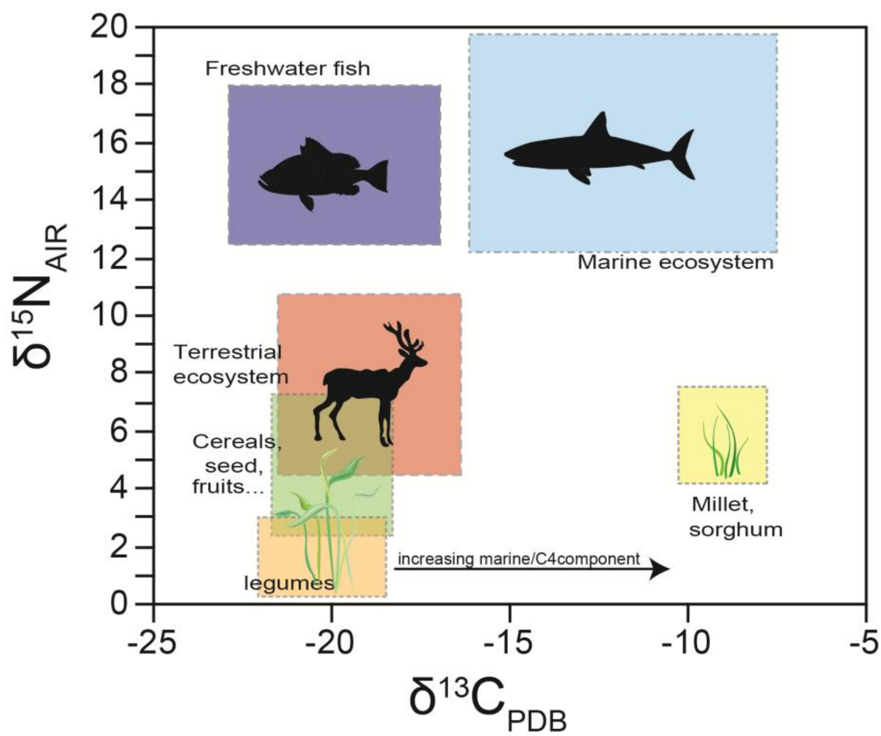

Figure 3. Plot of $\delta^{13}C$ and $\delta^{15}N$ ecosystem boxes.

Carbon isotopes

Carbon isotope abundance is expressed as $\delta^{13}C$ values as parts per thousand (‰) and is calculated with respect to the Pee Dee Belemnite (PDB) standard as follows:

$\delta^{13}C$ (‰):[(13Rsample/13Rstandard)--l] X 1000

(where laR= $^{13}C/^{12}C$ and standard is PDB)

It follows that eating different plants will affect the $\delta^{13}C$ values in the consumer's body tissues.

Carbon isotopes are linked to diet by the intermediary of the foodweb from the primary producers, which are photosynthetic plants. Most plants are of two types: C3 and C4 plants, whereas a third group is named CAM. The CAM plants exhibit the crassulacean acid metabolism pathway and use either the C3 or C4 photosynthetic pathway, depending on environmental conditions.

Thus, carbon isotope analysis provides information about the plant types consumed and the ecosystem the foodstuffs come from, distinguishing between terrestrial and marine ecosystems. In the case of a terrestrial diet, the C_3 plants comprise most vegetables including most trees and shrubs in temperate regions such as north-west Europe and northern Spain. They also include different types of crops, wheat (*Tritium*), barley (*Hordeum vulgare*) and oats. The C_4 plants grow in more arid environments and include millet (*Pennisetum*), maize (*Zea mays*) and sugar cane (*Succharum officinarum*) (Smith and Epstein 1971).

Carbon originated during photosynthesis is depleted of the heavier isotopes. Thus C_4 plants exhibit greater enrichment in carbon values than C_3 plants, resulting in mean $\delta^{13}C$ values of -13‰ and -27‰ respectively (Epstein, 1971; O'Leary, 1981). Marine plants are all C3 plants and exhibit mean $\delta^{13}C$ values about 7.5‰ higher than terrestrial C3 plants. Carbon isotope composition can also be used to distinguish marine protein consumption in terrestrial C3-based diets, but when C4 plants are involved marine and terrestrial values can overlap (Hoefs, 2009; White, 2015). Carbon fractionates in $\delta^{13}C$ by only about 1‰ throughout the food chain (Ambrose, 1993; DeNiro and Epstein, 1978; Hedges and Reynard, 2007; Malainey, 2011). In freshwater ecosystems the $\delta^{13}C$ composition of plants is variable and consequently freshwater fish exhibit a broad range of $\delta^{13}C$ values that are largely depleted (Dufour et al., 1999; Pazdur et al., 1999). Therefore, $\delta^{13}C$ ratios more negative than -22‰, the value corresponding to the low end of a diet based only on C3 terrestrial plants, suggest freshwater fish consumption.

In addition, when both types of plants are present in a given environment, it is also possible to quantify the amount of C3-plants and C4-plants consumed by vertebrates. The application of these techniques is now widespread in tropical Africa (e.g. Vogel, 1978; Ambrose and DeNiro, 1986; Tieszen and Boutton, 1988). Moreover, carbon isotopes can be used to reconstruct terrestrial palaeoenvironments, providing information regarding the presence of C3 and C4 vegetation in ecosystems and the degree of canopy cover versus openness in habitats. Herbivore carbon isotopic compositions reflect the source of carbon in the diet, indicating the proportions of C3 and C4 vegetation in their habitat and the degree of canopy closure or openness in wooded habitats (Van der Merwe and Medina, 1991; Palmqvist et al., 2003; MacFadden et al., 2004; Kohn, 2010). C4 plants are adapted to hot growing seasons (above 20°C, monsoon system) usually in tropical, low-latitude environments (Bender 1968; Smith and Epstein 1971) whereas C3 trees are adapted to all tropical, temperate and boreal environments and C3 herbaceous plants to temperate and cold or cool high-altitude regions with cool growing seasons (Vogel et al. 1978). Both types of plants are 13C-depleted relative to atmospheric CO_2, and exhibit different isotopic signatures. In forest environments, differences in $\delta^{13}C$ composition can result in a vertical $\delta^{13}C$ gradient known as the canopy effect (Van der Merwe and Medina 1991). The canopy effect has also been observed in pure C3 environments between the herbivores feeding in a heavily enclosed wooded environment and more open forest environments (Drucker et al. 2003; Feranec and MacFadden 2006; Drucker et al. 2008; Drucker and Bocherens 2009).

Nitrogen stable isotopes

Nitrogen isotope values reflect the intake of animal proteins and provide information about the trophic level of an individual (Lee-Thorp, 2008; Sandford, 1993; Bocherens and Drucker, 2003). Nitrogen isotopes are expressed as $\delta^{15}N$ values as parts per thousand (‰):

$\delta^{15}N$ (‰) = [(15Rsample/15Rstandard) - -1] x 1000 where lSR = $^{15}N/^{14}N$

and the standard is atmospheric nitrogen (Mariotti, 1984). Thus, nitrogen isotopes in terrestrial ecosystems are enriched in $\delta^{15}N$ by 2-5‰ (on average, 3‰) from food to body tissue as trophic levels increase (Reynard, 2007; Schoeninger and Moore, 1992). Terrestrial protein sources have $\delta^{15}N$ values ranging from 5‰ to 12‰, while aquatic food sources range from about 12‰ to 22‰ for marine fish and 7.2‰ to 16.7‰ for freshwater fish (Schoeninger and DeNiro, 1984; Walker and

DeNiro, 1986; Katzenberg and Weber, 1999; Fuller et al., 2012; Robson et al., 2015). When C3 plants are consumed, nitrogen isotope analysis is combined with carbon isotope analysis to distinguish between proteins derived from terrestrial, freshwater and marine resources. Other factors to considerer in the interpretation of the isotope composition of collagen include consumption of brackish estuarine species or marine resources (Salazar-Garcia et al. 2014).

Other reasons for variability in $\delta^{15}N$ ratios of plants and animals include natural environmental conditions such as salinity and aridity or anthropogenic factors like manuring (Bogaard et al. 2007; Fraser et al. 2011). The use of manure as fertilizer causes an increase in $\delta^{15}N$ values and consequently, domestic animals foddered with manured chaff and grains will present higher $\delta^{15}N$ values. Natural environmental conditions such as salinity or aridity also cause $\delta^{15}N$ values to increase (Malainey 2011). Since the isotopic composition can vary according to the environment, it is necessary to establish the local composition. For this purpose the palaeofauna found in the cemetery under study is usually used. In general, human diet corresponds to a mixture of food with different isotope signatures. Plots of collagen $\delta^{13}C$ vs $\delta^{15}N$ values can be interpreted as mixtures of multiple components (Schwarcz and Schoeninger, 1991; Phillips and Gregg, 2003) that do not yield unique solutions, but may outline the dominant components in the diet of the studied individuals.

Moreover, stable nitrogen isotope analysis can also be used to investigate breastfeeding and weaning practices. In fact, during breastfeeding, children exhibit $\delta^{15}N$ values enriched about 2-3‰ over that of their mothers (Fogel et al., 1989). Besides, the magnitude of nitrogen isotope enrichment is not only able to determine breastfeeding and weaning practices but also to explore weaning practices and infant mortality (Katzenberg et al., 1996; Schurr, 1997; Pearson et al., 2010). The introduction of solid food, possibly plant-based, and the lack of breast milk causes disease and nutritional stress as a result of nutritional deficiency.

Trace elements analysis on teeth by LA-ICP-MS

Trace element analysis has been used as complementary to the carbon and nitrogen isotope analysis to study dietary habits. Trace element analysis not also provides knowledge of nutrition but also of health.

Trace elements are those compounds present in the human diet to maintain normal physiological functions. However, some microelements may become harmful at high levels of exposure, or, on the other hand, may give rise to malnutrition, when their exposure is too low. While some elements are essential to health, other elements are likely to be essential (e.g. Ni, B, Va), although their positive role in human nutrition remains to be confirmed (e.g. Cr, Co, Cu, Fe, Mo, Mn, Zn etc.) (Kalicanin and Nikolic, 2008; Chew et al., 2000; Al-Mahroos and Al-Saleh, 1997). In addition, other elements have no proven essential functions in humans and are likely to have adverse physiological effects (e.g. Al, As, Li, Sn) (1). Furthermore, some elements (e.g. Pb, As, Hg, Cd) are well known to be toxic if their exposure through diet and/or inhalation is excessive (World Health Organization, 1996). On the other hand, some elements (rare earths, Th and U) are related to diagenetic processes and indicate that the original geochemical composition of the tooth has been altered (Kohn et al., 1999; Longerich et al., 1996). Nevertheless, the choice of analyses should be based on the ecological complexity of the site and whatever archaeologists are able to provide about diet.

The amount of trace elements ingested by an individual depends on dietary habits. Teeth are principally composed of nearly 40 elements present in enamel and dentine, ranging from >1000ppm (i.e. Zn, Sr, Fe, Ba) to <100ppb (i.e. Ni, Hg, Li). Variations in the content of trace elements in the teeth have been demonstrated previously (Brown et al., 2004). Trace elements allow the characterization of the relative amounts of particular plant types and animal types, as well as discriminating between plants versus meat food in the diet because they are widely distributed in food (Gilbert, 1977; Schoeninger, 1979; Price and Kavanagh, 1982; Sillen and Kavanagh, 1982; Connor and Slaughter, 1984; Brown and Blakely, 1985; Byrne and Parris, 1987).

Objetives

To understand the dynamics of historical socio-economic systems, biological systems are more confusing than geochemical ones. Multi-isotopic studies, including radiogenic strontium, stable oxygen, carbon and nitrogen, and chemical analysis of bones and teeth do not open a clear window to the past but provide information about nutrition, life history, dietary sources and mobility of past populations. They are also able to reconstruct regional identities by characterizing places of birth and residence and thus establish social dynamics.

Carbon and nitrogen stable isotopes provide insights into dietary habits whereas strontium and oxygen isotopes contribute information about residential movements. In addition, trace element analysis of teeth not only allows palaeodietary patterns to be investigated but also provides information about health.

The aim of this study is to analyze the variation in isotope and trace element composition of bones and teeth as a record of dietary habits in rural communities belonging to different cultures in the north of the Iberian Peninsula. For this purpose, it is first essential to differentiate local individuals, i.e. those born and resident in the studied community, from the non-local individuals. The present study has been carried out in several medieval archaeological sites, dated from the 6th to 12th centuries, corresponding to a limited regional area but culturally diverse populations (Christians and Muslims). They represent a historical time marked by a number of invasions, a mixture of cultures and landscape transformation.

Summary of the findings

This report presents the results of the research carried out and the discussion through articles published (or in progress). Therefore, before the chapters corresponding to each of the articles, this summary will provide an overview of the findings that make up the body of this doctoral thesis.

As previously discussed, the objective of this work has been the study of different populations in the North Iberian Peninsula during the Early Middle Ages, based on the characterization of diet and mobility patterns through the analysis of stable carbon and nitrogen, oxygen and strontium isotopes and trace elements. This study will enhance the understanding of medieval northern Iberian ways of life based on diachronic and isolated cases with different social issues.

This research has involved multi-isotope analysis of human skeletal remains from rural medieval Christian graveyards and a Muslim cemetery in the North Iberian Peninsula, dated between the 6th and 12th centuries. The selected archaeological sites represent different populations in distant geographic areas within the North Iberian Peninsula dated in the Middle Ages, enabling a more profound understanding of the social organization during this historical period of time. This report brings together the isotopic results and discussion of these communities and is structured so that each chapter corresponds to a specific site.

As already mentioned, the study focuses on the northern Iberian Peninsula during the medieval period. In history, the Middle Ages began with the fall of the Roman Empire. The political situation and social instability facilitated invasions and large movements of people from European territories, which are known as the Germanic migration.

To seek the real impact of these migrations and the historical role of Germanic elites, individuals buried with Germanic grave goods have been researched across Europe. However, these studies do not assist in the cases of individuals buried without Germanic grave goods, a situation corresponding to most burials even in so-called “Germanic cemeteries”. Thus, in Chapter I the study of a rural community with “Germanic cemeteries” in a broad time frame will allow the establishment of mobility patterns and detect the presence or absence of the mentioned mass migrations in the Middle Ages. This site is the graveyard of Alegria-Dulantzi (Alava, North Iberian Peninsula) which was in use between the 6th and 12th centuries. Strontium isotope composition was analysed to determine the origin of exhumed individuals. The results showed that the Dulantzi population consisted mainly of a local society with some foreign individuals, although their precise geographical origin cannot be established. The migration cycles occurred during the 6th-9th centuries, involving the movement of a very limited number of adults and youths of both genders. With the exception of Individual 204, none of the immigrants was buried with grave goods. The data question the real impact of supposed large migrations and are consistent with isotope studies performed in other European sites.

After the collapse of the Roman Empire not only movement of people occurred but a profound transformation affecting territorial organization. Unlike other parts of the Iberian Peninsula, fortresses did not articulate the territory in this region. In the Cantabrian region of north Spain, the post-Roman landscape showed a high degree of territorial fragmentation with a lack of villages or rural structures. In the course of the 9th century, peasant settlement densification occurred with the creation of true village networks. Besides, in AD 711 Muslims invaded most of the Iberian Peninsula and remained for the next seven centuries, until 1492 when the Christian Kingdoms totally reconquered the peninsula. Although the Muslim occupation was almost total, in the northern part of the Peninsula, to the north of the Cantabrian mountain range, the territory continued under Christian rule (Upper March).

In this context, Chapters II and III provide two cases of rural communities in the northern Iberian Peninsula. The first case involved the abandonment of the graveyard and the creation of a new village and the second shows the disuse of the graveyard due to ecclesiastical reorganization.

Chapter II describes Las Gobas site (Burgos, northern Spain), which consists of a rock-hewn dwellings settlement and adjoining farmsteads that were in use between the 6th and 12th centuries. Isotope composition gives insight into different socio-economic aspects of the rural medieval population. Carbon and nitrogen stable isotopes provide insights into dietary habits whereas strontium isotope provides information about residential movements. The formation of the village involved mobility of individuals, who in Las Gobas were mainly females probably because of patrilocal marriages. As regards dietary patterns, temporal dietary differences have been observed at the time the nearby Laño village was formed, but not according to sex nor to age.

In Chapter III, San Juan de Momoitio graveyard (Biscay, North Iberian Peninsula) isotope results are discussed. The graveyard was gradually abandoned around the 12th century due to the ecclesiastical reorganization. This study again shows temporal dietary changes towards a less diversified diet in the last period. The unusually high number of infants in Momoitio graveyard is noteworthy and suggests high infant mortality that can be attributed to the weaning practice. Contrary to Las Gobas site, San Juan de Momoitio does not show great mobility of individuals since only a few individuals were of non-local origin, but once again the region of origin cannot be established.

At the same time, in the 8th century Muslims conquered the main towns of the Iberian Peninsula delimiting their territory from the Christian Kingdoms by the Upper March that was centred in Saragossa. Within the Muslim territory, the Banu Qasi dynasty ruled a semi-autonomous territory for nearly two centuries until the late 9th century, when the Cordova emir recovered most of the Banu Qasi territories.

In this context, the Muslim population of Tauste (Zaragoza) has been studied in Chapters IV and V. The necropolis of Tauste constitutes a suitable site to examine human mobility since it was located on the northern frontier during a very convulsive period. Tauste was placed midway between the two most significant cities: Saragossa, (metropolis of the Upper March) and Tudela (center of the Banu Qasi territory).

Chapter IV presents a classic isotope analysis to reconstruct palaeomobility and palaeodiet patterns of the medieval Muslim population. Although Tauste was located on the northern al-Andalus frontier most individuals were of local origin and only three females and two males were non-locals. Establishing the provenance of incoming individuals is difficult but according to the oxygen isotope composition, males would come from a warmer region while females would come from a more mountainous geographic area. As regards the medieval Muslim diet, isotope results illustrate not only differences in diet according to sex and age but also the environmental conditions. Differences by sex and age indicate different diets related to the sexual division of labour since Muslim female work was restricted to the household. Moreover freshwater fish intake and the manuring effect have also been observed.

As complementary to the isotope diet study, Chapter V illustrates the chemical composition analysis of teeth by laser ablation-inductively coupled plasma-mass spectrometry (LA-ICP-MS). Biofunctional elements have been taken into consideration to monitor paleodiet patterns since the concentration of these elements is related to food products and therefore human dietary intake. The chemical composition variations confirm the isotope results, with differences in the food intake according to sex and age. Moreover adults enjoyed a larger consumption of animal proteins, and males more than females, which is attributed to the sexual division of labour or to the social status. LA-ICP-MS analysis can be used not only for palaeonutrition studies but also to provide information about health. In fact, five of the Tauste individuals display lead intoxication, suggesting work-related poisoning.

In conclusion, dietary patterns either obtained by isotopes or by geochemistry illustrate consumption habits both in medieval rural societies and in villages. The studied cases of peasants (i.e. Las Gobas and San Juan de Momoitio) show temporal dietary variations but no significant differences according to the sex or age of individuals. On the contrary, in the Muslim population of Tauste, differences in diet have been observed according to the sex and age of individuals. Additionally, evidence of weaning practice, freshwater fish intake, manuring practice and work-related poisoning has also been observed.

Regarding individuals' mobility during the Early Middle Ages, the data suggest few people travelled. Female mobility was subject to the avoidance of inbreeding marriages, so women moved for patrilocal marriages. In contrast, males would move to acquire better economic opportunities and the possibility of improving their status, so males would move from farms to a village.

Methodology

The study was carried out by measuring the different stable isotopes composition and punctual chemical analysis in the skeleton of the human individuals from the different archaeological sites. Strontium and oxygen isotopes were used for the characterization of residential mobility of the individuals whereas the carbon and nitrogen isotopes and trace elements were used for the characterization of the palaeodiet habits within the population.

The selection of samples was performed before the analyses. If possible, tooth enamel was collected to research mobility while bones were selected for the diet.

For mobility studies, individual's dental enamel is preferred because it maintains the composition of the tooth since it formed during childhood. Thus, strontium and oxygen isotope ratios will show the composition in the place of origin when the individual was a child. On the other hand, dietary studies were performed in bone collagen. The selected materials were, if possible, ribs and long bone fragments.

Besides, the local baseline must be established to reconstruct both mobility and the diet. Water, coeval or modern fauna, and soil samples from the surrounding area of the graveyard were considered to define local bioavailable strontium isotopic composition. Since water and soil radiogenic strontium isotope values are influenced by bedrock lithology, in archaeology a common procedure to establish the local isotope composition is to analyze local archaeofauna (Price et al., 2002; Slovak and Paytan, 2011). Archaeological fauna samples fed locally in the same area as the humans (Price et al., 2002; Bentley, 2006), resulting indicative of local isotope signature. However, the use of livestock as indicative of the local baseline has been subjected to debate since domestic animals can undergo the same mobility patterns as humans (Shaw et al., 2009; Knudson et al., 2012). The materials used to define the local strontium baseline in each of the archaeological sites studied are specified in the corresponding chapter.

Besides, faunal bone samples were analysed to establish the carbon and nitrogen isotope baseline. Depending on the studied case, palaeofauna or modern fauna was taken into consideration. Nevertheless, establishing the local isotope composition baseline can be problematic because of palaeofauna absence (i.e. Taute site since the Islamic burial ritual forbade any objects being buried with the body). On the order hand, when domestic palaeofauna are considered to determine the local baseline, herding or feeding practices, or browsing or grazing in different habitats with different isotopic baselines (Oelze et al. 2011) may introduce varying baseline isotopic signatures. Furthermore, present-day local fauna must be discarded because livestock are fed with non-local resources and the isotopic signal will not correspond to local plant resources. Pasture-fed livestock will also exhibit nitrogen isotope depletion due to the widespread use of mineral fertilizers (Bol et al., 2005; White, 2013). In addition to the fodder and fertilizer effect, livestock trade and transhumance are other factors affecting isotope composition.

Sample selection

Human remains sampling

This study deals with archaeological skeletal material and all necessary permits were obtained for the described study, which complied with all relevant regulations. Sampling has been conducted to a high ethical standard according to the principles expressed in the Declaration of Helsinki (Fig.1). The bones and teeth samples were transferred to the University of Basque Country-UPV/EHU for investigation. At present all archaeological human remains recovered at the sites are stored in the corresponding museums, Tauste at the Museo de Zaragoza, Alegria-Dulantzi and Las Gobas at the Museo de Arqueología de Alava and San Juan de Momoitio at the Museo de Arqueología de Bizkaia.

Although all the analytic techniques applied in this study involve destructive sample preparation procedures, sampling was performed causing the least possible damage to the specimens to obtain the minimum sample amount required for the analytic procedures.

Bones were sampled to extract the collagen used to perform isotopic analyses for dietary studies. Bone fragments were selected if possible, but for complete bones a small section was cut for sampling. Ribs were the preferred bone because they are the most abundant bones; they do not contribute relevantly to anthropological studies and are usually already fragmented. In the absence of ribs, long bones or the skull were sampled. To guarantee the adequate amount of collagen, the bone amount necessary is about 5 cm length of bone.

For mobility investigation, two teeth from each individual were collected to extract a large enough sample for the analysis of the strontium isotopic composition while for LA-ICP-MS chemical analysis a complete tooth per individual was chosen.

Figure 1. Selection of human samples has been conducted to a high ethical standard according to the principles expressed in the Declaration of Helsinki.

Lower molars are preferred because they represent the first years of life of the individual and are the best teeth to study individuals' change of residence. Moreover, molars are more resistant to post depositional alteration and have a larger sample amount. In the case of unavailability of molars, premolars, canines or incisors were used. Being aware of the value of ancient human teeth, loose teeth were selected whenever possible.

When there is no tooth available, jaw and/or maxilla are sampled always trying to damage the skeletal element as little as possible.

Fauna remains sampling

Archaeological fauna samples (wild and/or domestic) are used to establish the local isotope baseline. When archaeological fauna are not available, modern fauna in the area surrounding the graveyard are collected. Similar to human remains, teeth and large bones were selected. The fauna sampling adhered to the same requirements as in humans sampling.

Soil and water sampling

Soil and water samples are also used to determining the local isotope baseline. Previous to water sampling, the geology in the surrounding area was studied since isotope composition is related to geology and varies according to the composition and age of bedrock. 250 ml of surface water were collected from the banks of the rivers near to the site (Fig.2) and the number of water samples at each site depended on the heterogeneity of the geology in the area around the graveyard. Soil samples were collected in different parts of the studied graveyards.

Figure 2. Collecting freshwater samples at the riverbank.

Sample preparation

Human and fauna bones and teeth samples were washed in an ultrasonic bath to remove impurities and further cleaned by mechanical abrasion to remove the outer surface and avoid potential contamination. The samples were washed ultrasonically for 30 min in distilled water and rinsed in ultrapure water. This procedure was repeated the same times for each sample to remove all impurities. When bones were larger than necessary, previously to cleaning the bone, the samples were cut with a diamond-cutting disc.

Possible burial modification of the isotopic signal must be evaluated prior to isotope analysis. To establish diagenetic alteration and the presence of secondary minerals (calcite, oxides and others), Fourier transform infrared spectroscopy (FTIR) was performed.

For the FTIR analysis, previously washed and powdered bone (1 mg) was mixed with 100mg of potassium bromide (Aldrich 22186-4, FT-IR grade) previously dried at 100 °C. The infrared spectra are in the 400-4000 cm^{1} range, with a resolution of 4 cm^{1} and an accumulation of 40 scans using an FTIR- 8400S Shimadzu spectrometer. The crystallite size was determined by calculating the crystallinity index (IC) as IC ¼ (A605 þ A565)/(A595), where Ax is the absorbency at wavelength x (Shemesh, 1990) assuming a straight baseline between 700 and 500 cm^{1}. This index is correlated with the crystallite size of the biogenic apatite, which is indicative of the degree of crystal rearrangement and therefore of diagenesis. Apatites with larger and more ordered crystals show a greater separation of these peaks and a higher CI index (Shemesh, 1990; Wright and Schwarcz, 1996; Greene et al., 2004).

Sample preparation for isotope analyses

Carbon and nitrogen isotopes

For carbon and nitrogen isotope analyses, human and faunal bone collagen was extracted following the procedure in Bocherens et al. (1991). Previously washed and powdered bone samples (300 mg) were demineralised in 1M HCl for 20 min at room temperature until the sample dissolved. The samples were rinsed with distilled water and treated with 0.125 M NaOH to remove humic acid. The resulting insoluble fraction, after being rinsed again with distilled water, was gelatinized in HCl solution at pH3 for 17 h at 90ºC. Then, samples were filtered with disposable syringe filters (5 µm), freeze-dried and finally lyophilized. Isotopic analysis was performed on 2.5-3.5 mg lyophilized collagens enclosed in tin capsules.

Carbon and nitrogen isotope analyses were performed using an elemental analyzer on line with a continuous-flow isotope ratio mass spectrometer (EA-IRMS) at Iso-Analytical (Cheshire, UK). Replicate measurements of the liver standard NBS-1577B and ammonium sulphate IA-R045 working standard were run to confirm instrument accuracy. Isotopic values are reported as δ values in per thousand (‰) relative to international defined standards for carbon (VPDB: Vienna Pee Dee Belemnite) and nitrogen (AIR: Ambient Inhalable Reservoir). The instrumental precision for $\delta^{13}C$ was ± 0.06‰ or better and for $\delta^{15}N$ was between ± 0.06‰ and ± 0.08‰, determined by replicated analyses of internal standards.

Strontium isotopes

For strontium isotope analysis, a small fraction of human and faunal dental enamel was collected mechanically with a diamond-coated trepanation drill (MF-perfect, W & H Dentalwork, Bürmoos, Austria) (Fig.3). The enamel sample was taken transversally. Enamel and bone samples (~10 mg) were dissolved in 7 ml Savillex® vials (Minnetonka, MN, USA) with 1.5 ml of 2N HNO_3 (analytical grade purified by subboiling distillation).

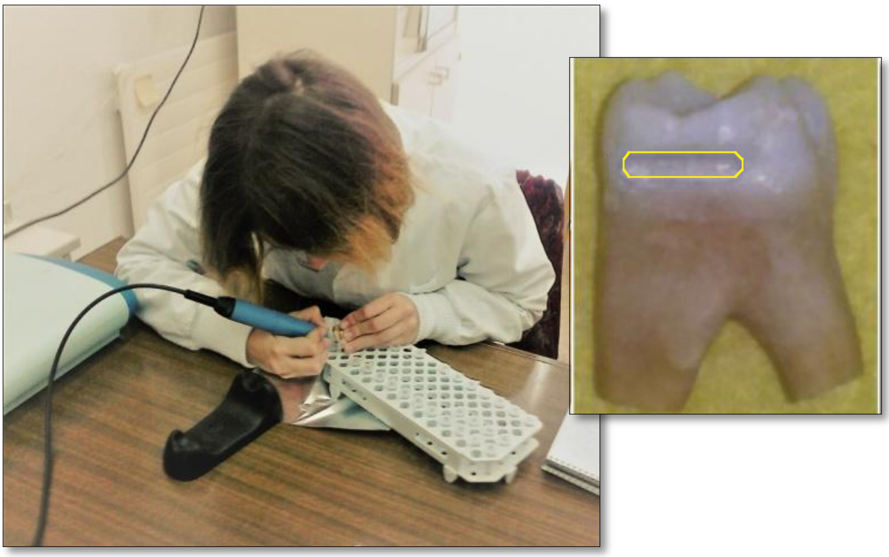

Figure 3. A small fraction of human and faunal dental enamel was collected mechanically with a diamond-coated trepanation drill.

Water samples were filtered with disposable syringe filters (0.45 um) to remove suspended particles. 15 ml of freshwater was evaporated to dryness and then dissolved in 2 ml HNO_3.

A 1g aliquot soil sample was leached by adding 2.5 ml 1 M ammonium nitrate (NH_4NO_3) and shaking for 8 h to obtained the bioavailable Sr. After samples were centrifuged at 3000 rpm for 15 min, the supernatant was extracted (~1-2 ml) and evaporated to dryness and then redissolved in 2 ml HNO_3.

The solutions were loaded into cation exchange columns filled with Sr.spec® (ElChroM industries, Dariel, IL, USA), a strontium selective resin (Fig.4). The resin was used once to elute the sample and then discarded. Strontium procedural blanks were less than 100 pg and hence provided a negligible contribution.

Figure 4. The solutions were loaded into cation exchange columns filled with Sr.spec® (ElChroM industries, Dariel, IL, USA), a strontium selective resin.

The radiogenic strontium isotope samples were analyzed on a Neptune multi-collector inductively coupled plasma mass spectrometer (MC-ICP-MS) and Finnigan MAT 262 thermal ionization mass spectrometer at the Advanced Research Facilities (SGIker) of the

University of the Basque Country (UPV/EHU) (Fig.5). Multiple samples of the strontium standard NBS-987 were run to confirm instrument accuracy. Repeated analyses were at the NIST SRM-987 international standard. Moreover, $^{87}Sr/^{86}Sr$ by MC-ICP-MS measurements were corrected for krypton (Kr) and rubidium (Rb) interferences.

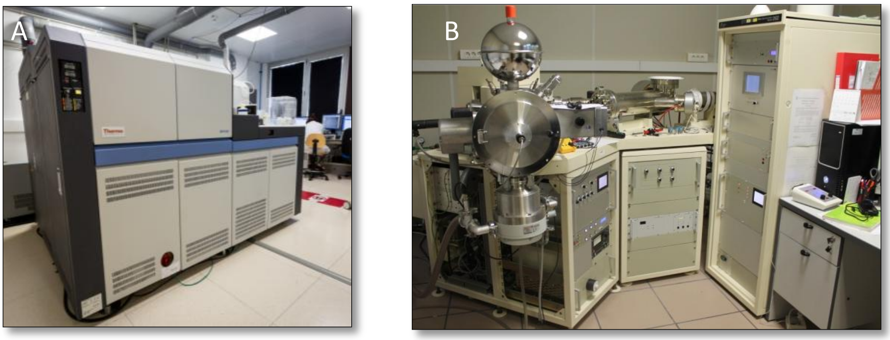

Figure 5. A. Neptune multi-collector inductively coupled plasma mass spectrometer (MC-ICP-MS) and B. Finnigan MAT 262 thermal ionization mass spectrometer at the Advanced Research Facilities (SGIker) of the University of the Basque Country (UPV/EHU).

Oxygen isotopes

Oxygen isotope sample preparation was performed following the procedure described in Stephan (2000). 60 mg of dental enamel powder was processed. The organic matter was removed with a solution of 2.5% NaOCl for 24 h at room temperature followed by a 48 h treatment in 0.125M NaOH at room temperature. The hydroxyapatite powder free of organic matter was dissolved in 2 ml of HF for 24 h. The phosphate solution and the residue composed of CaF_2 were separated by centrifugation, pipetted into a 100 ml glass tube and neutralized with 3 ml 2M KOH. Silver phosphate (Ag_3PO_4) was precipitated by adding 30 ml of a buffered silver amide solution (0.2 M $AgNO_3$; 1.16 M NH_4NO_3; 0.75 M NH_4OH) gradually warmed to 70ºC, holding the temperature for 5-6 h and cooling down slowly. Silver phosphate crystals (Fig.6) were filtered on a weighed 0.2µm filter and washed several times with double distilled water, then dried at 50ºC for 1-2 h.

To measure oxygen isotopes, 0.3 mg of Ag_3PO_4 was mixed with 0.5-1 mg of AgCl and 0.3 mg of graphite in silver capsules. These silver capsules were transferred into the autosampler carousel of the Temperature Conversion Elemental Analyser (TCEA) and degassed for 30 minutes at 80°C in a vacuum. The oxygen isotope analyses were performed on a Thermo Finnigan TCEA coupled to a Delta Plus XP Spectrometer at the University of Parma (Fig.7). Isotopic compositions were given in the conventional δ-notation relative to V-SMOW (Vienna-Standard Mean Ocean Water). Normalization to the V-SMOW scale was based on four replicated international reference materials provided by the International Atomic Energy Agency (IAEA): IAEA-601, IAEA-602, IAEA-CH6, and IAEA-SO-6. The analytical precision of a single determination was better than ±0.4‰.

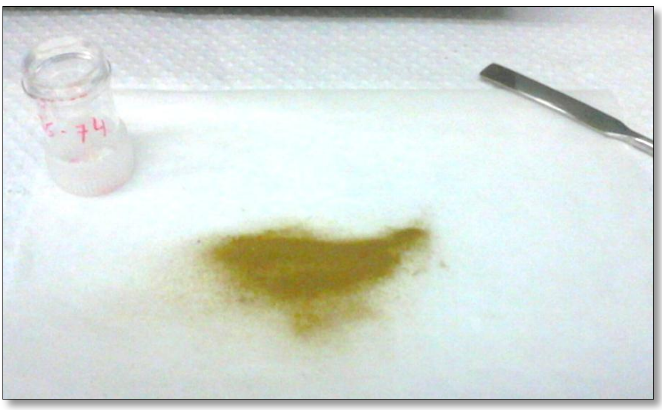

Figure 6. Aspect of silver phosphates precipitation.

Figure 7. Thermo Finnigan TCEA coupled to a Delta Plus XP Spectrometer at the University of Parma (give up by Paola Iacumin and Enrico Maria Selmo, Universitá degli Studi di Parma).

Punctual chemical analysis by LA-ICP-MS sample preparation

LA-ICP-MS technique was used to determine the chemical composition of teeth. This technique enables direct analysis of solid samples using only a small amount of material and minimal sample destruction.

Before analyses, tooth enamel and dentine structure samples were examined using a scanning electron microscope (JEOL JSM-6400) fitted with a backscatter detector operating at 15 kV. Specimens were sputter-coated with a thin layer of carbon. These observations were performed in the Materials and Surface Unit of the Advanced Research Facilities (SGIker) in the Basque Country University (UPV/EHU). Analyses were performed in both dentine and tooth enamel. Elemental composition of teeth varies in concentration of ppm or µg/g. Spot ablation experiments consisted of spots arranged in line-scanning, covering the enamel and dentine (Fig. 8).

Figure 8. Microphotograph of transverse tooth section showing LA-ICP-MS laser spots. Black dots indicate laser ablation spots in the enamel and dentine.

Analyses were carried out using quadrupole-inductively coupled plasma-mass spectrometer (Q-ICP-MS) model Thermo X7 updated to XSeries2 coupled to the UP213 laser ablation system equipped with Xt interface unit (Fig.9). To improve equipment sensitivity, a second vacuum pump was used at the interface of the system. The laser ablation system was New Wave Nd:YAG operating at a wavelength of 213 nm. Calibration of LA-ICP-MS was achieved using a standard reference glass material NIST SRM 612 (Birbaum et al., 2011: Günther et al., 2001; Herwartz et al., 2013; Kowal-Linka et al., 2014; Kowal-Linka et al., 2015). Measurements were taken for the spot size of 100 µm at a distance of

300 µm from each other. Time delay between the end of LA of one spot and the initiation of LA of the next spot was 15s. LA was performed with a laser spot diameter of 100 µm, laser fluency 4.5 J/cm^2, and a repetition rate of 10 Hz.

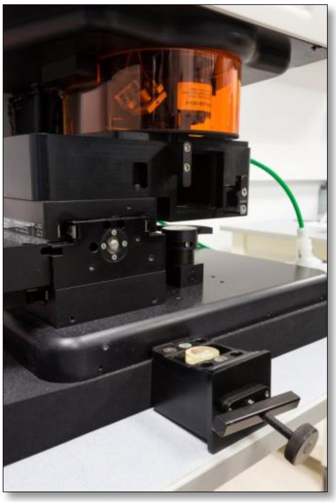

Figure 9. The laser ablation system showing the tooth sample placement.

The analysed elements were Na, Mg, Al, Sc, Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, Rb, Sr, Y, Ba, Pb, Th, U and the rare earth elements (La-Lu). Data collection was performed by rapid peak-hopping (5-30 ms) dwell time between selected isotopes of each analyte element for a period of 60 s. A background signal was collected during the first 30 s of analysis. The laser was fired for 60 s from which the middle 30 s were used for signal integration. For data reduction, Iolite 3 data processing software package for time resolved mass spectrometry data was used (Paton et al., 2011: Paul et al., 2012). The background from the “gas blank” is used to background subtract the gross analyte data. The concentration of element, even though calculated directly with the Iolite 3 software, can also be obtained by defining the intensity integration point for the background and the sample analysed by laser. ^{43}Ca was used as an internal standard to account for variation in ablation efficiency, which is caused by variations in the mass of the material ablated. The internal standard was used to correct for the multiplicative effect of matrix and more importantly the different volume of teeth tissues ablated. Calcium concentration was assumed from the stoichiometry of biogenic hydroxyapatite. The estimated calcium content is 252mg/g for the dentine and 360mg/g for the enamel tissues (Kohn et al., 1999). The mean and standard deviation of repeat analyses of a matrix matched (Durango apatite), in-house standard indicated that analytical reproducibility was 2-6% (Table 2). The accuracy for the analysis was further indicated by the close agreement between results for the Durango apatite and determinations made by ICPMS (typically <5-10%). The analytical accuracy is similar to that reported by Trotter and Eggins (2006).

Detection limits (LOD) for each element and spot was determined based on Longerich et al. (1996). The equation used to calculate these detection limits was the following:

$$LOD = \frac{3\sigma_{BCG}}{SxY} \; x \; \sqrt{\frac{1}{N_{BCG}} + \frac{1}{N_{PK}}}$$

where σ_{BCG} is the standard deviation of replicate analysis of the pre-ablation background identification; N_{BCG} and N_{PK} are the number of replicate determinations used for background and peak signal integration, respectively; S is the normalized sensitivity in cps per unit of concentration for the reference material; Y is the ablation yield relative to the reference material, determined from the counting intensity measurement and the known concentration of the integral standard.

Statistical analysis

Statistical tests were performed using SPSS for windows version 20 (Statistical package for Social Sciences). Both parametric and non-parametric statistics were used to describe isotope distribution and compare carbon and nitrogen isotope values between groups. Thus, differences between sample groups were analysed by applying an unpaired Student’s t-test or the two-tailed Mann-Whitney U test depending on the shape of the population distribution. The latter was selected over the t-test because of small sample sizes, important differences in sample size between groups and some heterogeneity between variances. The null hypothesis states that there is no difference between the ranks of two samples. A probability level of 5% was considered significant to reject the null hypothesis. Statistical significance was accepted as $p < 0.05$.

Besides, parametric statistics were also used to describe oxygen and strontium isotope distribution and compare isotope values between sample groups. To identify outliers, boundaries of intra-sample variation based on two measurements of scales were defined: ± 2 standard deviation (2SD) from the mean and Tuke’s inter-quartile range method (IQR) considering 1.5xIQR and 3xIQR (Lightfood and O’Connell, 2016).

The correlation matrix of dental tissue chemical composition obtained by LA-ICP-MS was performed in order to distinguish differences in the tooth contents in the trace element. In addition to the multivariate analysis, principal component analysis (PCA) was used for discrimination of whole tooth samples and for the discrimination of the different tooth tissues separately.

Chapter I:

Dulantzi site

"Strontium isotopes of human remains from the San Martín de Dulantzi graveyard (Alegría-Dulantzi, Álava) and population mobility in the Early Middle Ages"

Quaternary International 2013, 303: 54-63

Abstract

Strontium isotope analysis of human remains from San Martín de Dulantzi (Alegría-Dulantzi, Alava, Spain) graveyard has been used to establish mobility patterns during the Early Middle Ages. Some archaeological human remains had Germanic grave goods. Through radiogenic strontium isotope analysis, local origin individuals and immigrants were differentiated. Archaeological human bone samples exhibit $^{87}Sr/^{86}Sr$ = 0.70779-0.70802 values similar to domestic fauna isotope composition, indicating local origin of individuals or long residence time in the region. Comparing these data with tooth enamel values, two groups of immigrants from distinctive geological environment were established. The Dulantzi population constituted mainly a local society with influxes of immigrants. The foreign individuals are distributed through the studied period of time, suggesting that migration movements were limited in number. Isotopic signatures indicating mainly local individuals, linked to grave goods with archaeological attribution to Germanic origin, question the previous ethnic paradigm

Introduction

The study of the role of German migrations during the disintegration of the Roman Empire and the transformation of Roman society during the Early Medieval period has seen renewed interest in recent decades, because the implementation and generalization of new analytical techniques has transformed interpretative frameworks. In recent years, revisions carried out by some scholars downplayed the impact of the Germanic migrations, even extending to denying them, whereas some central European researchers had revisited the ethnogenesis paradigm and redefined the historic events in terms of systemic transformation processes (Pöhl, 1998; Gillett, 2006; Castellanos, 2007; Hakenbeck, 2008; Heather et al., 2010; Hakenbeck, 2011; James, 2011). However, in the archeology of southern Europe the disruptive approach that gives a remarkable role to Germanic migrations prevails (Valenti, 2009). This explains the effect of cultural historicism which defined ethnic groups as historical subjects identifiable based on cultural material, thereby allowing the definition of migration flows due to the spatial distribution of the diagnostic objects. Therefore, the grave goods found in cemeteries and other markers, such as architectures and wares, allow identification of immigrants (Brogiolo and Chavarria Arnau, 2008).

In recent years, several research projects have been started up across Europe, seeking to verify the real impact of migration and the historical role of Germanic elites by means of strontium isotope analysis of archaeological human remains, in particular those individuals buried with Germanic grave goods. However, these studies do not assist in the cases of individuals buried without

Germanic grave goods, a situation corresponding to most burials even in so-called “Germanic cemeteries” (Quirós Castillo and Vigil-Escalera, 2011). Therefore the study of rural communities with “Germanic cemeteries” in a broad time frame will allow establishment of mobility patterns and detect the presence or absence of mass migrations in the Middle Ages.

Strontium isotope studies to decipher human migration have become common in recent years. Human remains from a wide variety of other times and places have been studied, demonstrating the usefulness of this method in archaeology. These studies correspond to archaeological sites from the Anasazi in Arizona (Price et al., 1994b; Ezzo et al., 1997; Ezzo and Price, 2002), the late Stone Age and the historical period of South Africa (Sealy et al.,1991, 1995; Sealy, 2006), the Neolithic in southern Germany (Price et al., 1994a, 1998; Grupe et al., 1997; Grupe et al., 1999; Price et al., 2001; Bentley, 2003; Bentley et al., 2003, 2004; Bentley and Knipper, 2005), the Mexican Classical period (Price et al., 2000), the pre-Hispanic period in the Central Andes of South America (Knudson et al., 2004, 2005; Knudson, 2008; Knudson and Torres- Rouff, 2009; Knudson et al., 2009), the Scandinavian colonies in north-western Scotland and Ireland (Montgomery et al., 2003, 2005; Knudson et al., 2012), Roman movements in England (Eckardt et al., 2009; Chenery et al., 2010, 2011; Mueldner et al., 2011), and the Scandinavian countries (Frei et al., 2009; Frei and Price, 2012).

Strontium is present in variable concentrations in the bedrock, in groundwater, in the soil, in plants, and in the animals of a given ecosystem. Although the strontium concentration in organisms varies in accordance with their trophic level, the strontium isotope composition in living beings is insensitive to this phenomenon (Burton, 1996; Hoppe et al., 1999; Blum et al., 2000; Balter et al., 2001, 2002; Hoppe et al., 2003; Balter, 2004; Faure and Mensing, 2005; Hoppe and Koch, 2007). In contrast, the $^{87}Sr/^{86}Sr$ isotopic ratio of radiogenic strontium of bedrock, soil, water, plants and animals is variable. The strontium isotope ratio varies as a function of the age and the chemical composition of the bedrock (Dickin, 2005; Faure and Mensing, 2005).

Radiogenic ^{87}Sr is formed by the radioactive decay of rubidium and the amount of rubidium in a rock and its age determines the amount of ^{87}Sr in the bedrock, and therefore the $^{87}Sr/^{86}Sr$ isotope ratio (Faure and Powell, 1972). The $^{87}Sr/^{86}Sr$ isotope ratio in plants, animals, groundwater of a given region, and the humans that consume these resources reflect the bedrock $^{87}Sr/^{86}Sr$ values (Price et al., 2002; Bentley, 2006; Malainey, 2011) as there is not fractionation related to biological processes (Graustein, 1989; Capo et al., 1998; Blum et al., 2000). Strontium substitutes for calcium in the hydroxyapatite of bones and teeth. The $^{87}Sr/^{86}Sr$ isotope ratio in tooth enamel and human bones reflects the isotope ratios of strontium in the food and water ingested during the individual's lifespan (Ericson, 1985, 1989; Price et al., 1994a, 1994b). Individuals who have consumed local foods and have lived in a specific geological region will have $^{87}Sr/^{86}Sr$ isotope ratios in their teeth and skeleton reflecting the area where they lived. In addition, dental enamel, in contrast to bones, does not incorporate other elements after its formation in infancy (Hillson, 1986, 1996) and the $^{87}Sr/^{86}Sr$ ratios in tooth enamel reflect the composition of the place of residence during childhood. In contrast, bone continually regenerates and incorporates strontium (Parfitt, 1983; Smith, 1991) and the $^{87}Sr/^{86}Sr$ ratios reflect the composition of the place of residence for the last few years of life. The aim of this study is to analyse the migration patterns in a rural community during Early Middle Ages (6th-10th centuries) in the northern Iberian Peninsula based on the strontium isotope composition, and recognize the presence of immigrants. This study has been performed in San Martin de Dulantzi site, a graveyard with both individuals buried without associated grave goods and individuals with grave goods (weapon, wares, personal ornaments, hobnails).

The San Martín de Dulantzi site

The Alegría-Dulantzi site is located in the province of Álava (northern Spain). The town was founded by Alfonso XI in the year 1337 in the village of Dulantzi, which has been documented since the 11th century. It is located near Tullonium, one of the localities of the Varduli mentioned by Ptolemy and cited in the Antonine Itinerary (Gurruchaga, 1951) (Fig. 1). The site was located on the Iter 34 ab Asturica Burdigalam, an important Roman communications road that currently corresponds to the Lisbon-Madrid-Irún-Bordeaux main road.

Excavations in 2009 and 2010 in the area of the temple of San Martín de Dulantzi had identified occupation from prehistoric times to the Middle Ages (Fig. 2). However, the greatest funerary occupation corresponds to between the 6th and 12th centuries, associated with a church and a few household structures (Loza Uriarte and Niso Lorenzo, 2011; Loza Uriarte and Niso Lorenzo, 2012). After two phases of occupation in the Bronze Age and Late Antiquity, in the 5th century, the entire area was converted to a burial sector (Phase 3). Around the 6th century, a new building was built, which has been interpreted as a private church with baptistery and with a foundation burial (Phase 4a) which included a privileged cemetery until the 7th century (Phase 4b). Nineteen graves located both inside the church and in the space between the church and the baptistery have been attributed to this phase. In 9 of these burials, grave goods including weapons, pottery and personal ornaments have been found. These resemble grave goods from other Basque Country sites, and exhibit more similarities to archaeological sites from Gaul than to those from the Iberian Peninsula. Towards 700 AD, occupation became denser, which led to an expansion of the necropolis outside the temple where the burials had no associated grave goods (Phase 5). During the 10th to the 12th centuries (Phase 6), large silos were built inside the temple and it continued to be used for funerary purposes. Individuals studied here correspond to Phases 3, 4, and 5, dating from the 5th to 10th centuries (Table 1, Fig. 3).

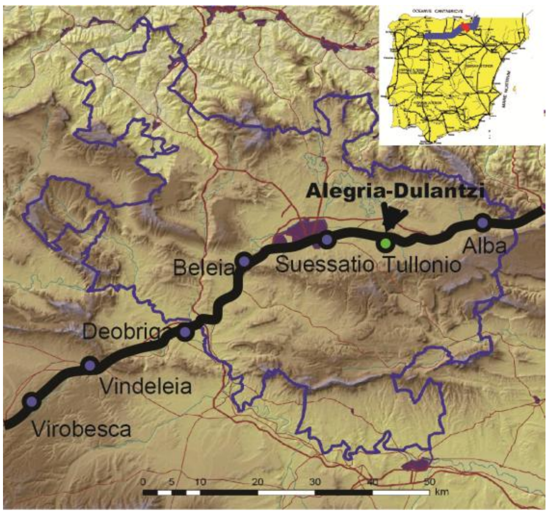

Figure 1. Geographic location of the site showing the route of Iter 34 ab Asturica Burdigalam in the province of Alava and the different stationes. The inset map illustrates the Roman roadways (Solana Sáinz and Sagredo San Eustaquio, 2006).

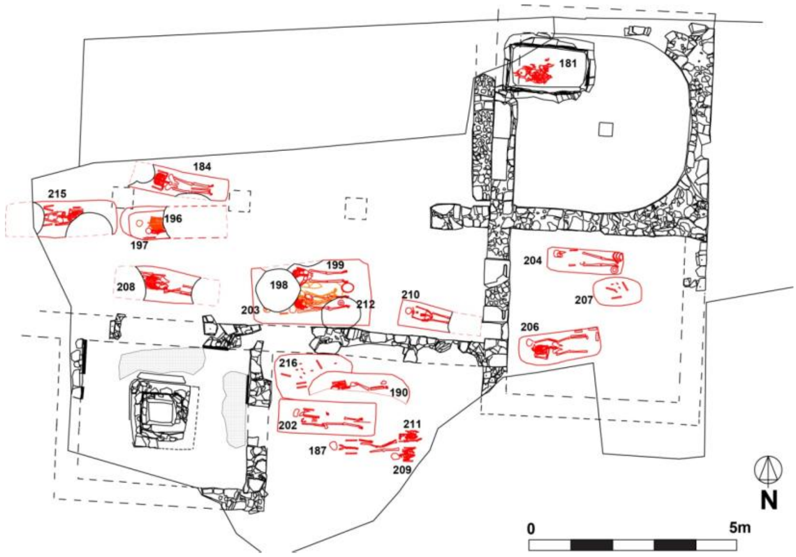

Figure 2. Map of San Martin de Dulantzi church and the necropolis with the samples location.

The dating of the occupation sequence has been achieved from the stratigraphical relationships, from the study of archaeological materials and from 19 radiocarbon dates of human remains (long bones) recovered in the necropolis. The measurements were performed using conventional 14C counting at the Institute of Physical Chemistry Rocasolano, CSIC-Madrid. Twelve individuals had been studied also by strontium isotope analysis.

Table 1. Strontium isotope results for samples from Alegría-Dulantzi site.

Sample	Material	Tooth	Phase	14C date	Age	Sex	Grave goods	$^{87}Sr/^{86}Sr$
218	bone		3	1580±30	J	ind		0.707905 ± 12
181	dentine	M2	4a	1626±37	AJ	F		0.707948 ± 18
181	enamel	M2	4a	1626±37	AJ	F		0.708547 ± 9
184	bone		4b		AJ	M	Y	0.707793 ± 6
187	bone		4b		AJ	M	Y	0.707921 ± 12
190	bone		4b	1365±32	J	F	Y	0.707901 ± 8
196	rib		4b		A	F		0.707887 ± 10
197	dentine	M2	4b	1490±30	AJ	M	Y	0.707896 ± 12
197	enamel	M2	4b	1490±30	AJ	M	Y	0.707946 ± 25
198	bone		4b	1441±41	AJ	M		0.707892 ± 6
199	bone		4b		AJ	M	Y	0.707889 ± 11
202	bone		4b		AJ	M		0.707918 ± 7
203	bone		4b		A	M	Y	0.707873 ± 7
204	dentine	M2	4b	1417±35	A	M	Y	0.708068 ± 18
204	enamel	M2	4b	1417±35	A	M	Y	0.708509 ± 16
206	dentine	M2	4b		A	M		0.707902 ± 11
206	enamel	M2	4b		A	M		0.708008 ± 22
207	bone		4b		I	Ind		0.708014 ± 15
208	bone		4b		AJ	M		0.707858 ± 6
209	rib		4b		A	M		0.708019 ± 6
210	dentine	M3	4b		AJ	M		0.707975 ± 9
210	enamel	M3	4b		AJ	M		0.709157 ± 26
211	dentine	M1	4b		AJ	Ind		0.708199 ± 14
211	enamel	M1	4b		AJ	Ind		0.709196 ± 25
212	bone		4b	1520±30	A	M	Y	0.707882 ± 12
215	rib		4b		A	M		0.707915 ± 6
216	dentine	M3	4b		AJ	F		0.707966 ± 14
216	enamel	M3	4b		AJ	F		0.708061 ± 12
14	dentine	M3	5	1150±31	AJ	M		0.707978 ± 10
14	enamel	M3	5	1150±31	AJ	M		0.707925 ± 11
43	dentine	M2	5		A	M		0.708008 ± 10
43	enamel	M2	5		A	M		0.708053 ± 15
63	bone		5		AJ	F		0.707879 ± 6
72	dentine	M2	5	1237±31	AJ	F		0.708058 ± 15
72	enamel	M2	5	1237±31	AJ	F		0.708445 ± 21
83	bone		5	1342±36	AJ	F		0.707858 ± 6
87	bone		5		AJ	F		0.707846 ± 7
90	bone		5	1273±36	I	Ind		0.707870 ± 6
168	rib		5	1198±36	AJ	F		0.707901 ± 7
188	bone		5	1189±33	AJ	M		0.707922 ± 8
188	dentine	M2	5	1189±33	AJ	M		0.707970 ± 12
188	enamel	M2	5	1189±33	AJ	M		0.709129 ± 24
12	bone		6		AJ	M		0.707800 ± 7
81	bone		6		A	F		0.707818 ± 7
83-C	sediment							0.707753 ± 6
83-D	sediment							0.707790 ± 12
190-C	sediment							0.707724 ± 7
190-D	sediment							0.707714 ± 8
218-C	sediment							0.707718 ± 6
218-D	sediment							0.707735 ± 8
102-B	cow (bone)							0.707916 ± 11
161-BM	cow (bone)							0.707935 ± 8
161-BE	cow (enamel)							0.707936 ± 10

Table 1. Continued. Strontium isotope results for samples from Alegría-Dulantzi site.

Sample	Material	Tooth	Phase	14C date	Age	Sex	Grave goods	$^{87}Sr/^{86}Sr$
241-S	pig (enamel)							0.707889 ± 7
241-OV-T	sheep/goat (bone)							0.707878 ± 9
241-OV-P	sheep/goat (bone)							0.707898 ± 6
241-OV-M	sheep/goat (bone)							0.707890 ± 8
241-OV-E	sheep/goat (enamel)							0.707820 ± 8
241-SM	pig (enamel)							0.707865 ± 9
246-OV	sheep/goat (bone)							0.707829 ± 12
246-S	pig (enamel)							0.707916 ± 7

in-run precision (2se) given to the last significant digits

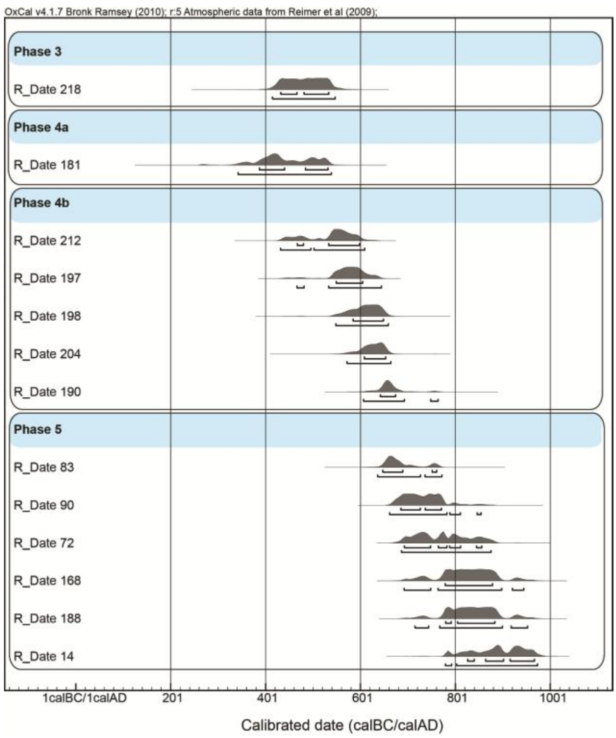

Figure 3. Calibrated radiocarbon dating of the burial attributed to Phases 3, 4a, 4b, and 5 (Quirós Castillo et al., in press) obtained with OxCal v4.1.7 (Bronk Ramsey, 2009) and IntCal09 atmospheric data (Reimer et al., 2009).

Materials

The studied samples correspond to 33 individuals (16 women, 10 men, and 7 undetermined). All of them are mature/senile individuals, with the exception of one child. Some individuals from Phase 4 possess grave goods including weapons, personal ornaments, and other items (Table 1). Eleven samples of domestic animals (pig, cow and sheep/goat) were included to determine the isotope composition of bioavailable strontium.

Analysed samples correspond to 22 long bone samples and 11 dental samples. Whenever possible, bones and teeth from the same individuals were sampled, but the conservation conditions of the individuals, sometimes cut or affected by subsequent occupations, have affected this sampling strategy. Two fractions were extracted from each tooth, one from the dental enamel and the other from the dentine. A micro-drill was used for the purpose (MF-Perfecta, W&H Dentalwerk, Bürmoos, Austria). The bone samples were cut with a diamond-cutting disc and carefully washed in an ultrasonic bath. The specimens were etched ultrasonically for 30 min in distilled water and rinsed in ultrapure water. This procedure was repeated four times for each sample.

Prior to analysis of sample isotope composition, possible burial modification of the isotopic signal must be evaluated. To establish diagenetic alteration and the presence of secondary minerals (calcite, oxides, and others), Fourier transform infrared spectroscopy (FTIR) was performed.

In order to determine the isotopic composition of the surrounding area, three sediment samples from the burial were also analysed. Two different leachates were performed to determine the most soluble $^{87}Sr/^{86}Sr$ isotopic ratio. These dissolutions with weak acids do not dissolve silicates or other refractory minerals, and allow determination of the composition of the Sr that can be ollected by plants and enter the trophic chain (Schweissing and Grupe, 2003). This bioavailable strontium is also the most mobile fraction during burial. For each sediment sample, two aliquots of 10 mg of powder were used. Samples were weighed on 15 ml PFA vials in an ultraclean room. The first aliquot was leached using 2 ml of 0.1 N acetic acid, and for the second aliquot 2 ml of 6 N acetic acid was added.

For the FTIR analysis, 1 mg of ground bonewasmixed with 100mg of potassium bromide (Aldrich 22186-4, FT-IR grade) previously dried at 100 ºC. The infrared spectra are in the 400-4000 cm^{-1} range, with a resolution of 4 cm^{-1} and an accumulation of 40 scans using an FTIR-8400S Shimadzu spectrometer. The crystallite sizewas determined by calculating the crystallinity index (IC) as IC ¼ (A605 + A565)/(A595), where Ax is the absorbency atwavelength x (Shemesh,1990) assuming a straight baseline between 700 and 500 cm^{-1}. This index is correlated with the crystallite size of the biogenic apatite, which is indicative of the degree of crystal rearrangement and therefore of diagenesis. Apatites with larger crystals andmore ordered showa greater separation of these peaks and a higher CI index (Shemesh, 1990; Wright and Schwarcz, 1996; Greene et al., 2004).

For thermal ionization mass spectrometry (TIMS), bone samples (5-10 mg) or dental samples (1-2 mg) were weighed in Savillex 15 ml PFA vials and dissolved with 1 ml HNO_3 2N (analytical grade purified by sub-boiling distillation). The solutions were loaded into cation-exchange columns filled with Sr.spec (EIChroM industries, Dariel, Illinois), a strontium-selective resin. The resin was used once to elute the sample and then discarded. The strontium procedural blanks provided a negligible (<100 pg) contribution. The Sr extracted was loaded on an Re filament with TaF following the method proposed by Birck (1986), and the isotope ratios were measured in a Finnigan MAT 262 thermal ionization mass spectrometer at the University of the Basque Country (UPV/ EHU, Spain). Multiple samples of the strontium standard NBS-987 were run to confirm instrument accuracy. External precision of analysis was ±0.00002 (2 sigma absolute) based on 206 analyses of NBS-987. Replicate analysis of the NBS 987 Sr standard during runs gave $^{87}Sr/^{86}Sr$ 0.710279 ±12 (2 s, n = 4).

An unpaired Student's t test was used to determine the statistical significance. Statistical significance was accepted as $p < 0.05$. Statistical analysis was performed with SPSS v.20 (Statistical package for Social Sciences).

Results and Discussion

The bone samples infrared spectra show the same characteristic absorption bands as the synthetic apatite with CO_3 2 in positions A and B (Bonel, 1972). All the FTIR spectra are characterized by wide H_2O bands that mask the OH band at 3567 cm^{-1} and the presence of three amide group bands: amide I at 1660 cm^{-1}, amide II at 1550 cm^{-1}, and amide III at 1236 cm^{-1}. Absorptions at 1035 cm^{-1} [PO_4 (v3)], 962 cm^{-1} [PO_4 (v1)], 605 cm^{-1} and 565 cm^{-1} [PO_4 (v4)], are typical of phosphate and, 1455 cm^{-1} and

1419 cm^{-1} [CO_3 (v3)], 871 cm^{-1} [CO_3 (v2)] are typical of carbonate in biological apatite (Fig. 4).

The crystallinity indices (CI) range from 2.18 to 2.89, with an average value of 2.47, a value lower than CI = 3.1 measured in modern human bone, indicating that the bone are not significantly recrystallized (Wright and Schwarcz, 1996; Keenleyside et al., 2009). The lack of a band at 713 cm^{-1} denotes the absence of authigenic calcite reinforcing the absence of diagenetic modification.

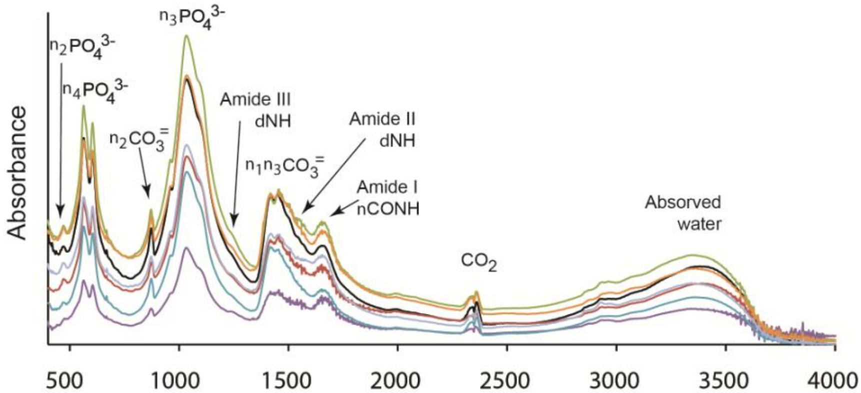

Figure 4. Fourier transform infrared (FTIR) spectra for selected bone samples from Alegría-Dulantzi. Peaks, bands assignments according Reyes-Gasga et al. (2008).

Mineral/matrix peak area ratios (A900-1200/A1585/1720) according to Boskey et al. (2003) are also used to evaluate incipient diagenetic processes. Mineral/matrix ratio ranges from 2.74 to 5.06, with an average value of 3.94, indicating large amounts of collagen in bones. The presence of absorbance bands of the amide II and amide III in most samples indicates a low grade of collagen deterioration, and absence of significant chemical changes in bone collagen. All these parameters indicate that the diagenetic processes in bones during burial had a negligible impact (Shemesh, 1990; Greene et al., 2004; Abdel-Maksoud and Abdel-Hady, 2011).

The results of the Sr isotope analysis for the different materials are shown in Table 1. Fig. 5 shows the composition intervals of long bones, dentine and enamel of human samples, of sediment samples and, of domestic animals (cow, pig and sheep/goat). $^{87}Sr/^{86}Sr$ isotope ratio of human long bones range from 0.707793 to 0.708019, whereas dentine samples have slightly more radiogenic $^{87}Sr/^{86}Sr$ isotope ratio values, ranging from 0.70789 to 0.708199. Dental enamel samples have a larger range of variation in $^{87}Sr/^{86}Sr$ isotope ratio ranging from 0.707925 to 0.709196. Domestic animals have similar $^{87}Sr/^{86}Sr$ isotope composition to human long bones, ranging from 0.707820 to 0.707936, whereas $^{87}Sr/^{86}Sr$ isotope ratio of sediments shows the less radiogenic values ranging from 0.707714 to 0.707790.

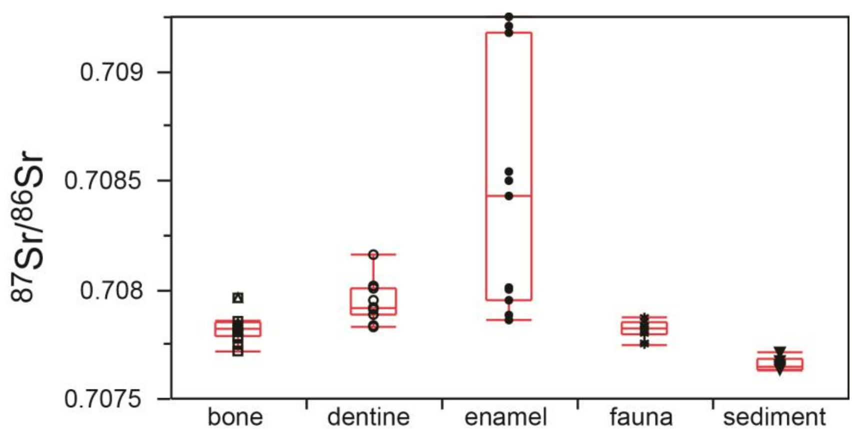

Figure 5. Box plot diagram showing the isotopic variation of the studied materials.

In order to use isotope analysis for reconstructing mobility, the range of values of local $^{87}Sr/^{86}Sr$ isotope ratio needs to be defined.

The geology of the archaeological site surrounding area consists mainly of Upper Cretaceous/Paleogene limestones and dolomitized limestones (Fig. 6). Typical strontium isotope values for the Cretaceous and Paleogene materials around Alegria-Dulantzi can be constrained between 0.70778 and 0.70795 (Gómez Alday et al., 2001; Baceta et al., 2013). The isotopic composition of both runoff and phreatic waters ranges from 0.707734 to 0.708012. Fernández de Ortega (2007) verified that waters become homogenized within a few kilometers and acquire the average isotope composition of carbonates. Therefore, the isotope ratio of local waters could be used as a reference to establish the signature of local strontium.

Usually in archaeology, a common procedure to establish the local isotope composition is the study of local fauna (Price et al., 2002; Slovak and Paytan, 2011). However, the use of domestic animals as indicative of the local composition has been a subject of debate since domestic animals can undergo the same migration patterns as humans (Shaw et al., 2009; Knudson et al., 2012).

However, isotope compositions of faunal remains are susceptible to be modified, similar to human remains, during burial.

FTIR studies indicate low to insignificant impact of diagenetic processes, both in collagen and mineral fraction of bones. To assess the presence of isotope equilibration between bones and sediment, several soil samples from San Martín de Dulantzi site graves were analysed. The isotope composition of sediments are significantly less radiogenic than humans ($t_{(16)}$ = -9.069, $p < 0.0001$) and domestic animals ($t_{(13)}$=- 9.049, $p<0.0001$), indicating the lack of significant diagenetic processes and the absence of bone/sediment isotope reequilibration during burial, reinforcing the infrared study.

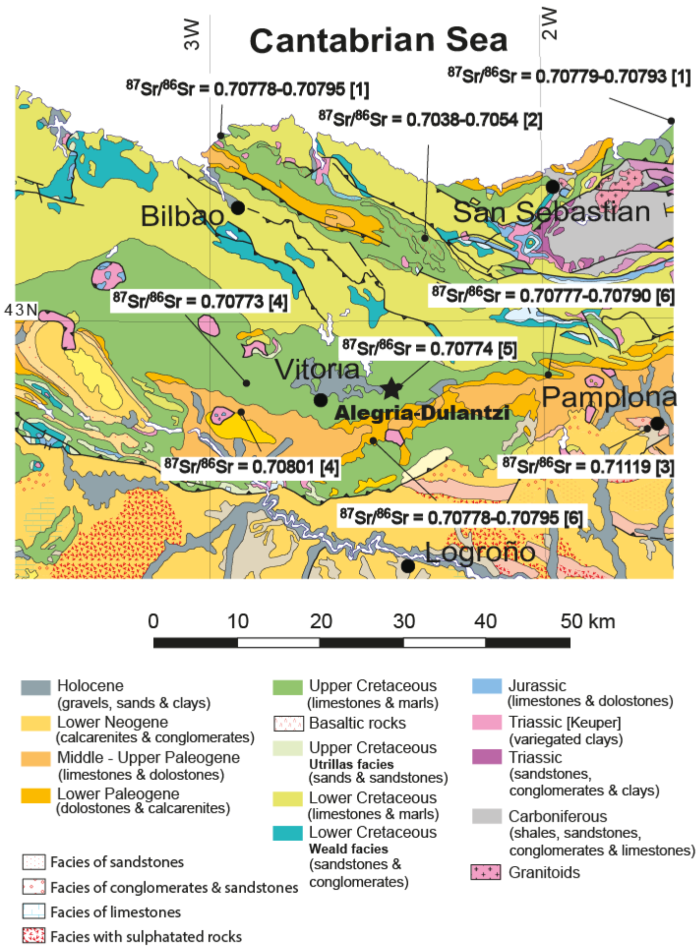

Figure 6. Geological Map of north Spain showing location of Alegria-Dulantzi with radiogenic Sr isotope ratios in bedrocks, soil and water. [1] Gómez Alday et al. (2001); [2] Rossy et al. (1992); [3] Prevedorou et al. (2010); [4] Fernández de Ortega (2007); [5] present study; [6] Baceta et al. (2013).

Human bones and domestic animals remains have similar $^{87}Sr/^{86}Sr$ isotope ratios, statistically indistinguishable ($t_{(27)}$=- 0.046, p < 0.4815), and are similar to the local isotope signature ($t_{(24)}$=- 0.699, p< 0.246) with average values of $^{87}Sr/^{86}Sr$= 0.70788 for human bones and 0.70789 for limestones and dolostones. The isotope compositional similarities indicate a local intake of strontium, suggesting local origin of individuals or long residence time in the vicinity of the site.

Within tooth sample $^{87}Sr/^{86}Sr$ isotope ratios, notable differences between dental enamel and dentine fractions can be observed (Fig. 7). Dentine isotope composition is slightly more radiogenic than bones, but values are more scattered. The $^{87}Sr/^{86}Sr$ isotopic ratio values in dental enamel fractions vary largely (Fig. 6). In several individuals, the isotopic composition of the enamel is identical to dentine. However, other individuals (samples 72, 181, than dentine fractions, indicating a childhood or adolescence outside of the place of residence at death (Alegría-Dulantzi). Therefore, the resulting 87Sr/86Sr enamel values indicate non-local provenance (Table 1, Fig. 6). Within foreign individuals, two distinct isotope compositional groups are distinguished. Samples 72, 181, and 204 have $^{87}Sr/^{86}Sr$ isotope ratios ranging from 0.708445 to 0.708547, whereas samples 188, 210, and 211 have distinctive higher of radiogenic strontium contents, with $^{87}Sr/^{86}Sr$ between 0.709129 and 0.709196. These two enamel groups indicate two foreign populations with provenance from different geological settings.

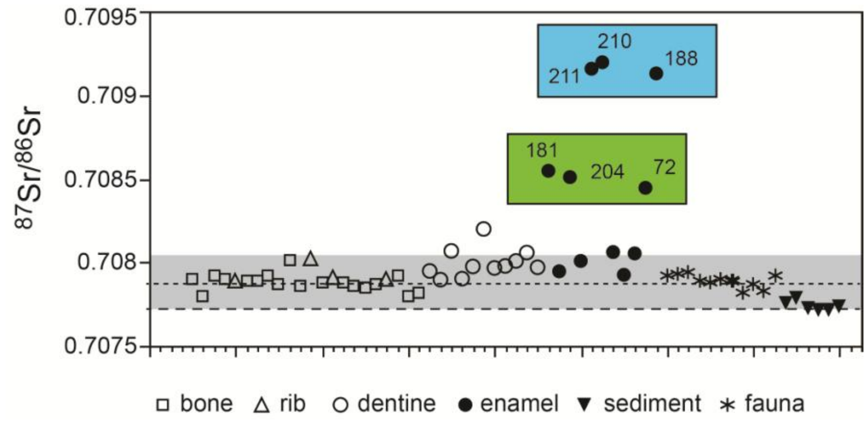

Figure 7. Isotope variation of the studied samples. Grey area corresponds to thevariation range in local waters (Fernández de Ortega, 2007). Dashed line indicates the average isotope ratio of soil at the site. Doted line corresponds to the average for the bones. Green and blue areas indicate immigrants from different geological environments. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

When comparing radiogenic strontium isotope data of local signature and dentine of foreign individuals, some values differ from local composition (Fig. 8). Individuals residing in the same place for multiple years will exhibit dentine $^{87}Sr/^{86}Sr$ values closer to, or within, the local $^{87}Sr/^{86}Sr$ range. In cases of migrant individuals who have resided at the location a few years prior to death, dentine and bone strontium isotope composition will rapidly turnover to local values (Slovak and Paytan, 2011). Dentine $^{87}Sr/^{86}Sr$ signatures probably will fall somewhere between the $^{87}Sr/^{86}Sr$ range of their previous location and the current place of residence.

The shift observed is consistent with migrants who relocated to Alegría-Dulantzi for the last years of their lives.

Finally, immigrant individuals include both men and women, and except for individual 204, a male buried in a privileged site, the individuals had no associated grave goods. Therefore, burials with grave goods correspond mainly to local origin individuals.

The results of $^{87}Sr/^{86}Sr$ isotope study of human remains from the Early Medieval Alegría-Dulantzi rural cemetery indicate a community mainly of local individuals and a low presence of immigrants.

Foreign individuals are distributed throughout the Early Middle Ages (6th-10th centuries, Fig. 8) and are not focused in the migration events related to the fall of the Roman Empire. This study leads to questions concerning the real impact of migration and the ethnic paradigm when interpreting burials with grave goods of the sixth and seventh centuries. This pattern of burials with Germanic grave goods is also observed at the Aldaieta site, located a few kilometers from Alegria-Dulantzi. Aldaieta corresponds to a large cemetery of 6th and 7th centuries with many weapons and grave good of trans-Pyrenean origin or influence (Azkarate Garai-Olaun, 1999). Despite this, palaeogenetic studies on skeletons from Aldaieta site have shown that the settlement population was substantially local individuals, without any clear evidence of immigrants (Alzualde et al., 2006).

Establishing the geographic origin of Alegría-Dulantzi immigrants is not possible given the current state of studies. The Dulantzi $^{87}Sr/^{86}Sr$ isotopic ratios are compatible with several French sectors (Ben Othman et al., 1997; Semhi et al., 2000).

However, the lack of strontium isotope data from different (204, 188, 210 and 211) have more radiogenic $^{87}Sr/^{86}Sr$ isotope ratios environments of the Iberian Peninsula and southern Europe do not allow accurate determination of the origin of immigrants. This work has only established the limited impact of migration in the northern Iberian Peninsula during the Early Middle Ages.

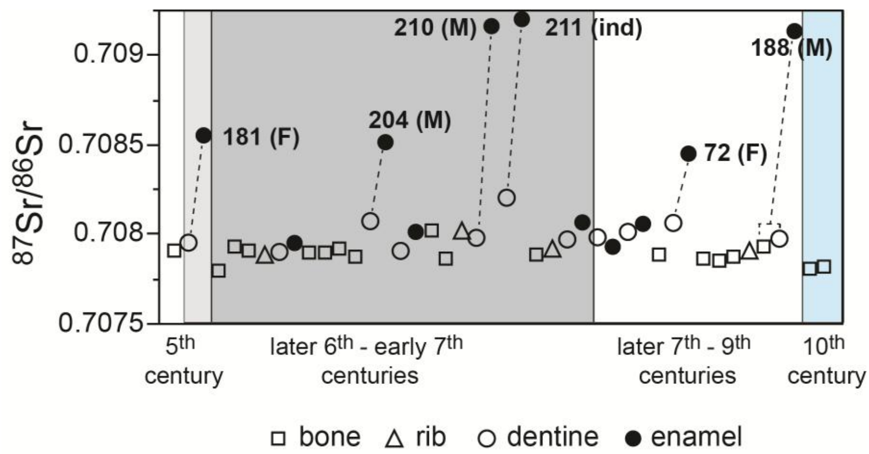

Figure 8. Isotope variation of the studied samples throughout time. Dashed lines join enamel and dentine of the same sample.

Conclusions

Domestic animal bones yield local strontium isotope composition. Human bone remains have similar isotope composition to domestic fauna, indicating local origin of individuals or long residence time in the Alegría-Dulantzi region. The sediment $^{87}Sr/^{86}Sr$ isotope ratio is significantly lower than in either human and domestic fauna bones, indicating insignificant isotope modification during burial processes, in accord with the FTIR analyses.

The tooth enamel analyses show three isotope composition groups. The individuals having similar isotope composition to the regional geological background are considered of local provenance.

Individuals showing different radiogenic isotope composition indicate childhood or adolescence outside of the Alegría-Dulantzi area, and therefore are considered immigrants. Analyses reveal two groups of immigrants from distinct geological environments.

The demographic profile indicates a community mainly of local individuals and a low presence of immigrants. Foreign individuals are distributed throughout the Early Middle Ages and are not focused in migration events related to the fall of the Roman Empire.

The data question the actual impact of suggested large migrations. The Dulantzi population consisted mainly of a local society with some foreign individuals, although the accurate geographical origin cannot be established. The migration cycles occurred during Phases 4 and 5 at the site (6th-9th centuries) involving the movement of a very limited number of adults and youth of both genders. With the exception of individual 204, none of the immigrants was buried with grave goods.

These results are consistent with isotope studies performed in other European sites. These studies analyse the meaning of grave goods and the movement patterns and reveal complex scenarios.

Hakenbeck et al. (2010) studied the Bavarian cemeteries of Altenerding and Strubung-Bajuwarenstrasse, and concluded that there were no statistical differences between the presence, amount, and value of the grave goods and the mobility patterns.

These results pose interesting challenges for understanding of the links between burial rites, artefacts and ethnic identity, and appear to support more recent theoretical approaches, which prefers to see ethnicity as fluid and constructed identify rather than a biological matter (Halsall, 2007).

Chapter II:

Las Gobas site

"Isotopic evidence for the reconstruction of diet and mobility during village formation in the Early Middle Ages: Las Gobas (Burgos, northern Spain)"

Archaeological and Anthropological Science 2017, DOI 10.1007/s12520-017-0510-9

Abstract

Strontium, carbon and nitrogen isotopes of human bone and tooth remains have been used to reconstruct residential mobility and diet of early medieval populations at Las Gobas from the 6th to 11th centuries. Most non-local individuals correspond to the 10th-11th centuries and were mostly women and infants. This residential mobility coincided with the formation of Laño village and the abandonment of artificial cave settlement. Carbon and nitrogen isotope ratios of bone collagen indicate an omnivorous homogenous diet based on terrestrial plant resources, with few animal-derived proteins from livestock. Millet consumption was restricted to an earlier period of time (7th-9th centuries), and in later periods (10th-11th centuries) mainly C3 plants such as wheat and barley were consumed. In general, there were no dietary differences between individuals according to sex or age. Sex-related dietary differences have only been observed in the 10th-11th centuries, when females consumed a more vegetarian diet and less animal protein. The higher $\delta^{15}N$ values in infants reflect the weaning effect, while the differences in $\delta^{15}N$ values between young adult men and young adult women can be explained as a physiological factor related to pregnancy or different origins. In a comparison with contemporaneous medieval populations in the northern Iberian Peninsula, both $\delta^{13}C$ and $\delta^{15}N$ values suggest similar foodstuff resources and diet among Christian and Muslim populations.

Introduction

A profound transformation affecting territorial organization occurred after the collapse of the Roman Empire. In the Cantabrian region of north Spain, the post-Roman landscape showed a high degree of territorial fragmentation with a lack of villages or rural structures. In this historical context, small and dispersed farmsteads and a few rock-hewn dwellings dated in the 6th and 7th centuries have been identified. In the course of the 8th century, a profound transformation of the Cantabrian region landscape started with the creation and gradual expansion of a network of villages (Quirós Castillo, 2009; 2011). In the 9th century, the former peasant settlement densification occurred with the creation of true village networks. Unlike in other regions, churches in the Cantabrian region never played a significant role in the formation of village networks. Early medieval churches were constructed once the villages were created and the construction of a church implied new ways of social organization and the exploitation of the territory.

The Las Gobas site consists of cave settlement and adjoining farmsteads. The use of artificial caves has been subject to the most varied interpretations. Traditional historiography has explained such occupations as a phenomenon related to different variables of Christian asceticism (e.g. Gonzalez Blanco 1993; Monreal 1997; Castellanos 1998; Espinosa 2006). Alternatively, they have been interpreted as farming communities that later moved to new settlements as they founded medieval villages (Quirós Castillo 2006; Azkarate and Solaun 2008). The cave settlement occurred over 300 years between the 6th and 9th centuries. In the late 9th century, a gradual abandonment of the settlement occurs and the community was relocated to a new settlement site in the valley (now Laño village) but the liturgical and burial function continued until the 12th century (Azkarate and Solaun 2015). New archaeological data together with biogeochemical analyses of human remains enables the observation of peasant landscape transformation from the collapse of the Roman Empire to the formation of medieval villages.

To reconstruct the lifestyle of the early medieval peasants, carbon and nitrogen stable isotopes provide insights into dietary habits whereas strontium isotope provides information about residential movements (Alt et al., 2014; Lopez-Costas and Müldner, 2015; Hemer et al., 2017; Salazar-Garcia et al., 2016). Diet, and its change over time, can shed light on the social and economic structure and such aspects as sex, age and wealth within the communities. The aim of this work is to determine the dynamics and articulation of early medieval peasant society in the Cantabrian region through strontium, carbon and nitrogen isotope studies of human remains from Las Gobas.

Isotope background

Most dietary reconstruction in recent decades is based on carbon and nitrogen isotope values in human skeletal tissues. The principle of the method is based on the chemical signature of skeletal tissues reflecting the isotope signature of food consumed. The carbon isotope values ($\delta^{13}C$) in human bone and tooth enamel depend on the types of plants consumed directly and via animal species incorporated into human diet (Ambrose and Katzenberg 2000). Early medieval dietary resources in the Cantabrian region of north Spain consisted of C3 plants combined with C4 plants. Cereals like millet and sorghum corresponding to C4 plants group were present in Europe in the Middle Ages but the archaeobotanical data from Cantabrian sites show that millet was the only C4 plant in the diet (Quirós Castillo, 2016). The $\delta^{13}C$ of modern of C3 plants ranges between -20 and -35 ‰ and between -9 ‰ and -14 ‰ for C4 plants (Katzenberg 2000). When the carbon isotopes of foods are incorporated into human bone collagen a shift of approximately 5 ‰ occurs (Ambrose and Norr 1993). Thus, measuring $\delta^{13}C$ of bone collagen makes it possible to obtain the proportion of C3 and C4 resources consumed (Schoeninger and DeNiro 1984; Schwarcz and Schoeninger 1991; Richards 2000).

In contrast, nitrogen isotope values ($\delta^{15}N$) provide a measure of animal protein consumption compared to plant source proteins although calculating the percentage corresponding to each protein source is difficult (Bocherens and Drucker 2003; Hedges and Reynard 2007). Between trophic levels, the fractionation of nitrogen isotope leads to an enrichment in δ15N values of 2-5‰ (an average of 3‰) from diet to body tissues, although recent studies estimate an offset of about 6‰ (Bocherens and Drucker 2003; Hedges and Reynard 2007; O'Connell et al. 2012). Individuals consuming mainly vegetarian diet have $\delta^{15}N$ values ranging from 3 ‰ to 9 ‰, while individuals consuming meat of terrestrial herbivores will have $\delta^{15}N$ values ranging from 9 ‰ to 12 ‰ (DeNiro and Epstein, 1981; Hedges and Reynard 2007). Meat protein (20-25%) dominates the $\delta^{15}N$ values of bone collagen over plants (10%) in individuals with an omnivorous diet. Cantabrian region medieval peasant communities practiced an economic structure of self-subsistence based on a reduction of risk of the production patterns of agriculture and livestock, and $\delta^{15}N$ values will reflect the protein sources (Quirós Castillo 2013a; Quirós Castillo 2016).

The territorial reorganization in the Early Middle Age, during the formation of villages, involved the restructuring of peasant society and hence residential mobility of individuals. Strontium isotope signature potentially enables the identification of local and non-local individuals, although it is crucial to define the local $^{87}Sr/^{86}Sr$ baseline (Bentley et al. 2004; Price et al. 2002, Tütken et al. 2011). Strontium is incorporated in bone apatite and dental enamel by the intake of food and water so individuals consuming local food and inhabiting a specific geological region will have an isotope signature reflecting the area (Ericson 1989; Bentley 2006; Price et al. 2002). Local freshwater, soil, bedrock and archaeofauna can usually be used to define the local bioavailable strontium isotope composition (Montgomery et al. 2006; Voerkelius et al. 2010; Frei and Price 2012).

Materials and Methods

Las Gobas is an excellent site for an investigation into rural landscape transformation in the Cantabrian region during the Early Middle Age until the creation of village networks. Las Gobas is located in the gorge of the Barrundia and Ayuda streams in Laño (Burgos, north of Spain). The rock-hewn dwellings on the western bank correspond to Las Gobas site whereas the artificial caves on the eastern bank form the Santorcaria site (Fig. 1). The whole complex is formed by 29 caves and is considered one of the best examples of rock-hewn dwellings in the north of the Iberian Peninsula (Azkarate 1988). In particular, Las Gobas consists of 13 artificial caves of different geometries and uses. Radiocarbon analyses date the site between the 6th and 11th centuries (Table 1) and the history and evolution of the site shows two main phases according to the use of the space, Phase I from the 7th to 9th centuries and Phase II from the 10th to 11th centuries (Azkarate and Solaun 2008, 2015). Phase I comprised the first rock-hewn church (Las Gobas 6) and several single-room dwellings, with a large wooden building and a graveyard with 15 graves on the terraced hillside (Fig. 2). The wooden building was later rebuilt in stone together with other archaeological evidences suggesting power and wealth (Wickham 2008; Bianchi 2012). In Phase II the second rock-hewn church (Las Gobas 4) and three huge silos were dug. These silos implied an increase in storage capacity due to the development of new farmland favoring the gradual abandonment of the settlement in favor of a new emplacement and the foundation of the present Laño village. At the same time, the graveyard was reorganized with the development of a new level of burials. Finally, the settlement was definitively abandoned although worship in the churches continued.

Carbon and nitrogen isotopes analyses were performed for 40 human bone collagen samples corresponding to all inhumed individuals and for 15 archaeological fauna bone samples (Tables 2, 3). In addition, enamel from twenty-six teeth was analyzed for strontium isotope composition (Table 2). To define strontium isotope baseline, 13 tooth enamel samples from archaeological fauna and two freshwater samples were analyzed (Table 3). The archaeological fauna analyzed correspond to 4 sheep/goats, 3 cows, 2 pigs, 2 horses and 2 red deer.

Figure 1. Geographical location of Las Gobas archaeological site (modified from Azkarate 2008).

Figure 2. Image showing rupestrian caves of the site of Las Gobas

Tabla 1. List of the directly dated samples from Las Gobas site (Laño, Burgos), with result of AMS dating and chronological range. The results were calibrated at 2 sigma based on the Intcal13 atmospheric data (Reimer et al., 2013) and calculated with the 'Calib Rev 7.0.4' software (Stuiver and Reimer 1993; Stuiver et al. 2005).

Lab Code	Sample	Material	C:N	BP ages	Calibrated date 2σ range (cal. a AD)	%	Calibrated date (cal. a AD) mean probability
Ua-47414	ENT 07	Bone	3.3	1002±31	981-1051	75	1025
Ua-43972	UE 231	Bone		1134±30	857-986	88	920
Ua-47415	ENT 42	Bone	3.5	1149±31	799-972	91	892
Ua-43969	ENT 37	Bone	3.4	1204±30	764-894	91	822
Ua-43975	UE 328	Bone		1348±30	640-712	92	667
Ua-43971	UE 227	Bone		1356±30	622-695	94	663
Ua-43974	UE 305	Bone		1370±30	610-687	100	656
Ua-43970	UE 102	Bone		1393±33	597-675	100	644
Ua-46155	ENT 26	Bone		1400±30	599-668	100	641
Ua-43973	UE 245	Bone		1467±30	549-645	100	598
Ua-43976	UE 357	Bone		1525±31	505-603	63	540

Table 2. Results of isotopic analysis for the human individuals from Las Gobas site (Burgos).

Sample	Tooth	Bones	Centuries	Sex	Age group	$^{87}Sr/^{86}Sr$ ± 1s(last digit)	$\delta^{15}N$‰	$\delta^{13}C$‰	C/N	%C	%N
LG-1	Max M3 Rigth	costal fragment	7th-9th	Male	Adult	0.707976 ± 6	8.6	-18.15	3.3	43.4	15.6
LG-28	Max I2 Left	costal fragment	7th-9th	Male	Adult	0.708090 ± 5	8.71	-19.05	3.3	42.8	15.1
LG-4		Rib	7th-9th	Indet	Infant I		7.68	-18.8	3.3	39.6	13.7
LG-8		costal fragment	7th-9th	Female	Adult		8.26	-18.96	3.3	41.9	14.9
LG-22		costal fragment	7th-9th	Male	Adult		8.5	-19.18	3.4	40.4	13.9
LG-26	Max M2Right	Metatarsal	7th-9th	Male	Young adult	0.708248 ± 6					
LG-28		Metatarsal	7th-9th	Male	Young adult	0.708863 ± 4	9.59	-18.21	3.4	43.2	15.1
LG-31		costal fragment	7th-9th	Female	Adult		8.23	-18.32	3.4	39.7	13.7
LG-32	Max PM2 Right	costal fragment	7th-9th	Male	Adult mature	0.708368 ± 5	8.78	-19.03	3.4	43.1	14.9
LG-33	Max M2 Left	costal fragment	7th-9th	Male	Adult mature	0.708901 ± 6	8.08	-19.27	3.4	42.4	14.7
LG-34	Max	Rib	7th-9th	Male	Adult	0.708113 ± 5	8.02	-19.26	3.3	43.1	15.1
LG-36	PM1	costal fragment	7th-9th	Male	Adult mature	0.708012 ± 4	9.43	-18.27	3.3	44.1	15.4
LG-37	Max M2 Left	Phalanx	7th-9th	Male	Adult	0.707889 ± 4	9.38	-18.36	3.4	42.9	14.7
LG-38	Max PM2 Left	costal fragment	7th-9th	Male	Young adult	0.708312 ± 5	9.41	-18	3.3	41.3	14.7
LG-47	Max PM1 Right	Phalanx	7th-9th	Female	Adult	0.707881 ± 4	9.01	-19.37	3.4	43.4	15.1
LG-2	Max M2 Left	costal fragment	10th-11th	Female	Young adult	0.708619 ± 5	8.4	-19.93	3.3	41.7	14.7

Table 2. Continued. Results of isotopic analysis for the human individuals from Las Gobas site (Burgos).

Sample	Tooth	Bones	Centuries	Sex	Age group	$^{87}Sr/^{86}Sr$ ± 1s(last digit)	$\delta^{15}N$‰	$\delta^{13}C$‰	C/N	%C	%N
LG-103	Max PM Left	3 metatarsal	10th-11th	Female	Adult	0.708863 ± 5	9.22	-17.21	3.3	42.3	14.9
LG-7		Vertebra	10th-11th	Female	Adult mature		8.71	-18.71	3.3	41.6	15.2
LG-7 ENT		costal fragment	10th-11th	Indet	Indet		8.95	-18.63	3.3	43.4	15.4
LG-9	Mand M1 Right	costal fragment	10th-11th	Indet	Infant I	0.708667 ± 4	7.7	-20.09	3.3	42.1	14.8
LG-10		costal fragment	10th-11th	Female	Adult		9.04	-19.09	3.3	41.9	14.8
LG-11		costal fragment	10th-11th	Male	Adult		8.24	-18.97	3.3	41.8	14.7
LG-13		diaphyseal radius	10th-11th	Indet	Infant I		10.66	-18.53	3.3	42.5	14.5
LG-14		costal fragment	10th-11th	Indet	Infant I		10.16	-19.16	3.3	41.6	14.7
LG-12		costal fragment	10th-11th	Indet	Infant I		11.5	-18.94	3.3	43.3	15.4
LG-17	Mand M2 Right	Rib	10th-11th	Female	Young adult	0.708622 ± 5	8.51	-18.94	3.3	43.4	15.5
LG-23		costal fragment	10th-11th	Female	Adult		8.39	-19.55	3.3	41.2	14.6
LG-24		diaphyseal ulna	10th-11th	Indet	Infant I		11.66	-19.02	3.3	44.0	15.6
LG-27		costal fragment	10th-11th	Indet	Indet		8.68	-19.58	3.4	43.1	15.0
LG-29	Mand PM1 Right	costal fragment	10th-11th	Male	Adult mature	0.707871 ± 5	8.55	-18.82	3.3	43.3	15.4
LG-30	Max M3 Left	Rib	10th-11th	Female	young adult	0.708675 ± 5	8.53	-19.49	3.4	40.9	14.2

Table 2. Continued. Results of isotopic analysis for the human individuals from Las Gobas site (Burgos).

Sample	Tooth	Bones	Centuries	Sex	Age group	$^{87}Sr/^{86}Sr$ ± 1s(last digit)	$\delta^{15}N$‰	$\delta^{13}C$‰	C/N	%C	%N
LG-35	Max M2 Left	Ulna	10th-11th	Male	Adult mature	0.708317 ± 5	8.43	-19.43	3.6	37.5	11.9
LG-39	Max M2 Left	Sacrum	10th-11th	Male	Young adult	0.708009 ± 5	9.12	-18.97	3.5	38.0	12.7
LG-40	MaxPM2 Right	costal fragment	10th-11th	Male	Young adult	0.708063 ± 5	8.09	-18.68	3.3	41.3	14.7
LG-41	Mand PM2 Right	costal fragment	10th-11th	Male	Young adult	0.708349± 6	9.48	-18.32	3.2	43.5	15.7
LG-42	Max M2 Right	costal fragment	10th-11th	Female	Young adult	0.708482 ± 5	8.47	-19.27	3.4	41.4	14.4
LG-43	Max M2 Right	calcaneus	10th-11th	Male	Young adult	0.708274 ± 4	8.31	-19.31	3.3	43.3	15.3
LG-44	Max M2 Right	rib	10th-11th	Female	Young adult	0.708412 ± 6	7.68	-19.51	3.2	42.3	15.3
LG-45	Mand M2 Left	costal fragment	10th-11th	Indet	Infant I	0.708234 ± 6	10.26	-19.71	3.5	41.6	14.4
LG-46	Max M2 Left	rib	10th-11th	Male	Adult mature	0.708056 ± 5	8.81	-19.67	3.5	36.7	12.2
LG-3		femoral shaft	10th-11th	Indet	Indet		8.71	-19.33	3.4	40,1	15.2

max., maxillary; mand., mandibular; M., molar; PM., premolar; I., incisor; Indet., indeterminate

Table 3. Results of isotopic analysis for the fauna and freshwater from Las Gobas site (Burgos).

Sample	Species	Period	Centuries	Material	Tooth type	$^{87}Sr/^{86}Sr \pm 1\sigma$ (last digit)	δ^{15}N‰	δ^{13}C‰	C/N	%C	%N
LG-163.12	sheep/goat	1	7th	Tooth	Mand M1-2	0.710219 ± 7					
LG-198.12	cow	2	10th	Tooth	Max M1-2	0.710323 ± 6					
LG-198.38	red deer	2	10th	Tooth	Max M1-2	0.708229 ± 7					
LG-199.92	horse	1	9th-10th	Tooth	Mand P2	0.711527 ± 12					
LG-249.1	sheep/goat	2	10th-11th	Tooth	Max M1-2	0.708163 ± 7					
LG-265.1	cow	1	8th	Tooth	Mand M3	0.707689 ± 6					
LG-301.1	cow	1	7th	Tooth	Mand M?	0.707990 ± 8					
LG-301.4	pig	1	7th	Tooth	Mand M?	0.708183 ± 7					
LG-340.1	sheep/goat	1	8th-9th	Tooth		0.708035± 8					
LG-199.92	horse	1	9th-10th	Tooth		0.708585 ± 6					
LG-163.12	sheep/goat	1	7th	Tooth	Mand M1-2	0.708600 ± 5					
LG-328.6	red deer	1	7th	Tooth	Max M1-2	0.708347 ± 8					
LG-102.9	horse	2	10th-11th	Tooth	Mand M3	0.707984 ± 6					
LG-Agua	water					0.707842 ± 8					
Rio Ayuda	water					0.707894 ± 8					
LG-160.228	sheep/goat	1	9th-10th	Bone	Vertebra		4.11	-20.57	3.3	41.5	13.2
LG-160.281	horse	1	9th-10th	Bone	Metacarpal		2.36	-21.5	3.3	41.8	13.8
LG-198.18	Cow	2	10th	Bone	Humerus		1.87	-21.72	3.3	40.8	15.2
LG-198.27	sheep/goat	2	10th	Bone	Axis		4.31	-20.43	3.3	39.9	14.6
LG-198.35	pig	2	10th	Bone	Calcaneus		4.8	-20.27	3.3	37.9	14.2
LG-198.40	red deer	2	10th	Bone	Scapula		4.45	-20.51	3.2	42.9	14.7
LG-227.17	red deer	1	7th	Bone	Vertebra		2.46	-21.9	3.3	41.3	13.9

Table 3. Results of isotopic analysis for the fauna and freshwater from Las Gobas site (Burgos).

Sample	Species	Period	Centuries	Material	Tooth type	$^{87}Sr/^{86}Sr \pm 1\sigma$ (last digit)	$\delta^{15}N$‰	$\delta^{13}C$‰	C/N	%C	%N
LG-227.79	pig	1	7th	Bone	Scapula		6.78	-20.53	3.3	41.3	15.2
LG-227.188	sheep/goat	1	7th	Bone	Scapula		4.91	-20.62	3.3	41.5	15.8
LG-245.84	sheep/goat	1	6th-7th	Bone	Pelvis		4.8	-21.27	3.3	40.7	15.3
LG-265.3	cow	1	8th	Bone	Rib		4.46	-21.04	3.3	39.7	15.3
LG-265.16	sheep/goat	1	8th	Bone	Humerus		5.41	-21.06	3.3	41.5	13.2
LG-312.2	cow	1	8th	Bone	Vertebra		2.64	-22.18	3.2	42.5	14.5
LG-312.7	sheep/goat	1	8th	Bone	Mandibular		5.47	-20.34	3.3	39.8	14.7
LG-312.15	pig	1	8th	Bone	Vertebra		5.9	-20.58	3.3	41.2	15.3

Human remains correspond to 19 males, 12 females, 7 infants and 3 of indeterminate sex (Table 2). The anthropological analysis was performed by Herrasti and Etxeberria (2014). Sex determination was carried out according to the classical patterns of dimorphism and age was defined by the most reliable markers: changes in auricular surface and pubic symphysis, epiphyseal closure, cranial sutures and dental eruption (Ferembach et al., 1980; White et al., 1991). Individuals were categorized by age into infants (aged younger than 7), young adults (aged 16–27), adults (aged 27–35), mature adults (aged 35–50) and senile (aged older than 60). Within this classification, the absence of individuals aged from 7 to 12 years old is noteworthy and all the individuals were younger than 50 years old, except for one man of over 60 years of age.

For stable isotope analyses, bone collagen was extracted following the Bocherens et al. (1991) procedure at the University of the Basque Country (UPV/EHU). Long bones, when possible, or rib bones were pulverized and 300 mg of bone powder was demineralised in 1 M HCl for 20 min at room temperature until the sample dissolved. To remove humic acid, the samples were rinsed with distilled water and treated with 0.125 M NaOH. After having been rinsed again with distilled water, the resulting insoluble fraction was gelatinized in HCl solution at pH 3 for 17 h at 90 ºC. Then, samples were filtered using a MCE membrane filter (5µm) before being freeze-dried and finally, lyophilized using a FreeZone Plus 12 Liter Lyophilizer. Lyophilized collagens (0.900-1.100 mg) were enclosed in tin capsule for isotopic analysis. Carbon and nitrogen isotopes analyses were performed using a continuous-flow isotope ratio mass spectrometer (EA-IRMS) at Iso-Analytical (Cheshire, UK).

To confirm instrument accuracy, internal standards of multiple samples of bovine liver NBS-1577B standard and ammonium sulphate IA-R045 standard were used. Isotopic values are reported as δ values in per mil (‰) relative to international defined standards for carbon (VPDB: Vienna Pee Dee Belemnite) and nitrogen (AIR: Ambient Inhalable Reservoir). The instrumental precision for $\delta^{13}C$ was ± 0.06‰ or better and for $\delta^{15}N$ was between ± 0.06‰ to ± 0.08‰, determined by replicated analyses of internal standards.

Tooth enamel was used to determine strontium isotope composition. The samples were washed by ultrasonic bath to remove impurities and further cleaned by mechanical abrasion to remove the outer surface to avoid potential contamination. A fraction of dental enamel was collected mechanically with a diamond-coated trepanation drill (MF-perfect, W & H Dentalwork, Bürmoos, Austria). The direction of the sampling was always perpendicular to the growth axis of the tooth. Enamel samples (~10 mg) were dissolved in 7 mL Savillex® vials (Minnetonka, MN, USA) with 1.5 mL of 2N HNO_3 (analytical grade purified by sub-boiling distillation).

The isotope ratios were determined by Thermal Ionization Mass spectrometry (TIMS) using a ThermoFinnigan MAT 262 multi-collector mass spectrometer at the Advanced Research Facilities (SGIker) of the University of the Basque Country (UPV/EHU). Multiple samples of strontium of reference material NBS 987 were run to confirm instrument accuracy. External batch reproducibility was ± 0.00002 (absolute 2σ) based on 232 measurements. Replicate analyses of the NBS 987 during runs was 0.710268 ± 12 (2σ, n = 7).

Statistical tests were performed using SPSS for windows version 20. Differences between sample groups were analyzed by applying the two-tailed Mann-Whitney U test. This test was selected over the t-test because of small sample sizes, import differences in sample size between groups and some heterogeneity between variances. The null hypothesis states that there is no difference between the ranks of two samples. A probability level of 5% was considered significant to reject the null hypothesis.

Results

Carbon and Nitrogen isotopes

The human and fauna analytical results are listed in Table 2. The collagen quality and diagenesis effect were verified according to C/N atomic ratios. Collagen yielding a C/N of 2.9-3.6 was considered acceptable for stable isotope analyses and radiocarbon dating (DeNiro 1985; Ambrose 1990; Schwarcz and Schoeninger 1991). The C and N in collagen samples are higher than +36.7 %wt and +12.0 %wt respectively, with a C/N atomic ratio between 3.2-3.6, indicative of well-preserved collagen.

The $\delta^{13}C$ ratios for Las Gobas individuals (n=40) range between -20.1 and -17.2‰ (mean -19.0‰ ±0.58, 1σ) and $\delta^{15}N$ values range from +7.7 to +11.7‰ (mean 8.9‰ ±0.9, 1σ). Unlike the $\delta^{13}C$ values, the $\delta^{15}N$ values display significant variations. Nitrogen isotope ratios of most individuals are lower than +10.0‰ (n=33) while five infants have $\delta^{15}N$ higher than +10.0‰ ($p<0.001$). Carbon and nitrogen isotope values of archaeological fauna reveal two compositional groups. The first fauna group consisting of two cows, one horse and one red deer is largely depleted in $\delta^{13}C$ (n=4) (mean -21.8‰ ±0.3, 1σ) and $\delta^{15}N$ (mean +2.3‰ ±0.3, 1σ) while the second group formed mainly by pigs, sheep/goats and one cow and one red deer (n=11) are less depleted in $\delta^{13}C$ (mean -20.7‰ ±0.3, 1σ) and enriched in $\delta^{15}N$ (mean +5.0‰ ±0.8, 1σ). The shift between the first group of fauna and humans are on average about $\Delta\delta^{15}N$ 6.5‰, $\Delta\delta^{13}C$ 2.8‰, while the shift between the second group of fauna and human is lower ($\Delta\delta^{15}N$ 4‰, and $\Delta\delta^{13}C$ 1.7‰, on average) (Fig. 3).

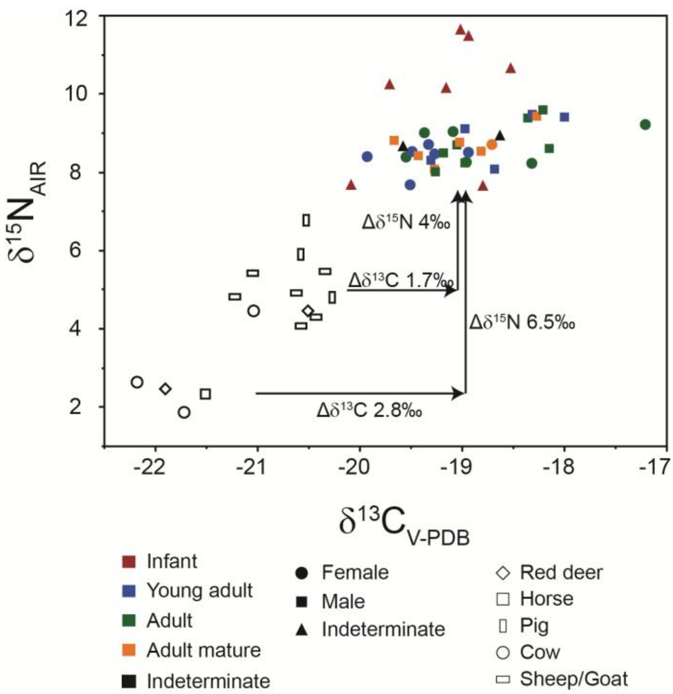

Figure 3. Human bone collagen $\delta^{13}C$ and $\delta^{15}N$ isotope values in comparison with the fauna isotope data from Las Gobas.

Nitrogen and carbon isotope composition of adult individuals does not shown statistical differences by sex or age. However, significant differences in $\delta^{13}C$ values were observed between settlement phases ($p=0.04$). Within Phase I, the $\delta^{13}C$ values are clustered into two groups; one group with $\delta^{15}N$ mean values of +9.1‰±0.6 (1σ, n=6) and $\delta^{13}C$ mean values of -18.2‰±0.1 (1σ), and the other with $\delta^{15}N$ mean values of +8.5‰ ±0.4 (1σ, n=7) and $\delta^{13}C$ mean values of -19.2‰ ±0.2 (1σ). In Phase II, only when considering young adult individuals, females have significantly lower mean values in $\delta^{13}C$ (-19.4±0.4, 1σ, n=4) than males (-18.8±0.4, 1σ, n=4) ($p=0.04$).

Strontium isotopes

The results of strontium isotope values of human and archaeological fauna dental enamels and local waters are shown in Table 2. Local freshwater composition ranges between 0.70784 and 0.70789 and, archaeological fauna composition varies between 0.70769 and 0.71153. Additionally Las Gobas bedrock lithology strontium values were taken into consideration, where $^{87}Sr/^{86}Sr$ composition varies between 0.70796 and 0.70813 (Baceta et al. 2013). The human dental enamel $^{87}Sr/^{86}Sr$ values range between 0.70787 and 0.70890.

Discussion

Residential Mobility

Archaeofauna, local freshwater and bedrock composition were considered to establish the local strontium isotope signature at Las Gobas. The lithology is quite homogeneous and is formed by carbonate sedimentary rocks; mainly dolostones and limestones. Since the bedrock in the area surrounding Las Gobas is uniform, strontium isotope composition of both bedrock and local freshwater can be used to establish the local strontium baseline. Archaeological fauna associated with the site also reflect the average bioavailable strontium isotope composition. However, one horse, one cow and one sheep/goat deviate significantly from local bedrock and freshwater isotope values, suggesting trade and transhumance of livestock. Considering the uncertainty of livestock to establish the isotope baseline, not only freshwater and bedrock but also contemporaneous wildlife (red deer) was considered (grey area in Fig. 4). Thus, the local baseline, at two standard deviations, exhibited a range between 0.7075 and 0.07084. Hence, most individuals of Las Gobas site were of local origin and eight are non-local although two females plot close to the upper limit of the local range (Fig. 4).

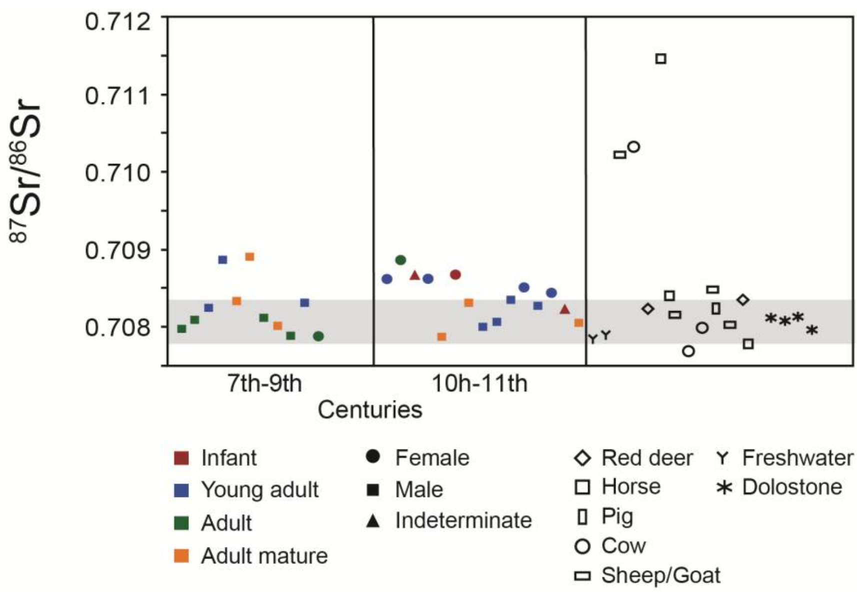

Figure 4. Strontium isotope ratios of human remains, archaeological fauna, freshwater and bedrock (Baceta et al. 2012) at Las Gobas.

Regarding the settlement phases, most individuals in the 7th-9th centuries were of local origin except two males (LG-28 and LG-33), whereas during the 10th-11th centuries, the number of non-local individuals increased and most were females. During the Early Middle Ages, few people regularly moved because it was simply too difficult and too dangerous. However, at the time of the formation of villages, the mobility of individuals increased probably from nearby areas. Las Gobas illustrates this restructuring of peasant society where males moved to achieve better economic opportunities and possibility to improve their status and females would move by patrilocal marriages (Bittel 2002).

Dietary patterns

For firm conclusions about human diet, the local baseline must be established. Since $\delta^{13}C$ and $\delta^{15}N$ values of Las Gobas archaeofauna plot in two compositional groups, it is difficult to set the local isotope baseline. The variation of $\delta^{13}C$ and $\delta^{15}N$ in the domestic animals might be caused by varying baseline isotopic signatures due to differences in herding or feeding practices or by browsing or grazing in different habitats with different isotopic baselines (Oelze et al. 2011). The different patterns observed among domestic animal species (sheep/goat vs. cows and horses) suggest dissimilar kinds of pasture between grazers and browsers. Additionally, the strontium isotope results of fauna reveal local and nonlocal livestock and therefore habitats with a different isotope baseline. Therefore the cluster formed by pig and sheep/goat with similar isotope composition has been used to establish the local baseline, according to the $^{87}Sr/^{86}Sr$ signature.

The analyzed fauna correspond to two different periods, like the human remains. Faunal $\delta^{13}C$ and $\delta^{15}N$ values do not display statistically significant differences between the two periods, so the farming structure was not modified despite the relocation of the settlement to the village. The $\delta^{13}C$ and $\delta^{15}N$ data from Las Gobas inhabitants indicate mainly the consumption of terrestrial plant and animal-derived food. Staple food was based on cereals, mainly C3 plants like wheat, barley and legumes ($\delta^{13}C$ mean -19.0‰ ±0.58, 1σ); as the archaeobotanical evidence confirms. However, archaeobotanical evidence also shows the presence of millet (C4 plants) (Azkarate and Solaun 2015). Consequently, in addition to consumption of C3 cereals and legumes, C4 cereals like millet were also consumed directly or through fauna intake. $\delta^{15}N$ values indicate that the diet was omnivorous and meat was also consumed. However, most samples fall below the human-fauna offset for nitrogen of 6‰ indicating a diet with a low animal protein intake (O'Connell et al. 2012). Between human and faunal remains, two different shifts can be related to different use of animals. In the Middle Ages, zooarchaeological studies show that some domestic animals (cows and horses) were used for farm work and animals were sacrificed in their old age and generally were excluded from the primary production of meat (Woolgar et al. 2006). Zooarchaeological studies at Las Gobas show the predominance of ovicaprine livestock, slaughtered as both young and adult animals.

They were complemented by adult cattle and young pigs, hence providing evidence of a mixed livestock strategy oriented towards meat consumption and the production of secondary products (milk and wool).

Also worth mentioning is the important presence of wild animals, especially deer, while rabbit was also recorded and even bear (Castaños

Ugarte and Castaños de la Fuente 2014). The comparison of human values with archaeological fauna at Las Gobas indicates that the dietary spacing is as expected for trophic level enrichment (Bocherens and Drucker 2003). This can be indicated that Las Gobas inhabitants consumed pork and sheep/goat as their main source of proteins. Whether taking the potential dietary spacing for $\delta^{15}N$ being 6‰ (O'Connell et al. 2012), the consumption of beef and horse meat cannot be ruled out as an additional dietary source.

When considering the isotopic composition by sex of Las Gobas individuals, no significant difference in $\delta^{13}C$ and $\delta^{15}N$ exist. Same cases of peasant settlement studies in Iberian Peninsula showed differences between males and females, whereas in other cases no differences were seen between sexes. This heterogeneous behavior can be related to local dynamics (Mundee 2010). Assuming a consumption of typical C3 plants foods with similar values in the literature ($\delta^{13}C$ = -26‰, Schoeninger and DeNiro 1984) and the $\delta^{13}C$ value of consumer's collagen is approximately 5‰ higher than that of their diet (Ambrose and Norr 1993), the individuals consuming only C3 plants have $\delta^{13}C$ values of about -20‰ (Chisholm et al. 1982; Schoeninger et al. 1983). In the absence of marine food, which can also cause an increase in $\delta^{13}C$, stable carbon isotope analyses of bone collagen can be used to determine the contribution of C3 vs C4 plants to the diet. Most individuals at Las Gobas fall into the region expected for C3 resource dependence, indicating that the main diet was based on such staples as wheat and barley. However six individuals, five males and one female (LG-3, LG-28, LG-36, LG-38, LG- 39, LG-41), shift slightly away from the mean composition with $\delta^{13}C$ values of ca -18.2‰, indicating a relatively major consumption of C4 resources such as millet, which might have been consumed directly or indirectly through consumed fauna.

Statistical comparison by age revealed no significant difference among young adults (18–27 years), adults (27–35 years) and mature adult (35–50 years) when calculated for each sex separately and both sexes combined. The lack of significant variation in the carbon and nitrogen isotope composition indicates the absence of dietary changes among adults, even those who are elderly. Five infant individuals (younger than 3 years of age) have more positive mean values of $\delta^{15}N$ +10.9 ± 0.7‰ (1σ) compared to adults.

The enrichment in nitrogen isotopic signal can be explained by breastfeeding (Schurr and Powell 2005). Breastfeeding results in higher nitrogen isotope values in the infant's tissue compared to mothers due to the trophic level effect (Fuller et al. 2005; Schwarcz and Schoeninger 2011). However two infants have the lowest $\delta^{15}N$ values (<8‰) as found in post-weaned infants that died aged between 3-6 years. The lower δ15N values could be attributed to a major solid food ingest in the diet (Richards et al. 2002). Lower δ15N values in immature individuals (<8‰) are also attributed to nitrogen imbalance during periods of intense growth (De Luca et al. 2012).

When addressing questions of temporal variations in diet, the isotope data of infants were excluded to avoid the nursing effect. Variations in $\delta^{15}N$ and $^{87}Sr/^{86}Sr$ between time periods are not significant. On the contrary, $\delta^{13}C$ mean values in the 7th-9th centuries are significantly enriched (p=0.042) compared with the 10th-11th century samples (Fig. 5). Besides, within the 7th-9th centuries, two sets of individuals were observed and one of them shift towards a slightly less negative mean value of $\delta^{13}C$ corresponding to millet consumers. Such variation does not reflect a large change of diet in the broad population but it can indicate two groups with different diets. The absence of precise dating of 7th-9th century individuals with apparently different diets does not allow us to establish whether these groups correspond to different times or if both groups coexisted throughout the whole time. The hypothesis of two populations from different times who change diet is more probable than two groups coexisting with a different diet.

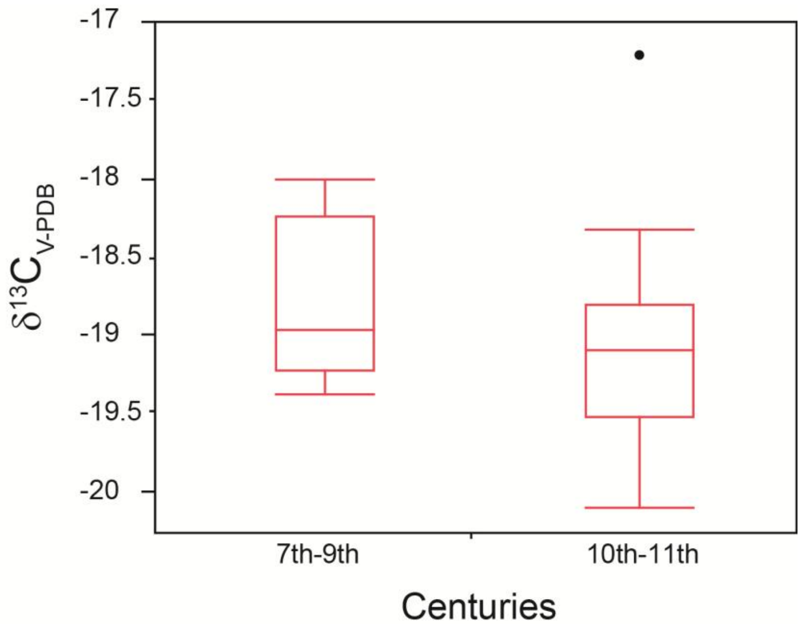

Figure 5. Boxplot of $\delta^{13}C$ values from Las Gobas individuals for different periods.

The dissimilar distribution of sex by periods of time complicates population comparisons. During the 7th- 9th centuries, most individuals were males and only three of the ten were females. In the 10th-11th centuries within a group of 26 individuals, infants (n=6) and females (n=9) were more numerous; and men were less abundant (n=8), excluding the three individuals of indeterminate sex. However, age and sex distribution provides details about differences in life expectancy between males and females. Only one female reached a mature age, corresponding to 7% of females, whereas nine men reached a mature age (43% of men). This difference in life expectancy is common in medieval ages (Acsádi and Nemeskéri 1970; Šlaus 2000; Šlaus et al. 2002). Higher female mortality was related to pregnancy and childbirth (Högberg et al. 1987; Šlaus 2000; Joyce 2001; Tocheri et al. 2005). It should be noted that most individuals were young adults, particularly females, so most females died before 30 year of age (4/5).

When comparing only the 10th-11th century young adults by sex, females have significantly lower mean values in $\delta^{13}C$ and $\delta^{15}N$ than males (Fig. 6). This difference can be attributed to differences in diet. Thus, lower isotope values in females indicate a more vegetarian diet and relatively less meat consumption than the average males. Different diets may result from sexual division of labor, characteristic of the medieval period. However, considering the number of females that died at fertile ages, the most likely hypothesis seems to be a physiological factor related to pregnancy rather than to different diet. This hypothesis is strongly supported by studies performed by Fuller et al. (2004) who found $\delta^{15}N$ depletion of 1‰ in hair of modern pregnant females. However, another option to describe the $\delta^{15}N$ depletion in females could be the different origins of the individuals of both sexes.

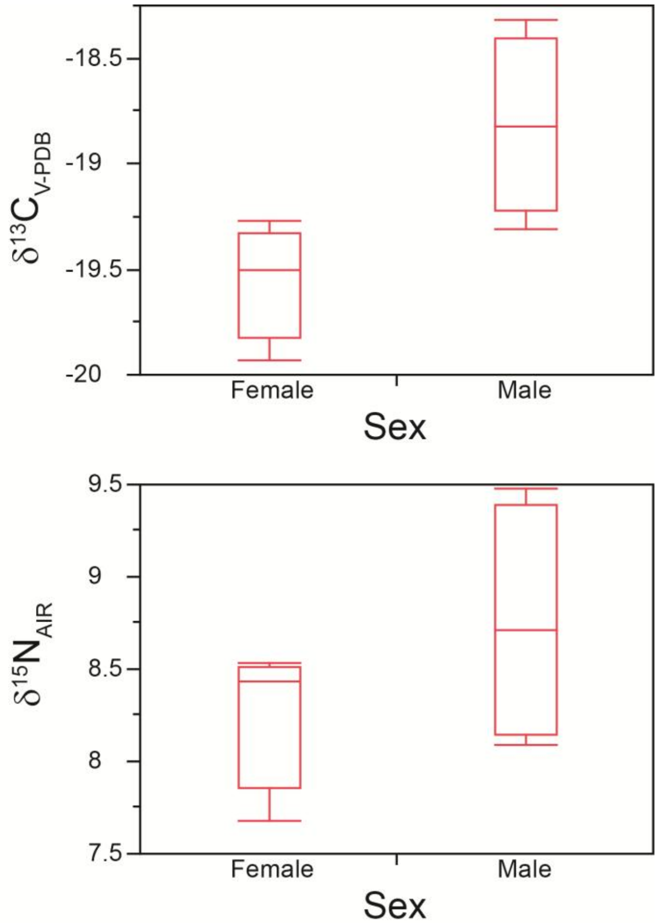

Figure 6. Boxplot of $\delta^{13}C$ and $\delta^{15}N$ values of 10th-11th century young adult individuals.

Diet compared with other settlements in Iberian Peninsula

The isotope data from Las Gobas were compared to other contemporaneous archaeological sites in order to better integrate the evidence of diet patterns with historical written sources or other archaeological proxies. Table 4 summarizes archaeological sites taken into consideration: San Martín de Dulantzi (6th- 11th centuries), Zornoztegi (7th-14th centuries), Aistra (7th-9th centuries), Zaballa (10th-15th centuries), Treviño (12th-14th) and Tauste (8th-10th centuries) (Quirós Castillo 2013b, 2013a; Guede et al. 2017, Sirignano et al. 2014). Tauste is located around 160 km away (Aragon, NE Iberian Peninsula,) and represents a Muslim population while the other sites were Christian. Since Zaballa, Zornoztegi, Aistra and Treviño sites are geographically close to Las Gobas (between 10 km and 50 km away), they are considered regional sites. Since local isotope baseline varies according to climatic conditions, only the nearby sites with archaeofauna were included for comparison. All the Christian sites correspond to the same climate region with temperate oceanic climate (Cfb type) according to the Koeppen-Geiger classification system, while the Muslim site is located in a semi-arid climate (Bsk type) (Peel et al. 2007).

Muslim population at Tauste exhibits enrichment in $\delta^{15}N$ with respect to Christian populations, which at first sight seems to suggest dietary differences between both groups of population probably linked to the differing culture and faith. Guede et al. (2017) used modern fauna from Tauste to calculate the carbon and nitrogen offset in the mean $\delta^{13}C$ and $\delta^{15}N$ values. Muslim individuals have a human-fauna offset of c. 1‰ in $\delta^{13}C$ and 4.5‰ in $\delta^{15}N$, indicating slightly higher nitrogen offset value than one trophic level, suggesting also consumption of freshwater fish.

The nearby Christian communities have mean human-fauna offsets ranging between 0.1‰ and 2.7‰ for carbon and between 2.9‰ and 5.2‰ for nitrogen (Table 4). The human-fauna mean offset for carbon higher than 2‰ suggests consumption of some C4 plants (millet) or low trophic level marine proteins.

The lack of correlation between $\delta^{15}N$ and $\delta^{13}C$, the location of the sites far from the coastline and the archaeozoological data discard marine resources, whereas archaeobotanical data point to some millet consumption. Treviño and Las Gobas show the highest mean offset for nitrogen, suggesting higher animal protein intake. Treviño shows a mean offset of 5.2‰ for nitrogen, near to a trophic level increase, suggesting regular access to animal protein intake. In fact, archaeological data indicate that the Treviño population consisted of a social structure formed mainly by an elite that regularly consumed animal proteins (Quiros Castillo 2013a). On the contrary, Las Gobas was a peasant community and the proximity to a river could explain the mean offset for nitrogen, also near to one trophic level, by the consumption of some freshwater fish.

In summary, most of the medieval samples fall below a $\Delta\delta^{15}N$ of 6‰, indicating that the diet was based on low animal protein, except for the individuals at Treviño. It might be expected that Muslim and Christian individuals had dietary differences according to religious laws. Muslims were forbidden to consume pork and any meat not prepared in the halal way (Insoll 1999; Zaouali 2007). Christians were prohibited from consuming meat during fast days and Lent, which accounts for a total of 150 days per year (Tomas 2009), and in some religious orders, meat was entirely forbidden due to their own fasting practices (Sesma 1977; Grumett and Muers 2010). However, the isotopic evidence at the sites does not support dietary differences due to these religious requirements.

Table 4. $\delta^{13}C$ (‰) and $\delta^{15}N$ (‰) mean values of human and fauna from contemporaneous medieval Iberian archaeological sites and the offset ($\Delta\delta^{13}C$, $\Delta\delta^{15}N$) between human and fauna.

Site	$\delta^{13}C$ (‰)				$\delta^{15}N$ (‰)				Reference
Human	Mean	Std Dev	Max	Min	Mean	Std Dev	Max	Min	
Las Gobas (7th-11th)	-18.9	0.6	17.2	19.9	8.7	0.5	9.6	7.7	This work
Las Gobas (7th-9th)	-18.7	0.5	-18	19.4	8.8	0.6	9.6	8	This work
Las Gobas (10th-11th)	-19.1	0.6	17.2	19.9	8.6	0.4	9.5	7.7	This work
Aistra (8th-9th)	-19	1	16.7	-22	7.9	1	12.1	6.8	Quiros. 2013a
Treviño (12th-14th)	-19.6	0.7	18.7	-22	9.6	1.2	12	7.5	Quiros. 2013a
Dulantzi (6th-11th)	-19.8	1.4			9.2	1.2			Quiros. 2013b
Zaballa (10th-15th)	-19.8	0.7	18.8	21.3	9	0.8	10.4	7.6	Quiros. 2013a
Zornoztegi (12th-14th)	-18.1	1.1	16.7	9.91	8.3	0.6	9.2	7.5	Quiros. 2013a
Tauste (8th-10th)	-17.7	1.3	14.2	18.9	15	1.4	16.6	9.3	Guede et al 2017
Fauna									
Aistra (8th-9th)	-21.7	0.3	-21.8	4.0	4.0	1.0	4.7	2.3	Quiros. 2013a
Treviño (12th-14th)	-20.6	1.1	-21.3	4.5	4.5	2.1	5.9	5.9	Quiros. 2013a
Dulantzi (6th-11th)	-20.8	0.7	-21.7	6.1	6.1	1.5	9.8	9.8	Quiros. 2013b
Zaballa (10th-15th)	-19.9	1.0	-20.6	6.1	6.1	1.8	7.5	7.5	Quiros. 2013a
Zornoztegi (12th-14th)	-20.2	2.2	-22.8	5.3	5.3	1.6	7.4	7.4	Quiros. 2013a

Table 4. Continued. $\delta^{13}C$ (‰) and $\delta^{15}N$ (‰) mean values of human and fauna from contemporaneous medieval Iberian archaeological sites and the offset ($\Delta\delta^{13}C$, $\Delta\delta^{15}N$) between human and fauna.

Site	$\delta^{13}C$ (‰)				$\delta^{15}N$ (‰)				Reference
Fauna	Mean	Std Dev	Max	Min	Mean	Std Dev	Max	Min	
Tauste (8th-10th)	-20.6		-23	10.7	10.7		14.5	14.5	Guede et al 2017
Fauna-human	$\Delta\ \delta^{13}C$				$\Delta\ \delta^{13}N$				
Aistra (8th-9th)	2.7				3.9				
Treviño (12th-14th)	1.0				5.2				
Dulantzi (6th-11th)	1.0				3.1				
Zaballa (10th-15th)	0.1				2.9				
Zornoztegi (12th-14th)	2.1				3.0				
Tauste (8th-10th)	2.9				4.3				

Conclusions

Archaeological data indicate two periods of occupation at Las Gobas in the course of five centuries from the territorial reorganization in the Early Middle Age until the formation of villages. Since the number of individuals at the site in these centuries was not large, the interpretations should be taken with caution.

Isotope composition gives insight into different socio-economic aspects of the rural medieval population. In earlier times, small communities were established in the vicinity of some rock-hewn dwellings and later they moved to a new medieval village. Although the village had been founded, liturgical and graveyard functions of the artificial caves continued centuries after leaving the site. The formation of the village involved mobility of individuals and in Las Gobas they were mainly females who would move because of patrilocal marriages. Stable isotope data indicated a steady omnivore diet consisting mainly of C3 plants, such as wheat and barley, and low animal protein intake from livestock, mainly pigs and sheep/goats. However, in the 7th-9th centuries a set of individuals differs, with $\delta^{13}C$ values ca -18.2‰, indicating a relatively major consumption of C4 resources such as millet that could have been eaten directly, or indirectly through consumed fauna.

In general, no significant sex-based variation in diet existed, nor is there any evidence of age-based variation in diet among the adults. In contrast, isotopic data reflect dietary differences between adults and infants due to the nursing effect. Dietary differences only existed between the young adult individuals in the 10th-11th centuries because of sexual division of labor, characteristic of the medieval period. In a comparison of contemporaneous Medieval populations in the northern

Iberian Peninsula, both $\delta^{13}C$ and $\delta^{15}N$ values do not suggest evident dietary differences between Muslim and Christian populations.

Chapter III:

Momoitio site

"Social structuration in medieval rural society based on stable isotope analysis of dietary habits and mobility patterns: San Juan de Momoitio (Biscay, North Iberian Peninsula)"

American Journal of Archaeological Anthropology, in revision

Abstract

Objectives: The aim of this study was to explore the social structure of a peasant community in the northern Iberian Peninsula within the historical evolution of the Middle Ages (9th to 12th centuries) through diet and mobility patterns.

Materials and Methods: A total of 93 individuals (93 bones and 63 teeth) from San Juan de Momoitio graveyard were analyzed. The study consisted of human palaeodiet characterization and mobility patterns based on $\delta^{13}C$, $\delta^{15}N$, $\delta^{18}O$, and $^{87}Sr/^{86}Sr$ isotope analyses.

Results: Carbon and nitrogen values of individuals showed variation according to burial periods defined by the grave types (Early and High Middle Ages). Oxygen and strontium isotopes showed that most individuals were from the immediate vicinity of Momoitio and only five individuals were non-local and would have come from Atlantic Ocean coastal areas (probably from south-western France).

Discussion: Carbon and nitrogen values showed a staple diet based on cereals (both C4 and C3 types), vegetables and pulses, combined with animal protein derived from husbandry and scarce marine resources. The variation in dietary habits reflected the rural social organization and changes in medieval rural society. Millet consumption increased during the High Middle Ages and coincided with the gradual abandonment of the graveyard. Nitrogen and oxygen isotope composition help a better understanding of high infant mortality related to weaning practice. Mobility patterns evidenced at least three regions for the provenance area of non-local individuals. The oxygen isotopes of

teeth also recorded climate changes throughout the time of the settlement.

Introduction

The Middle Ages in Europe extended from the fall of the Roman Empire to the Renaissance between the 5th and 15th centuries. The first period was the Early Middle Ages between the 6th and 10th centuries followed by the High Middle Ages between the 11th and 13th, and the Late Middle Ages in the 14th and 15th centuries.

In the Iberian Peninsula, the Middle Ages was defined by the conquest and prolonged Muslim presence from the 8th to 15th centuries. Although the Muslim occupation was almost total, in the northern part of the Peninsula, to the north of the Cantabrian mountain range, the territory continued under Christian rule.

Unlike other parts of the Iberian Peninsula, in this region fortresses did not articulate the territory. In the Early Middle Ages, the territorial structure in the northwestern Iberian Peninsula consisted of broad networks of rural communities organized around local elites and arranged near churches or chapels. The rural landscape showed certain disaggregation with a lack of village or rural structure and formed only by small and dispersed farmsteads. In the High Middle Ages, a profound transformation of the landscape took place as a result of the creation of a network of villages that became hegemonic.

In the framework of medieval peasantry, San Juan de Momoitio was a small community semi-dispersed in the hillsides, linked to a church and its corresponding graveyard (Garcia Camino, 2004). The presence of a church and graveyard did not imply the formation of village but served as instruments of control over the peasants. In the High Middle Ages, several churches and graveyards constructed during the previous period were abandoned, including San Juan de Momoitio, related to a process of ecclesiastical reorganization in order to integrate the territory into feudal structures and institutions that produced a profound change within peasant society and the progressive abandonment of the previous graveyards. This network was connected with the formation of new local power centers.

This study aimed to explore the structure of a medieval peasant society and the changes that occurred in the community with the reorganization of the ecclesiastical network. Palaeodiet and mobility analyses of the medieval population at San Juan de Momoitio, located in this historical and geographic context, enables the observation of these changes since the skeletal finds analyzed belong to two Medieval phases: 9-11th and 12th century.

Carbon and nitrogen stable isotopes

Carbon and nitrogen stable isotope compositions are measured as the ratios of the heavier isotope to the lighter isotope ($^{13}C/^{12}C$ or $^{15}N/^{14}N$) and are reported in standard delta (δ) notation as parts per thousand (per mil, ‰) relative to internationally defined standards for carbon (Vienna Pee Dee Belemnite, VPDB) and nitrogen (Ambient Inhalable Reservoir, AIR). Carbon ($\delta^{13}C$) and nitrogen ($\delta^{15}N$) stable isotope ratios are routinely analyzed in bone collagen. Collagen is the most abundant protein in skeletal remains and it is usually preserved after burial (Katzenberg and Saunders, 2000; LeeThorp, 2008). The isotopic composition of $\delta^{13}C$ and $\delta^{15}N$ allows the recognition of the main sources of protein in the diet (Ambrose and Norr, 1993), but does not allow any particular foods to be identified (Richards and Hedges, 1999).

Carbon isotope composition is related to the photosynthesis pathway sequences wherein the carbon of the atmosphere is fixed in plant tissues (Bender, 1968). Two large groups of plants can be differentiated, C_3 plants like wheat (*Triticum aestivum*) and barley (*Hordeum vulgare*) and C_4 plants such as millet (Panicum/Setaria) and sorghum (*Sorghum spp.*). In terrestrial environments, carbon isotopes are useful to distinguish between C_3 and C_4 plants or animals/humans consuming these plants (Katzenberg and Saunders, 2000; Lee-Thorp, 2008; Schoeninger, 1995; Schwarcz and Schoeninger, 1991). C3 plants tend to incorporate the light isotope (^{12}C) whereas C_4 plants the heavier isotope (^{13}C), and therefore C4 plants are more enriched in $\delta^{13}C$. Accordingly, $\delta^{13}C$ values of C3 plants range between -20‰ and -38 ‰ and those of C4 plants between -9 ‰ and -15 ‰ (Bender, 1968; Park and Epstein, 1961).

The $\delta^{15}N$ gives information about the origin of the proteins intaken (marine or terrestrial ecosystems) and about the trophic level DeNiro and Epstein, 1981. Moreover, the fractionation of nitrogen isotope leads to enrichment in $\delta^{15}N$ from diet to body tissue of 3-5‰ (on an average of 3‰), which is known as the trophic level effect. In marine ecosystems, the large number of steps in food chains induces very high $\delta^{15}N$ values, so high trophic level fish and birds, mammals consuming aquatic resources show greater enrichment in $\delta^{15}N$ (Richards and Hedges, 1999). In the case of $\delta^{13}C$, for each level in the food chain there is only an enrichment of ≈ 1‰ (Ambrose, 1990; DeNiro and Epstein, 1978; Hedges and Reynard, 2007; Malainey, 2011).

Stable nitrogen isotope is also used to investigate breastfeeding and weaning practices. Children with a $\delta^{15}N$ value about 2-3‰ higher than their mother indicate breastfeeding while a lower $\delta^{15}N$ value indicates weaning (Fogel et al., 1989).

Strontium and oxygen isotopes

Strontium and oxygen isotopes are two independent isotopic systems in which strontium reflects local geology and oxygen reflects geography; therefore both can be used to reconstruct movements of past populations. The combination of these two isotopic system data can restrict possible areas and provide information about the area of origin and thus mobility patterns of individuals (e.g. Bentley & Knipper, 2005; Evans et al., 2006a; Evans et al., 2006b). Oxygen and strontium isotope data do not directly determinate the area of origin of the individual. To determine the origin of the individual, human isotope values are compared with the values of the environment in which they were buried. When the strontium and/or oxygen isotope values of the individual and those of environment are similar, the individual is considered local, whereas if the values do not agree the individual is considered non-local.

Strontium has four stable isotopes (^{88}Sr, ^{87}Sr, ^{86}Sr, ^{84}Sr) of which ^{87}Sr is radiogenic, resulting from the long-lived radioactive decay of ^{87}Rb, and is therefore variable in nature. The $^{87}Sr/^{86}Sr$ ratio of a closed system is controlled by the initial $^{87}Sr/^{86}Sr$ ratio, the Rb/Sr ratio and time elapsed **(**Faure and Powell 1972**)**. Different geological substrates therefore have varying $^{87}Sr/^{86}Sr$ ratios according to the content of Sr-bearing minerals and bedrock geological age. Strontium isotope composition does not show metabolic fractionation in the body, thus indicating the bioavailable strontium in the provenance geological area. Although strontium isotope ratios ($^{87}Sr/^{86}Sr$) mainly reflect the geology of the underlying bedrock, $^{87}Sr/^{86}Sr$ may be modified by other sources, such as geological drift, sea spray or windblown dust (Bentley, 2006; Montgomery et al., 2007; Price et al., 2002).

Since $^{87}Sr/^{86}Sr$ is inherited from the local environment, it is necessary to define the so-called local bioavailable $^{87}Sr/^{86}Sr$ signature. To determine the local strontium isotope baseline, such materials as soil, freshwater, plants, ancient fauna and modern fauna can be used (Bentley et al., 2004; Evans et al., 2010; Tütken et al., 2011).

Strontium isotopes are measured in phosphate of bones and teeth because these isotopes were incorporated in the skeleton, substituting for calcium in biogenic phosphate by means of food and water consumption. In teeth, strontium isotopes substitute for calcium in enamel at the time of tooth formation and the enamel does not incorporate other elements after its formation during infancy (Hillson, 1996; Hoppe et al., 2003). Thus, the tooth enamel reflects the composition of the place of residence during childhood, while the bones are remodeling continuously and therefore their isotopic composition reflects the individual’s last years of life. This assumes the individuals mainly consumed food from the environment in which they lived.

Oxygen isotopes indicate geographic characterization of a region, which is reflected by the isotopic composition of drinking water, which varies regionally according to climatic parameters (Darling et al., 2006; Daux et al., 2008; Longinelli, 1984; White et al., 1998). The isotopic variation in precipitation on Earth is largely explained by Rayleigh fractionations that occur within air masses. The condensed water mainly contains ^{18}O isotope whereas the vapor phase is enriched in ^{16}O. As more "heavy" water is removed from the system, the remaining vapor becomes progressively lighter. Thus, the isotopic composition of precipitation at a given location is related to several environmental parameters, such as latitude, altitude, distance from the coast, amount of precipitation, surface air temperature and season (Dansgaard, 1964; Rozanski et al., 1993).

Drinking water is mainly derived from the local groundwater or local rivers, which are related to precipitation. Ingested water does not only include drinking water but also water from food. Both waters derive from precipitation and reflect the climatic conditions in the precipitation area. Milk consumption is also known to have an effect on $\delta^{18}O$. Both human (the breastfeeding effect) and animal milk are enriched in ^{18}O relative to ingested water because of metabolic fractionation. In fact, oxygen isotopes are subject to several stages of metabolic fractionation in the body from drinking water to skeletal phosphates. This fractionation is relatively well known and not well predictable and isotope ratios of drinking water ($\delta^{18}O_{dw}$) can be calculated only with a high predictive error (Bryant and Froelich, 1995; Iacumin and Venturelli, 2015; Levinson et al., 1987; Luz et al., 1984; Luz and Kolodny, 1985). To know in an approximative way the local $\delta^{18}O$ of precipitation, the IAEA databases can be used (i.e. http://www.iaea.org/water).

Dental enamel is the most adequate material for both strontium and oxygen isotope analyses since it preserves the isotopic signature of the environment at the time of tooth formation in childhood and is largely resistant to diagenetic changes (Budd et al., 2000; Hillson 1996; Hoppe et al., 2003).

Archaeological context

The settlement of San Juan de Momoitio is located in the mountain range of Oiz in Garai (Biscay, northern Spain) (Fig.1). The site is located near the Atlantic coastline at the eastern end of the Bay of Biscay, in the north of the Iberian Peninsula. A mountain range running parallel to the coast divides the region into two very different geographical areas, the northern half formed by narrow valleys flowing to the coast and a roughly plain region to the south. This geographical characteristic articulated the landscapes of both territories in different ways.

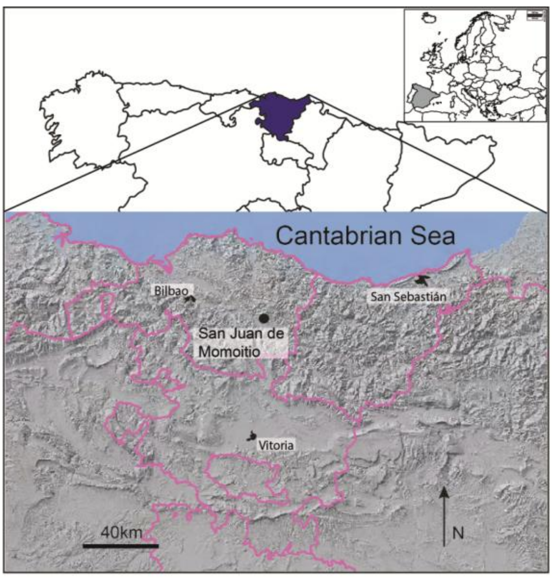

Figure 1. Location of San Juan de Momoitio archaeological site.

The graveyard of San Juan de Momoitio was arranged around a 9th century religious building. Graves were dug in soil, most of them covered with thick slabs. Three types of graves were distinguished: single earth graves, partially demarcated cist graves, and stone-lined graves. Also several upright stone slabs or steles were found, most of them without inscriptions although some show an invocation to God with the expression "In Nomine Dei ..." In many graves in Biscay found out of archaeological context this phase is very common and it is a clear sign of Christianization, based on the epigraphic characters dated around the 11th century.

The graveyard at Momoitio consists of 113 individual graves on one level of burials where most bodies appear oriented east-west. The burial area was divided into five sectors around the hermitage (Fig. 2). The cemetery expanded over time from sector 1 to sector 5. Sectors 1, 2 and 3 of the graveyard formed the first occupation phase (9th-11th centuries) in the Early Middle Ages and consist of high density areas with no spatial organization of burials. In Sector 2, two males (SJG-15 and SJG-22) stand out because they were buried in wooden graves and displayed rings as grave goods. The rings show similar characteristics both in the material and in the decoration, indicating they had the same manufacturing origin. Sector 4, located on the west side of the hermitage, showed greater dispersion of the burials with a spatial arrangement of burials and constituted the second occupation phase (12th century) within the High Middle Ages. Sector 5 consisted of only three graves and it was not possible to ascribe to which moment of the cemetery it corresponded. Unlike the first phase graves, where earth graves (A), partially demarcated cists graves (B) and stone-lined graves (C) existed, the second phase graves were less varied (Table 1, Fig. 3). In the late 13th century and early 14th century, the cemetery was abandoned and the reason was the ecclesiastical reorganization (Garcia Camino, 2002).

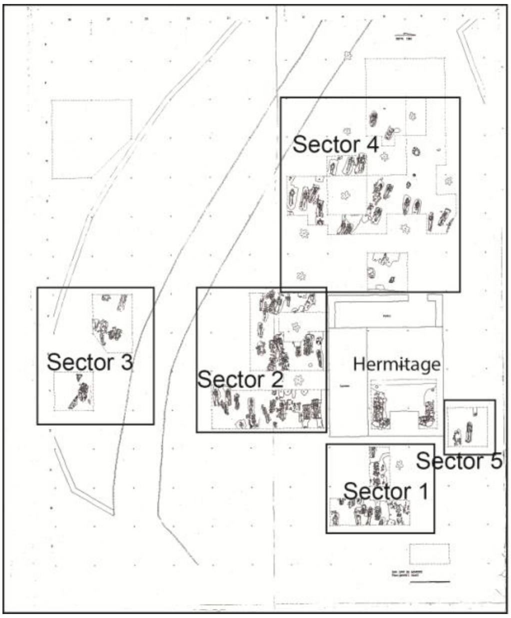

Figure 2. Aerial view of excavated sectors, showing studied individuals.

Figure 3. Types of tombs. A stone-lined grave and B partially demarcated cist grave.

Table 1. Strontium, carbon, nitrogen and oxygen isotope results for human bones and teeth from San Juan de Momoitio.

Samples	Type of tooh	Sex	Age	Tombs	Sector	%N	$\delta^{15}N$‰	%C	$\delta^{13}C$‰	C/N	$\delta^{18}O_{(V\text{-}MOW)}$	$^{87}Sr/^{86}Sr$	2SE (%)
SJG-1	M2	?	Y	A	4	14.54	10.05	41.54	-15.58	3.34	17.7	0.709333	0.00001
SJG-2		M	Y-A	A	4	13.45	9.25	38.34	-15.28	3.32			
SJG-3		F	Y-A	A	2	14.88	9.36	42.15	-16.45	3.30			
SJG-4	M2	M	Ma	B	2	14.98	9.67	43.45	-18.2	3.38	18.0	0.70880	0.00001
SJG-5	M2	M	Y	B	2	15.27	9.8	43.22	-17.54	3.30	18.5	0.70968	0.00001
SJG-6		M	Y-A	C	2	14.43	9.59	43.10	-19.33	3.50			
SJG-7		M	Ma	A	1	15.69	9.71	44.29	-16.63	3.29			
SJG-8	I1	F	Y-A	B	1	11.39	9.48	32.45	-16.36	3.33	19.1	0.708736	0.00001
SJG-9	M3	F	Y-A	A	1	12.36	9.35	36.87	-14.48	3.48	18.1	0.71001	0.00001
SJG-10	M1	F	Y-A	A	1	10.64	9.54	34.47	-17.16	3.78	18.0	0.708438	0.00002
SJ6-11	M1	F	Y-A	A	1	15.22	9.48	43.71	-15.5	3.34	17.9	0.70894	0.00001
SJ6-12	M2	F	Y-A	A	1	14.59	9.59	41.82	-16.18	3.34	17.2	0.709076	0.00001
SJG-13	M3	M	Ma	C	2	14.79	9.67	43.24	-17.83	3.41	17.0	0.70904	0.00001

Table 1. Continued. Strontium, carbon, nitrogen and oxygen isotope results for human bones and teeth from San Juan de Momoitio.

Samples	Type of tooh	Sex	Age	Tombs	Sector	%N	$\delta^{15}N$‰	%C	$\delta^{13}C$‰	C/N	$\delta^{18}O_{(V\text{-}MOW)}$	$^{87}Sr/^{86}Sr$	2SE (%)
SJG-14		F	Y-A	A	2	13.80	9.71	41.75	-18.05	3.53			
SJG-15	C	M	Y-A	A	2	14.41	9.97	41.31	-16.8	3.34	19.0	0.709318	0.00001
SJG-16	P4	?	Y	A	2	15.16	8.94	43.41	-15.8	3.36	18.7	0.710014	0.00002
SJG-17	M1	?	A	A	2	9.70	9.7	41.30	-16.73	3.37	18.2	0.708425	0.00001
SJG-18		M	A	B	2	9.18	9.18	41.77	-18.22	3.35			
SJG-19	P4	F	Ma	B	2	9.52	9.52	42.18	-16.56	3.42	18.8	0.70924	0.00001
SJG-20	I1	M	Ma	C	2	10.45	10.45	36.64	-18.11	3.43	18.4	0.709728	0.00001
SJG-21		M	Y-A	?	2	14.17	9.41	40.84	-16.76	3.36			
SJ6-22	M1	M	Y-A	B	2	11.56	9.17	-16.41	-16.41	3.43	19.8	0.709525	0.00002
SJG-23		M	Y-A	A	2	13.05	9.56	-16.24	-16.24	3.46			
SJG-24	M3	M	Y-A	B	2	9.68	9.98	-17.11	-17.11	3.51	18.7	0.70874	0.00001
SJG-25		M	Ma	B	4	14.16	9.7	-16.87	-16.87	3.42			
SJG-26	P4	M	Y-A	A	4	14.07	9.15	41.61	-15.94	3.46	19.4	0.70952	0.00001

Table 1. Continued. Strontium, carbon, nitrogen and oxygen isotope results for human bones and teeth from San Juan de Momoitio.

Samples	Type of tooh	Sex	Age	Tombs	Sector	%N	$\delta^{15}N$‰	%C	$\delta^{13}C$‰	C/N	$\delta^{18}O_{(V\text{-}MOW)}$	$^{87}Sr/^{86}Sr$	2SE (%)
SJG-27	M1	M	A	A	4	13.58	9.68	-15.48	-15.48	3.44	18.4	0.70922	0.00002
SJG-28	M2	F	Y-A	B	4	14.25	9.75	-16.56	-16.56	3.41	18.5	0.70887	0.00001
SJG-29	I2	M	Ma	B	4	13.83	9.91	-15.95	-15.95	3.41	18.8	0.708840	0.00002
SJG-30		F	Ma	B	4	11.37	9.37	32.95	-16.40	3.38			
SJG-31	?	?	A	B	4	14.07	9.95	-14.57	-14.57	3.28	18.6	0.709275	0.00002
SJG-32		F	A	B	4	13.50	10.08	39.26	-17.39	3.39			
SJG-33	M3	M	Y-A	B	4	11.08	10.31	33.21	-15.44	3.50	18.3	0.708639	0.00002
SJ6-34	P4	F	A	A	4	13.49	9.49	39.16	-17.02	3.39	17.0	0.708936	0.00002
SJG-35	M3	M	Ma	B	4	11.73	9.81	34.83	-16.89	3.46	16.0	0.70930	0.00001
SJ6-36	M2	?	A	A	4	14.02	9.52	40.19	-15.89	3.36	18.0	0.709461	0.00002
SJG-37		F	Y-A	B	4	15.68	10.15	44.01	-16.22	3.27			
SJ6-38	M2	M	A	A	4	14.61	8.83	42.68	-15.8	3.41	17.8	0.709096	0.00001
SJG-40		F	A	A	4	14.28	9.18	41.67	-15.78	3.40			

Table 1. Continued. Strontium, carbon, nitrogen and oxygen isotope results for human bones and teeth from San Juan de Momoitio.

Samples	Type of tooh	Sex	Age	Tombs	Sector	%N	$\delta^{15}N$‰	%C	$\delta^{13}C$‰	C/N	$\delta^{18}O_{(V\text{-}MOW)}$	$^{87}Sr/^{86}Sr$	2SE (%)
SJG-41	M2	F	A	A	4	14.27	9.33	40.79	-15.98	3.33	18.3	0.70888	0.00001
SJG-42		F	A	B	3	15.66	8.99	-17.63	-17.63	3.34			
SJ6-43	M2	F	Y-A	B	2	13.12	10	-17.67	-17.67	3.40	17.0	0.70861	0.00001
SJ6-44	M1	M	Y-A	?	2	13.55	9.53	-15.01	-15.01	3.39	18.6	0.709654	0.00001
SJG-45		?	Y-A	B	2	13.31	10.52	-15.73	-15.73	3.37			
SJG-46	M2	?	Y	?	2	15.91	9.71	44.62	-17.14	3.28	18.4	0.709300	0.00002
SJG-47		F	Y-A	B	2	10.21	10.21	38.24	-16.12	3.22			
SJG-48		?	A	A	2	10.17	10.17	35.88	-16.56	3.23			
SJG-49	P4	F	Y-A	A	2						18.5	0.709456	0.00001
SJG-50		?	Y	C	2	10.28	10.28	36.85	-19.2	3.59			
SJG-51	M1	?	Y-A	A	2	14.63	10.31	42.19	-17.9	3.35	18.6	0.708843	0.00001
SJG-52		?	A	A	2	6.05	10.57	21.46	-17.52	4.14			
SJ6-53	M1	?	Y-A	A	5	13.93	10.01	40.22	-18.12	3.37	18.5	0.708825	0.00001

Table 1. Continued. Strontium, carbon, nitrogen and oxygen isotope results for human bones and teeth from San Juan de Momoitio.

Samples	Type of tooh	Sex	Age	Tombs	Sector	%N	$\delta^{15}N$‰	%C	$\delta^{13}C$‰	C/N	$\delta^{18}O_{(V-MOW)}$	$^{87}Sr/^{86}Sr$	2SE (%)
SJG-55		M	A	A	5	12.84	10.45	37.72	-15.78	3.43			
SJG-56		?	?	?	?	5.17	10.11	16.49	-17.50	3.72			
SJG-57		?	?	?	?	12.05	11.27	34.83	-18.54	3.34			
SJG-61		F	A	?	?	8.33	9.37	-17.72	-17.72	3.67			
SJG-62		M	A	?	?	3.40	9.63	-18.93	-18.93	4.52			
SJG-64		?	Y-A	B	3	14.92	10.4	-16.53	-17.72	3.32			
SJG-65		?	Y-A	B	3	13.04	10.03	39.20	-16.48	3.50			
SJG-66	M1	F	Y-A	C	3	10.57	10.57	34.49	-17.1	3.60	18.2	0.709515	0.00001
SJ6-72	C	?	I	B	1	12.78	10.78	34.66	-14.38	3.37	20.2	0.708971	0.00001
SJG-73	M2	?	I	A	1	12.06	10.06	29.93	-15.03	3.56	19.4	0.70894	0.00001
SJG-74	M1	?	I	A	1	9.58	9.58	34.87	-16.56	3.342	19.3	0.708605	0.00002
SJ6-75	M2	?	I	B	1	10.42	12.99	30.56	-17.11	3.47	17.0	0.708497	0.00002
SJG-77	M2	?	I	B	1	13.82	9.86	39.46	-16.7	3.33	17.5	0.708563	0.00001

Table 1. Continued. Strontium, carbon, nitrogen and oxygen isotope results for human bones and teeth from San Juan de Momoitio.

Samples	Type of tooh	Sex	Age	Tombs	Sector	%N	$\delta^{15}N$‰	%C	$\delta^{13}C$‰	C/N	$\delta^{18}O_{(V\text{-}MOW)}$	$^{87}Sr/^{86}Sr$	2SE (%)
SJG-78	M1	?	I	?	?	15.55	12.68	43.42	-13.3	3.26	18.9	0.708982	0.00001
SJ6-80	M1	?	I	?	2	5.21	9.48	16.22	-18.67	3.65	19.7	0.708521	0.00002
SJ6-81	M2	?	I	B	2	14.28	9.95	40.30	-18.1	3.29	20.2	0.70899	0.00001
SJG-82	M2	?	III	A	2	14.82	8.92	42.03	-18.56	3.31	18.4	0.708680	0.00001
SJ6-83	P3	?	Iii	A	2	15.65	9.44	43.18	-14.41	3.22	17.2	0.709398	0.00001
SJ6-84	M2	?	I	A	2	14.70	9.85	41.05	-16.94	3.26	19.6	0.708372	0.00001
SJ6-85	M2	?	I	A	2	13.16	10.13	37.28	-16.18	3.24	17.2	0.708347	0.00001
SJ6-87	M1	?	I	A	2	14.99	11.43	41.98	-16.87	3.26	19.8	0.708658	0.00001
SJ6-88	P3	?	I	C	2	12.78	9.48	35.77	-17.23	3.27	19.9	0.709422	0.00001
SJ6-89	M2	?	I	A	2	9.85	10.75	28.42	-17.77	3.37	18.2	0.708739	0.00002
SJG-90	M1	?	Iii	C	2	14.86	9.49	41.54	-15.45	3.25	18.6	0.708837	0.00001
SJ6-91	C	?	Iii	A	2	14.03	9.23	40.07	-16.13	3.33	18.8	0.708656	0.00002
SJ6-92	I2	?	I	A	2	12.89	13	36.13	-19.11	3.27	19.4	0.708298	0.00002

Table 1. Continued. Strontium, carbon, nitrogen and oxygen isotope results for human bones and teeth from San Juan de Momoitio.

Samples	Type of tooh	Sex	Age	Tombs	Sector	%N	δ^{15}N‰	%C	δ^{13}C‰	C/N	$\delta^{18}O_{(V-MOW)}$	$^{87}Sr/^{86}Sr$	2SE (%)
SJ6-93	M1	?	I	A	2	13.88	11.34	38.73	-17.65	3.26	19.6	0.708743	0.00001
SJ6-94	M2	?	I	?	2	8.29	10.55	25.22	-18.64	3.55	18.9	0.708977	0.00001
SJ6-95	I2	?	I	A	2	14.02	8.89	39.78	-17.03	3.33	20.4	0.708634	0.00001
SJG-96	M2	?	I	B	2	13.42	9.25	37.85	-17.28	3.29	18.8	0.708837	0.00001
SJG-97	M2	?	Iii	A	2	13.08	9.6	37.40	-16.64	3.34	18.7	0.70862	0.00001
SJG-98	M2	?	I	C	3	7.80	12.46	26.57	-18.56	3.97	18.0	0.708564	0.00001
SJG-99	M2	?	I	C	3	12.11	13.12	37.51	-16.00	3.61	19.2	0.708295	0.00002
SJG-100		?	I	B	4	15.61	8.84	44.24	-18.42	3.32			
SJG 101	M1	?	I	B	4	13.99	8.43	40.84	-16.55	3.41		0.708631	0.00001
SJG-102	I1	?	I	D	4	10.04	11.6	30.33	-17.12	3.52		0.708244	0.00001
SJG-103		?	I	A	4	14.80	9.29	42.17	-16.24	3.32			
SJG-104	M2	?	I	A	4	15.47	8.5	42.66	-14.31	3.22	18.0	0.70868	0.00002
SJG-106	I2	?	Iii	?	4						18.2	0.708611	0.00001

Table 1. Continued. Strontium, carbon, nitrogen and oxygen isotope results for human bones and teeth from San Juan de Momoitio.

Samples	Type of tooh	Sex	Age	Tombs	Sector	%N	δ^{15}N‰	%C	δ^{13}C‰	C/N	$\delta^{18}O_{(V\text{-}MOW)}$	$^{87}Sr/^{86}Sr$	2SE (%)
209		?	I	?	?	14.91	7.85	42.28	-19.51	3.28			
211		?	I	?	?	8.38	11.67	23.98	-18.78	3.34			
Ent.4210		?	?	?	?	16.55	10.78	44.95	-18.38	3.17			

C: canine: I1: incisor 1; I2: incisor 2; M1: molar 1 M2: molar 2 M3: molar 3; P3:premolar3 P4:premolar4 Indeterminate; M: Male. F: Female; Y: young. Y-A: Young adult; I: Infant I; Iii: Infant II A: Adult; Ma: Mature A: earth graves; B: partially demarcated cists graves; C: stone-lined graves.

Materials and Methods

Ninety-three human bones from San Juan de Momoitio site were analyzed for dietary studies by means of carbon and nitrogen isotopes and 63 teeth samples were analyzed for migration pattern studies using strontium and oxygen isotopes. Besides, 10 faunal bone samples were analyzed to establish the carbon and nitrogen isotope baseline. In order to define the strontium isotope baseline, 6 fauna remains, 2 soil and 2 freshwater samples from the area around the graveyard were considered. The soil samples were collected in different parts of the cemetery and the freshwater samples were taken from the streams near Momoitio. Surface waters were collected from the banks of the rivers and, previous to analysis, water samples were filtered to remove suspended particles.

The measured individuals corresponded to 20 females, 23 males and 50 of indeterminate sex (Arenal Fernandez, 1992). The individuals were categorized by age into infant I (0-7 years), infant II (8-12 years), juveniles (14-20 years), young adults (20-40 years), middle-aged adults (40-60 years), and older adults (60 years). Sex determination was carried out according to the classical patterns of dimorphism (W.E.A., 1980) and age was defined by changes in epiphyseal closure, cranial sutures and dental eruption (Eguía, 1982; Brothwell, 1981; Perizonius and Pot, 1981; W.E.A., 1980).

The dietary studies were performed in bone collagen. The selected materials were, if possible, ribs and long bone fragments. Bones were washed in an ultrasonic bath for 30 min in distilled water repeatedly and rinsed in ultrapure water in order to remove impurities from the burial.

Bone collagen was extracted using the procedure of Bocherens et al. (1991). 300 mg bone was demineralised in 1 ml of HCl for 20 minutes at room temperature until dissolved. The samples were rinsed with distilled water and 5ml NaOH (0.125N) was added to remove humic acids. Then they were rinsed again with distilled water and gelatinized in a pH 3 HCl solution for 17 h at 90ºC. The filtered supernatant containing the soluble collagen then was collected, frozen and lyophilized.

Collagen (2.5-3.5 mg) was loaded into a tin capsule for continuous flow combustion and isotopic analysis. The carbon and nitrogen isotopes were analyzed by an Elemental Analysis- Isotopic Ratios Mass Spectrometer (EA-IRMS) at Iso-Analytical (Cheshire, UK).

Multiple analyses of NBS-1577B standard and IA-R045 ammonium sulphate were performed for quality control during the analysis of the samples. The replicas of the NBS-1577B standard results have δ^{13}CV-PDB = -21.61 ‰ ± 0.05 (s, n = 18) and δ^{15}NAir = 7.61 ‰ ± 0.12 (s, n = 18).

Water aliquot was filtered with disposable syringe filters (0.45µm), then 10 ml aliquot was transferred to a 15 ml Teflon (Savillex™) vial and evaporated down on a hot plate at 80 ºC overnight and then dissolved in 1.5 ml of 2N HNO_3.

Bones and tooth enamel were analyzed to determine strontium and oxygen isotope composition and establish the individuals' provenance. First, the samples were washed in an ultrasonic bath to remove impurities and further cleaned by mechanical abrasion to remove the outer surface to avoid contamination.

In particular, for strontium isotope analysis a fraction of dental enamel was collected mechanically with diamond-coated trepanation drill (MF-perfect, W & H Dentalwork, Bürmoos, Austria). The enamel sample was taken transversally to the tooth. Enamel and bone samples (~10 mg) were dissolved in 7 ml Savillex® vials (Minnetonka, MN, USA) with 1.5 ml of 2N HNO_3 (analytical grade purified by subboiling distillation). To establish the local isotopic composition, two freshwater samples and four soil samples from areas around the settlement were also analyzed. 15 ml of freshwater was evaporated to dryness and then dissolved in 2 ml HNO_3, while 1g aliquot soil sample was leached by adding 2.5 ml 1 M ammonium nitrate (NH_4NO_3) and shaking for 8 h to obtained the bioavailable Sr. After samples were centrifuged at 3000 rpm for 15 min, the supernatant was extracted (~ 1-2 ml) and evaporated to dryness and then redissolved in 2 ml HNO_3. The solutions were loaded into cation exchange columns filled with Sr.spec® (ElChroM industries, Dariel, IL, USA), a strontium selective resin. The resin was used once to elute the sample and then discarded. Strontium procedural blanks were less than 100 pg and hence provided a negligible contribution.

The strontium isotopes were analyzed on a Neptune multi-collector inductively coupled plasma mass spectrometer (MC-ICP-MS) at the Advanced Research Facilities (SGIker) of the University of the Basque Country (UPV/EHU). $^{87}Sr/^{86}Sr$ measurements were corrected for krypton (Kr) and rubidium (Rb) interferences and normalized for instrumental mass bias using $^{87}Sr/^{86}Sr = 8.375209$. Repeated analyses of NIST SRM-987 international standard yielded a value of $^{87}Sr/^{86}Sr = 0.710262 \pm 0.000026$ (2σ, n = 3). Long-term $^{87}Sr/^{86}Sr$ value, determined over a twenty-two month period, was 0.710266 ± 0.000021 (2σ, n = 47).

Tooth enamel was also analyzed for oxygen isotope analysis following the procedure described by Stephan (2000). 60 mg of dental enamel powder was used. The organic matter was removed with a solution of 2.5% NaOCl for 24 h at room temperature, then 48 h treatment in 0.125M NaOH also at room temperature. The hydroxyapatite powder without organic matter was dissolved in 2 ml of HF for 24 h. The phosphate solution and the residue composed of CaF_2 were separated by centrifugation, pipetted into a 100 ml glass tube and neutralized with 3 ml 2M KOH. Silver phosphate (Ag_3PO_4) was precipitated by adding 30 ml of a buffered silver amide solution (0.2 M $AgNO_3$; 1.16 M NH_4NO_3; 0.75 M NH_4OH) gradually warmed up to 70 ºC and holding the temperature for 5-6 h, then cooling down slowly. Silver phosphate crystals were filtered on a weighed 0.2µm filter and washed several times with double distilled water, and dried at 50 ºC for 1-2 h. Subsequently, 0.3 mg of Ag_3PO_4 was mixed with 0.5-1 mg of AgCl and 0.3 mg of graphite in silver capsules. The capsules were transferred into the autosampler carousel of the Temperature Conversion Elemental Analyser (TCEA) and degassed for 30 minutes at 80 ºC in a vacuum. The oxygen isotope analyses were carried out on a Thermo Finnigan TCEA coupled to a Delta Plus XP Spectrometer at the University of Parma. Isotopic compositions were given in the conventional δ-notation relative to V-SMOW (Vienna-Standard Mean Ocean Water). The V-SMOW scale normalization was based on four replicated international reference materials provided by the International Atomic Energy Agency (IAEA): IAEA-601, IAEA-602, IAEA-CH6, and IAEA-SO-6. The analytical precision of a single determination was better than ±0.4‰.

In addition, two statistical techniques were used to identify outliers in $\delta^{18}O_{PO4}$ within the San Juan Momoitio population. Boundaries of intra-sample variation based on two measurements of scales were defined: ± 2 standard deviation (2SD) from the mean and Tuke's inter-quartile range method (IQR) considering 1.5xIQR and 3xIQR (Lightfoot and O'Connell, 2016). Parametric statistics were used to describe isotope distribution and compare isotope values between sample groups. Differences between groups were observed by applying an unpaired Student's t test. Statistical significance was accepted as $p < 0.05$. Statistical analysis was processed with SPSS v.20 (Statistical package for Social Sciences).

Results

Carbon and nitrogen isotope ratios in bone collagen

Table 1 summarizes the carbon and nitrogen isotope composition of 93 individuals' bones from San Juan de Momoitio (Biscay, Spain). Sample SJG-71 was excluded because it did not contain enough collagen. Most individuals showed well-preserved collagen yielding a C/N of 3.2 and 3.6 (Ambrose, 1990; DeNiro, 1985), only 10 samples were discarded from the discussion (SJG-10, SJG- 52, SJG-56, SJG-61, SJG-62, SJG-71, SJG-80, SJG-98 SJG-99, Ent.4 and 210) because C/N>3.6. Thus, a total 82 individuals were considered for the paleodiet study.

Overall, the mean values of nitrogen and carbon isotopic composition were +9.7‰ ± 0.9 (1σ) and -16.7‰ ± 1.3 (1σ) respectively. The range of $\delta^{15}N$ varied from +7.9‰ to +13.0‰ and $\delta^{13}C$ values ranged from -19.5‰ to -13.3‰. Bone stable isotope values are plotted in Fig. 4 and separated into gender and age groups. The $\delta^{15}N$ and $\delta^{13}C$ values did not show significant statistical differences between genders or by ages of individuals. Infants showed pronounced dispersion of nitrogen isotopes values with a mean $\delta^{15}N$ value of +10.1‰ ± 1.4 (1σ) and a range of +7.9‰ to +13.0‰. Table 1 and Fig. 4 show also the faunal $\delta^{13}C$ and $\delta^{15}N$ dataset. Faunal sample $\delta^{15}N$ and $\delta^{13}C$ mean values ranged from +4.7‰ to +8.9‰ and from -21.9‰ to -20.1‰, respectively.

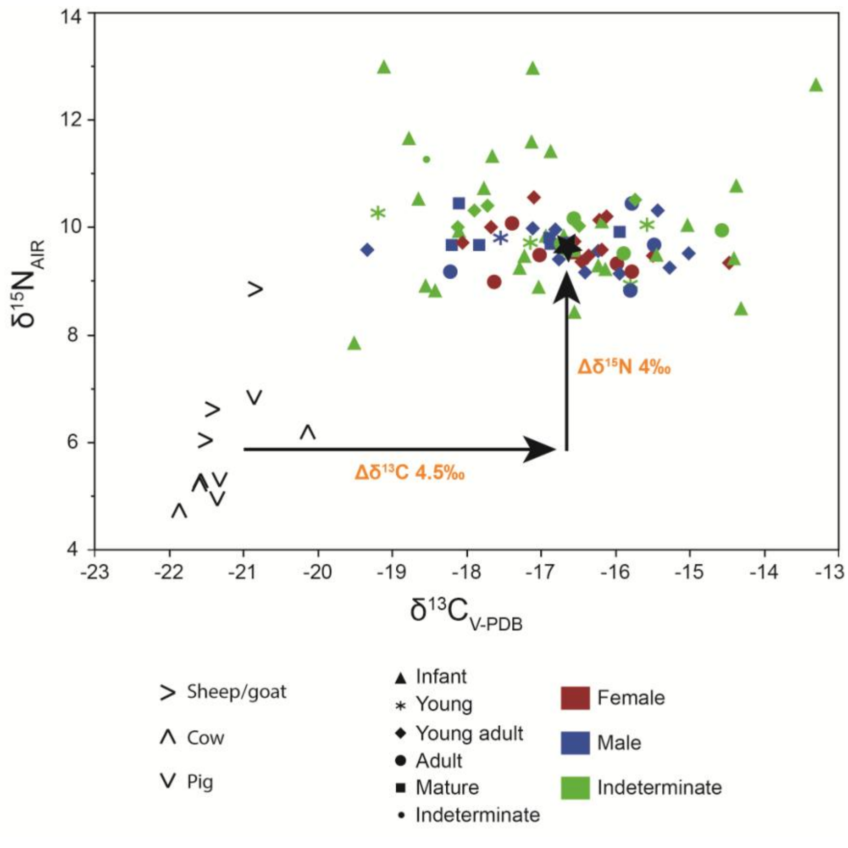

Figure 4. Human and fauna carbon and nitrogen isotope values from San Juan de Momoitio. The star symbol shows the mean value.

In order to assess if the burial phases reflected time-related differences and if grave types reflected social status, the infant individuals were excluded from the statistical analysis.

Likewise, when first occupation phase (9th-11th centuries) was compared with second occupation phase (12th century, High Middle Ages), only $\delta^{13}C$ showed significant statistical differences (p=0.004), the mean values are -17.0 ± 1.1 (1σ, n=29) and -16.1 ± 0.7 (1σ, n=18) respectively (Fig. 5).

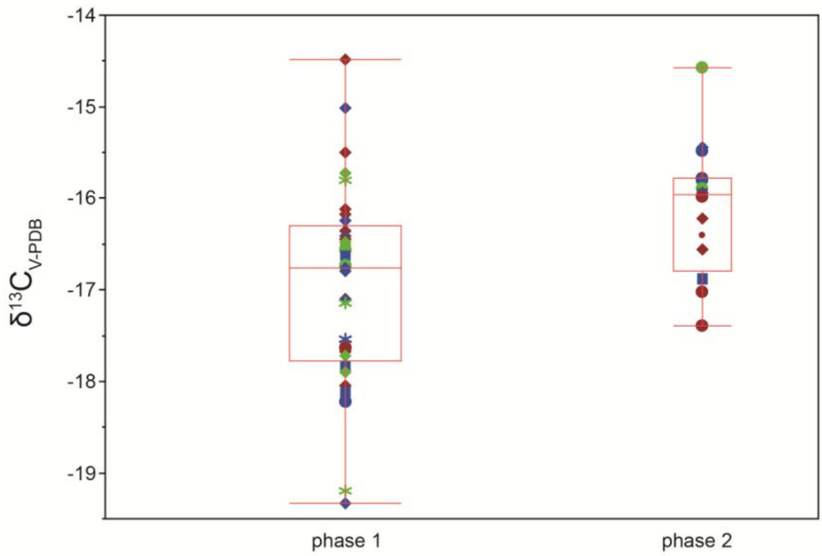

Figure 5. Box plot of $\delta^{13}C$ and $\delta^{15}N$ values by phases in San Juan de Momoitio graveyards.

Within the first occupation phase of the graveyard (9th-11th centuries, Early Middle Ages), individuals from Sectors 3 and 5 were not taken into consideration because suitable cases were not enough for statistical analysis. Between individuals from Sectors 1 and 2, significant statistical differences existed in $\delta^{13}C$ (p=0.003). The $\delta^{13}C$ mean value of individuals from Sector 1 was -15.8 ± 0.9 (1σ, n=5) compared to -17.1 ± 1.1 (1σ, n=24) for Sector 2 individuals.

Only in Sectors 2 and 4 statistical analyses were performed to observe differences within types of graves. Comparing carbon and nitrogen composition between individuals of different grave types of sector 2, statistical differences existed for mean $\delta^{13}C$ between individuals buried in earth graves (A) and graves delimited partially by cists (B) in comparison with those buried in stone-lined graves (C) (p=0.002 and p=0.004, respectively). The mean $\delta^{13}C$ value for earth graves (A) was -16.8 ± 0.8 (1σ, n=8), for partially demarcated cist graves (B) -17.1 ± 0.9 (1σ, n=9) and for stone-lined graves (C) -18.6 ± 0.8 (1σ, n=4). Although no statistical differences existed, the highest $\delta^{15}N$ values were for individuals buried in stone-lined graves. The mean values of earth graves and partially demarcated cyst graves were +9.7 ± 0.4 (1σ, n=8) and +9.8 ± 0.5 (1σ, n=9) respectively compared to +10.0 ± 0.4 (1σ, n=4) for stone-lined graves.

In Sector 4 only earth graves and partially demarcated cist graves existed, giving only a statistical difference for $\delta^{15}N$ (p=0.002), with mean values of 9.4 ± 0.4 (1σ, n=9) and 9.9 ± 0.3 (1σ, n=9), respectively.

Strontium and Oxygen ratios in teeth enamel

The results of human strontium isotope analysis are shown in Table 1 while soil, water and faunal strontium isotope data are shown in Table 2. $^{87}Sr/^{86}Sr$ isotope ratios of human dental enamel vary largely, ranging from 0.708244 to 0.710014. Domestic animals show a similar

$^{87}Sr/^{86}Sr$ isotope composition to human samples, ranging from 0.70861 to 0.7102, water values range from 0.708034 to 0.70888, whereas $^{87}Sr/^{86}Sr$ isotope ratio of sediments shows the lowest radiogenic values, ranging from 0.70815 to 0.70816. In order to use strontium isotopes to reconstruct mobility, the local $^{87}Sr/^{86}Sr$ isotope ratio baseline must be established. Water, fauna and soil samples were considered to define local bioavailable strontium isotopic composition. Momoitio water sample strontium isotope ratios show a clear discrepancy. The $^{87}Sr/^{86}Sr$ of water mostly comes from the product of mineral weathering. So the lithology controls the strontium isotope composition of the water. The regional geology in the surroundings of Momoitio is complex and consists of Upper Cretaceous carbonated rocks and Tertiary terrigenous rocks and, within Cretaceous materials, dikes of volcanic basalts (Fig. 6).

The $^{87}Sr/^{86}Sr$ of Momoitio waters reveals the effect of the drainage over the geologically complex terrain. Sample water showing $^{87}Sr/^{86}Sr$ = 0.70888 reflected water draining terrigenous material whereas sample water showing $^{87}Sr/^{86}Sr$ = 0.708034 reflected volcanic basalts exhibiting radiogenic strontium isotope values between 0.7038 and 0.7054 (Rossy et al., 1992). Soil $^{87}Sr/^{86}Sr$ values reflected the lithology underlying in Momoitio and allowed the lower limit of the local baseline to be established.

Since water and soil radiogenic strontium isotope values are influenced by the bedrock lithology, in archaeology a common procedure to establish the local isotope composition is to analyze local archaeofauna (Price et al., 2002; Slovak and Paytan, 2011). Archaeological fauna samples fed locally in the same area as the humans (Bentley, 2006; Price et al., 2002), resulting indicative of local isotope signature. However, the use of livestock as indicative of the local baseline has been subjected to debate since domestic animals can undergo the same mobility patterns as humans (Knudson et al., 2012; Shaw et al., 2009). In the case of Momoitio, except two samples, most of the archaeofauna exhibited $^{87}Sr/^{86}Sr$ values of 0.7092 and this value was considered to establish the local signature upper limit. So only one sample with $^{87}Sr/^{86}Sr$ = 0.7102 can be considered to be outside the local radiogenic strontium isotope range.

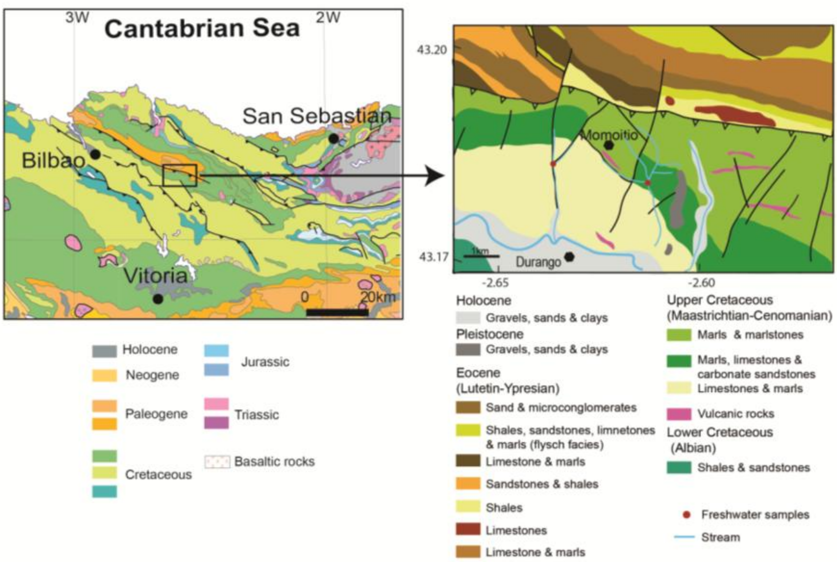

Figure 6. Geological map of San Juan de Momoitio surrounding area and the location of analyzed freshwater samples (modified from Rodríguez Fernández et al., 2015; http://igme.maps.arcgis.com/home/webmap/viewer.html?webmap=44df600f5c6241b59edb596f54388ae4).

Altogether, water, soil and archaeofauna were considered to determine the local bioavailable strontium isotopic composition that was established between 0.7082 and 0.7092 (Fig. 7). The geological complexity of Momoitio and the underlying bedrock nature determined the wide range of the local strontium isotope signature.

Table 2. Strontium isotope results for fauna, freshwater and soil samples from San Juan de Momotio.

Samples	Material	$^{87}Sr/^{86}Sr$	2SE (%)
MS1	soil	0.70816	0.00001
MS4	soil	0.70815	0.00001
MA	freshwater	0.70888	0.00002
M2A	freshwater	0.708034	0.00002
S11178	Cow	0.70918	0.00002
S131149	Pig	0.70916	0.00001
X1911100	Pig	0.71020	0.00001
ZY21116	Cow	0.70861	0.00001
A131183	Ovi/capra	0.709224	0.00002
S171170	Ovi/capra	0.709160	0.00001

As regards of oxygen isotope analyzes, enamel phosphate oxygen ratios of human individuals ($\delta^{18}O_p$) showed a broad value range from 16.0‰ to 20.4‰ (total range: Δ4.4‰). The highest value was observed in an infant and the lowest value in a mature male. The mean $\delta^{18}O_p$ value for infants (n=24) was 18.8±1.0 ‰ (1σ) and for adults (n=33) it was 18.2±0.8 ‰ (1σ). The mean $\delta^{18}O_p$ value for infant I (n=16) was 19±1.1 ‰ (1σ), for infant II (n=8) it was 18.6 ±0.8‰ (1σ). The mean $\delta^{18}O_p$ value for the juveniles (n=4) was 18.3±0.4‰ (1σ), for the young adults (n=16) (1σ) 18.5±0.7‰, for the middle-aged adult (n=7) 18.0±0.5‰ (1σ), and for the older adults (n=6) 17.8±1.1‰ (1σ). Tooth enamel phosphate values for infants range from 17.0‰ to 20.4‰.

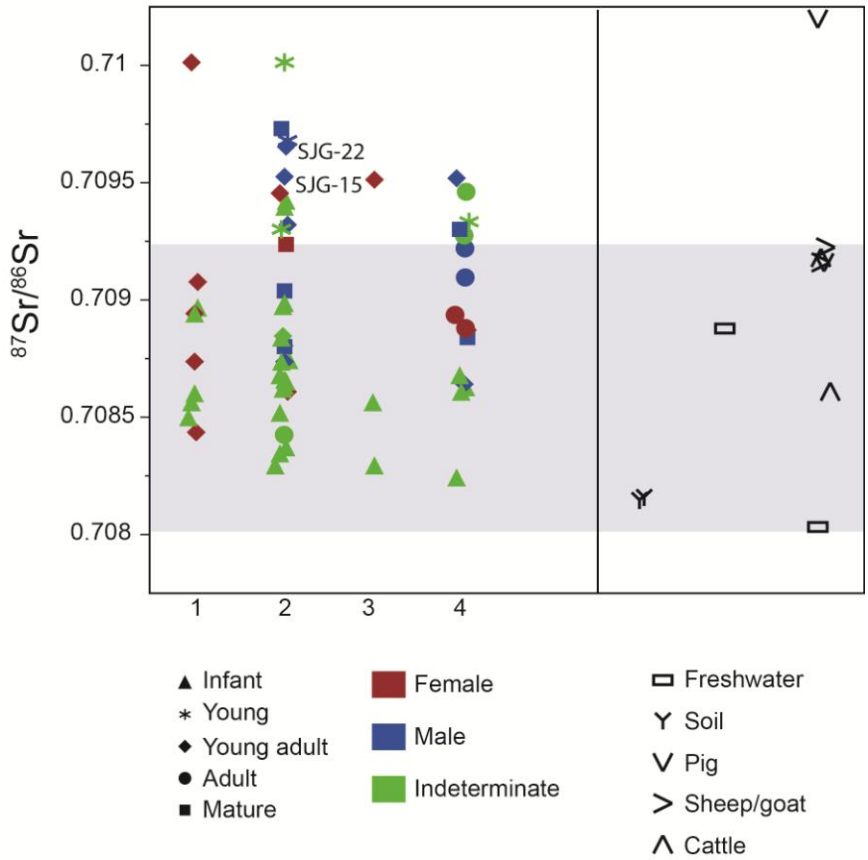

Figure 7. $^{87}Sr/^{86}Sr$ composition of human enamel by sector, domestic animals (cow, pig and sheep/goat) samples, water samples and soil samples. Grey area showed the baseline in the surrounding area of San Juan de Momoitio defined by local freshwater, fauna and soil samples.

Momoitio population $\delta^{18}O_{dw}$ values were calculated using the available phosphate/drinking water equations (Daux et al., 2008; Iacumin and Venturelli, 2015; Levinson and Kolodny 1987; Luz et al., 1984; White et al., 1998) and compared with expected local water values derived from IAEA/WISER data set. The equation by Iacumin and Venturelli (2015) was used to estimate the drinking water isotope value. To establish the local oxygen isotope composition, Santander meteorological station was selected because it is located closest and in the same latitude as the Momoitio area and shows similar seasonal characteristics in precipitation, temperature and humidity. The $\delta^{18}O_{dw}$ of local meteoric water considering data from Santander station, is -5.2‰ (average values from 2000 to 2010) [http://www.iaea.org/water]. The calculated $\delta^{18}O_{dw}$ of individuals from Momoitio ranged from -4.0‰ to -10.0‰ ±2.5‰. To determine the outliers, 1.5xIQR and 2SD statistical methods were performed.

Discussion

Dietary patterns

The $\delta^{13}C$ and $\delta^{15}N$ values showed an omnivorous diet based on mixed C3 plants such as wheat and vegetables and C4 plants such as millet and, some protein derived from animal husbandry and agriculture. The isotope values for both adult males (n=4) (mean $\delta^{13}C$ = -16.7 ± 1.1‰, mean $\delta^{15}N$ = 9.7 ± 0.4‰) and females (n=5) (mean $\delta^{13}C$ = -16.5 ± 0.9‰, mean $\delta^{15}N$ = 9.6 ± 0.5‰) indicated there were no gender restrictions in the diet or in the access to animal protein, contrary to what is observed in other coeval Christian and Muslim communities (Alexander et al., 2015; Quiros-Castillo, 2013).

Compared to the faunal baseline, the variation in $\delta^{15}N$ of adult human individuals ($\Delta\delta^{15}N$ = 4‰) was consistent with one trophic level enrichment (Drucker and Bocherens, 2004; Schoeninger and Moore, 1992). However, not all individuals showed $\delta^{15}N$ one trophic level enrichment, infant individuals showed higher $\delta^{15}N$ values compared with females, which can be attributed to the nursing effect (Fuller et al., 2005; Richards et al., 2002). The magnitude of nitrogen isotope enrichment is not only able to determine breastfeeding and weaning practices but also to explore weaning practices and infant mortality (Katzenberg et al., 1996; Pearson et al., 2010; Schurr, 1997; Williams et al., 2005). The $\delta^{15}N$ values higher than 10.7‰ indicate nursed infants and correspond to children younger than two to three years old, whereas the lowest values 8.4‰ indicate post-weaned infants approximately older than seven years (Infants II type). Most infants showed $\delta^{15}N$ values similar to the mean values for adults, indicating they were fully weaned with a similar diet to adults. The unusually high number of infants in Momoitio graveyard, corresponding to 35% of the studied individuals, is noteworthy and suggests high infant mortality. Establishing infant mortality causes by skeletal remains is difficult because some illnesses do not lead to a skeletal response. Few studies detect relationship between weaning behaviors and infant survival (Katzenberg et al., 1996; Katzenberg and Lovell, 1999; Mays et al., 2002; Pearson et al., 2010). Within Momoitio infants, more than a half showed $\delta^{15}N$ values similar to mean values for young adult females, indicating weaning food consumption. The introduction of solid food, possibly plant-based, and the lack of breast milk causes disease and nutritional stress as result of nutritional deficiency. Weaning practice was probably a key factor in the peak in infant mortality in Momoitio between children of two to seven years.

Contrary to the nitrogen isotope values, carbon exhibited an offset of $\Delta\delta^{13}C$ = 4.5‰, unusually large by more than 2‰ over the herbivore baseline. The offset indicates that the base of the diet was different for animals and humans. Humans ate also C_4 plants in different proportions as indicated by the $\delta^{13}C$ values higher than -18‰. Archaeobotanical evidences in Momoitio indicated the cultivation of millet as C4 cereal (Le Huray and Schutkowski, 2005; Le Huray et al. 2006). The consumption of a small amount of marine food is evidenced by the discovery of *Ostrea* shellfish remains in the archaeological site (Castaños, 1992). This fact was not surprising because Momoitio is located about 40 km from the Bay of Biscay coast and marine sources were reasonably accessed by the population.

This result opened another possible interpretation regarding the identification of differences within the community based on funerary rituals. Historically, studies of medieval graveyards showed great diversity of funerary practices and changes in the typologies of graves over time were observed. The typology of grave might reflect social stratification or could be related to family traditions and religious beliefs (Vauchez, 1985). Individuals at Momoitio showed differences in diet according to the type of grave. Partially demarcated cist graves (B) of sector 2 show lower $\delta^{13}C$ values in comparison to the other grave types. Lower $\delta^{13}C$ values means smaller quantities of millet in diet. Millet (C4 crop) was generally regarded as a low-status foodstuff by medieval society, particularly in rural settlements (Adamson, 2002; Braudel, 1972; Glick, 1982; Quiros Castillo, 2013; Sarasa, 1995).

Although it was not possible to determine the ages of each burial with radiocarbon dating, the chronological distribution of the burial periods (broadly Early Middle Ages and High Middle Ages) was considered to investigate a possible shift in diet through time. Individuals from the first burial period, that includes Sectors 1 to 3 (Early Middle Ages), showed a large spread in $\delta^{13}C$ values, ranging from -14.5‰ to -

19.3‰, whereas individuals from the last period (High Middle Ages, Sector 4), showed $\delta^{13}C$ values ranging from -14.6‰ to -17.4‰ (Fig. 5). However, within the first burial period (9th-11th centuries), dietary differences were observed between individuals from Sector 1 and Sector 2. The $\delta^{13}C$ values of individuals in Sector 1 were more enriched (mean values -15.8±0.9‰) compared with individuals in Sector 2 (mean values -17.1±1.1‰) indicating dietary changes over time. Such shift in diet consisted in lower millet consumption by Sector 1 in comparison to Sector 2, indicating, probably, access to a wider variety of food resources. A shift in diet was also observed between Sector 2 (Early Middle Ages) and Sector 4 (High Middle Ages), concerning an increase in millet consumption. This change in diet can be related to crops specialization. The lack of radiocarbon dates does not allow us to go back to the chronology of these changes.

Data from other coetaneous medieval populations in the northern Iberian Peninsula were considered to illustrate particular differences and similarities in diet (Treviño, Zaballa, Aistra and Zornoztegi populations; Quiros Castillo, 2013) (Fig. 8).

Note that the local isotope baselines exhibited different mean values due to regional environmental differences. In all cases, the $\delta^{15}N$ value differences can be explained by trophic level enrichment. However, the higher values of carbon isotope composition of Momoitio individuals (on average, -16.8±1.3‰) are significantly different from those at other sites and attributed to greater millet consumption.

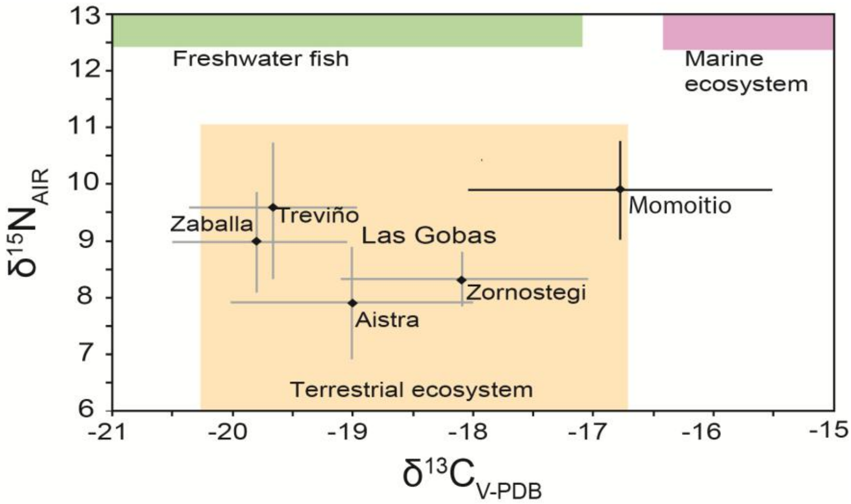

Figure 8. Plot of $\delta^{13}C$ and $\delta^{15}N$ values (mean and SD) from San Juan de Momoitio and coeval settlements in the Basque Country (Aistra, Zornostegi, Zaballa, Treviño) (Quiros Castillo, 2013). Ecosystem boxes are based on faunal data reported by Ambrose (1990) and Müldner and Richards (2007).

Mobility: $\delta^{18}O$ and $^{87}Sr/^{86}Sr$

According to the defined strontium local baseline, two groups of individuals in the Momoitio population were observed: first showing $^{87}Sr/^{86}Sr$ values consistent with local origin (the majority of the individuals) and the second one plotting outside the estimated local composition consisting mainly of young adults. Except two children, most infants were of local origin and exhibited less radiogenic $^{87}Sr/^{86}Sr$ values compared with most local adult individuals. Although enamel is less likely to be affected by diagenesis and environmental conditions (e.g. Khon and Cerling, 2002; Trueman and Tuross, 2002), infant $^{87}Sr/^{86}Sr$ values suggested that deciduous teeth are easier to alter during burial. In fact, rare earth elements U and Th content in milk teeth are sensitive indicators of diagenesis (Guede et al., 2017).

The oxygen isotopic composition also allowed residential mobility to be distinguished. Infant individuals' $\delta^{18}O_p$ values were higher than adult individuals' $\delta^{18}O_p$ values and differences were statistically significant (t-test, $p<0.01$). Deciduous teeth mineralised in-utero and during breastfeeding and record the isotopic milk signal which is enriched in ^{18}O compared to that of the water. To consider intra-sample variation in $\delta^{18}O_p$, infant individuals were excluded from the statistical analysis.

Adult individuals $\delta^{18}O_p$ values show a large variation, suggesting the Momoitio population recorded different climatic conditions. Only few outliers show $\delta^{18}O_p$ values that likely indicate a non-local origin (Fig. 9). The variations observed among the oxygen isotope ratios may be due to temporal climate differences and the characteristics of the Momoitio rural landscape. In fact, the occupation period at Momoitio lasted three centuries and coincided with the Medieval Warm Period (MWP) followed by a cooler period (Diaz and Hughes, 1994; Grove and Switsur, 1994; Hunt, 2006). Besides, the rural structures in the Momoitio region were conditioned by the topographical features of the environment, formed by narrow valleys and hills and consisted of a disaggregated landscape with small and dispersed farmsteads.

Comparing Sector 2 as representative of the first occupation period and Sector 4 of the last occupation period, the $\delta^{18}O_p$ values variation indicated warmer climatic conditions during the first occupation period coinciding with MWP and lower $\delta^{18}O_p$ values in Sector 4, suggesting colder climate conditions.

An outlier (SJG-22) in Sector 2 exhibited higher $\delta^{18}O_p$ values than the normal adult range, suggesting a coastal origin. Three outliers with lower $\delta^{18}O_p$ values suggest an origin from colder or a higher altitude area (Fig. 9).

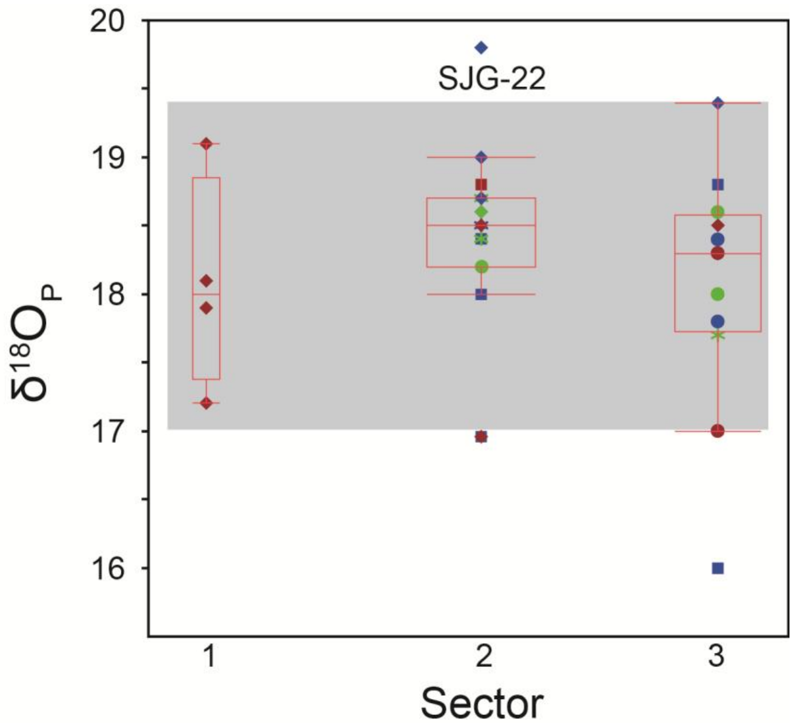

Figure 9. $\delta^{18}O$ values of human enamel by sector. Grey area shows local individuals, individuals with $\delta^{18}O$ values lower than Grey area values were from a colder or higher latitude area while SJG-22 individual was from a warmer or coastal region.

It is worth mentioning the large variation of $\delta^{18}O_p$ values of infant individuals. Infants showing $\delta^{18}O_p$ values inside of current estimate for a local range are considered local origin. However infants outside of the local origin range are difficult to consider as non local individuals since are not consistent with the non-local adults number.

Besides studying mobility of individual's oxygen isotope analysis also have been applied to explore breastfeeding and weaning practices in archaeological populations (Wright and Schwarcz 1998, 1999; Britton et al., 2015). These studies relies on the breast milk, due to isotopic fractionation, will be isotopically enriched compared to the water ingests by the mother (Bryant and Froelich 1995; Kohn et al. 1996; Wright and Schwarcz 1998; Lin et al. 2003). Thus, $\delta^{18}O_p$ high values of Momoitio infants could reflect the breastfeeding effect (Wright and Schwarcs, 1998, 1999), whereas $\delta^{18}O_p$ low values just under the local range could suggest weaning practice. While $\delta^{18}O_p$ values within the normal and just over the local range could be attributed to perinate/neonate individuals.

However, this alternative explain should be taken with caution since metabolic differences associated with ill health, extensive intra-annual climatic variability and birth seasonality could potentially influence oxygen isotope values in infants.

When comparing $\delta^{18}O_p$ with $^{87}Sr/^{86}Sr$ ratios, nearly half the Momoitio population was of non-local origin, including two infants. Within non-local individuals, half of them moved during their early life stage as they correspond to juveniles. However, not all non-local individuals originated from the same geological and geographical areas. Individuals outside the local $^{87}Sr/^{86}Sr$ signature show $\delta^{18}O_p$ values within the local range, which indicates a different geological provenance area but similar climatic conditions. The entire coastline in the north of the Iberian Peninsula and south-western France along the Atlantic Ocean (Aquitaine region) exhibits similar climatic conditions to the Momoitio area. So it is difficult to determinate the origin of this group of non-local individuals. Non-local individuals with $\delta^{18}O_p$ values lower than local range but with the local strontium signature were from a similar area geologically, but from a higher altitude than the Momoitio local population. Considering the geographic characteristic of the region, the isotope values seems to be consistent with an origin from a mountainous area that was likely be quite near the Momoitio region

Chapter IV:

Tauste site

"Isotope analyses to explore diet and mobility in a medieval Muslim population at Tauste (NE Spain)"

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Abstract

The Islamic necropolis discovered in Tauste (Zaragoza, Spain) is the only evidence that a large Muslim community lived in the area between the 8th and 10th centuries. A multi-isotope approach has been used to investigate the mobility and diet of this medieval Muslim population living in a shifting frontier region. Thirty-one individuals were analyzed to determine $\delta^{15}N$, $\delta^{13}C$, $\delta^{18}O$ and $^{87}Sr/^{86}Sr$ composition. A combination of strontium and oxygen isotope analysis indicated that most individuals were of local origin although three females and two males were non-local. The non-local males would be from a warmer zone whereas two of the females would be from a more mountainous geographical region and the third from a geologically-different area. The extremely high $\delta^{15}N$ baseline at Tauste was due to bedrock composition (gypsum and salt). High individual $\delta^{15}N$ values were related to the manuring effect and consumption of fish. Adult males were the most privileged members of society in the medieval Muslim world and, as isotope data reflected, consumed more animal proteins than females and young males.

Introduction

Muslims invaded most of the Iberian Peninsula in the Early Middle Ages (AD 711) and remained for the next seven centuries, until 1492 when the Christian Kingdoms totally reconquered the peninsula. The northern frontier of the country captured by the Muslims, known as al-Andalus, extended eastward on the southern slopes of the Cantabrian range from the present Galicia to Catalonia. Following the Muslim conquest, al-Andalus was at first (711–750) a province of the Umayyad Caliphate centered on Damascus. From 740 a series of civil wars between various Muslim groups resulted in the breakdown of the Arab empire and the Emirate of Cordova (c. 750–929) emerged. In 929 the emir of Cordova proclaimed himself Caliph and the period of the Caliphate of Cordova was established (929–1031). The Cordova Caliphate collapsed during a civil war and Al-Andalus broke up into a number of mostly independent states called taifa kingdoms. The independent taifas were too weakened to defend themselves against the Christian Kingdoms in the north and west, allowing the Reconquest. The Christian reconquest of Iberia ended with the final assault on the Emirate of Granada in 1492. From 711 to 1492, as political dominions changed, the boundaries between the Christian north and the Islamic south shifted constantly.

In the Ebro Valley, the first Muslims arrived in the early 8th century conquering the main towns without any relevant or attested resistance and the Upper March (or northern frontier) was established along the Ebro basin. Thus began the Muslim period in the Ebro Valley that, for four centuries, was centered on the metropolis of Saragossa. Shortly after the Muslim conquest, the nobleman Count Cassius converted to Islam to preserve his lands and political power and founded the Banu Qasi dynasty. In the 9th century the Upper March was under the dominion of the Banu Qasi dynasty as a semi-autonomous territory within the Cordova caliphate (García de Cortazar and Sesma, 2011; Hitchcock, 2011). During the 9th century, the Banu Qasi lineage was successively loyal and rebellious toward the Cordova emir. In the second half of the century, the Banu Qasi domains increased considerably, extending north to the Pyrenees and east nearly to the Mediterranean coastline. However, in the later 9th century the Cordova emir recovered most of the Upper March territories and in the early 10th century, harassed by its Christian neighbors and without the support of Cordova, the Banu Qasi dynasty lost all its territories (Cañada Juste, 1980).

The society of Al-Andalus was made up of three main religious groups: Christians, Muslims and Jews, who inhabited distinct neighborhoods in the cities. Islamic society stratification was mainly by ethnic division. The kinship system ascribed importance only to relationships through males and endogamous marriages were viewed as the ideal system (Coope, 2014). The more powerful a tribal group was, the more women it would attract from outside and the fewer it would lose, and the more endogamous it would become. Under Islamic law, the most privileged members of society were devout Muslim men, and women were treated as second-class citizens (Coope, 2014). In particular, women’s rights were contingent on their place within society on several levels, including their religious, economic and marital status. Under Islamic law, other groups in society such as Jews and Christians had fewer rights and privileges, to varying degrees.

Within this framework, Tauste was placed midway between the two most significant cities: Saragossa, metropolis of the Upper March, and Tudela, the centre of the Banu Qasi territory.

The Muslim occupation of Tauste (Zaragoza, Spain) has been considered incidental and even non-existent, according to traditional and written sources. However, recent excavations suggest a large stable Muslim population lived in the town from the early Islamic period in the Iberian Peninsula. In 2010, a cemetery with several human skeletons aligned perpendicular to Mecca was discovered. The bodies were placed on their right side, facing towards Mecca, as is characteristic of a Muslim cemetery (Petersen, 2013). In contrast, no remains of the Muslim village associated with this necropolis have yet been found. Multi-isotopic studies, including radiogenic strontium, stable oxygen, carbon, and nitrogen, have been used to reconstruct the geographic origin, mobility and dietary practices of the Tauste individuals during the Islamic period in the Iberian Peninsula. Stable isotope composition of bone collagen reveals information about nutrition, life history, and mobility in past populations (Ambrose, 1993; DeNiro and Epstein, 1978; Hedges and Reynard, 2007; Katzenberd, 2008; Lee-Thorp, 2008).

Isotope analyses in bioarchaeology

The analysis of carbon and nitrogen isotope composition in bone collagen constitutes an approach to palaeodietary reconstruction. It can provide information about the protein portion of the diet averaged over at least the last 10 years prior to death and also about different protein sources (Schwarcz and Schoeninger, 1991; Meier-Augenstein, 2010).

Carbon isotope analysis provides information about the ecosystem that foodstuffs come from, distinguishing between terrestrial and marine ecosystems. In the case of a terrestrial diet, it informs about the plants that were consumed. Two classes of plants are distinguished according to their photosynthetic pathways: C_3 plants and C_4 plants. C_3 plants are most vegetables, wheat (*Tritium*) and barley (*Hordeum vulgare*), while C_4 plants include millet (*Pennisetum*), maize (*Zea mays*) and sugar cane (*Succharum officinarum*). C_4 plants exhibit more enriched carbon values than C_3 plants, so that the mean $\delta^{13}C$ values are -13‰ and -27‰ respectively (Smith and Epstein, 1971; O`Leary, 1981). Marine plants are all C_3 plants and their average values are about 7.5‰ higher than terrestrial C_3 plants. Carbon isotope composition can be used to distinguish marine protein consumption in terrestrial C_3-based diets, but when C_4 plants are involved marine and terrestrial values can overlap (Hoefs, 1981; White, 2015). Carbon fractionates in $\delta^{13}C$ by only about 1‰ throughout the food chain (Ambrose, 1993; DeNiro and Epstein, 1978; Malaney, 2011). In freshwater ecosystems the $\delta^{13}C$ composition of plants is variable and consequently freshwater fish exhibit a broad range of $\delta^{13}C$ values that are largely depleted (Dufour et al., 1999; Pazdur et al., 1999). Therefore, $\delta^{13}C$ ratios more negative than -22‰, the value corresponding to the low-end of a diet based only on C_3 terrestrial plants, suggest freshwater fish consumption.

Nitrogen isotope values reflect the intake of animal proteins and inform about the trophic level of an individual (Lee-Thorp, 2008; Sandford, 1993; Bocherens and Drucker, 2003). Thus, nitrogen isotopes in terrestrial ecosystems are enriched in $\delta^{15}N$ by 2-5‰ (on average, 3‰) from food to body tissue as trophic levels increase (Hedges and Reynard, 2007; Schoeninger and Moore, 1992; Schoeninger et al., 1983).

Terrestrial protein sources have $\delta^{15}N$ values ranging from 5‰ to 12‰, while aquatic food sources range from about 12‰ to 22‰ for marine fish and 7.2‰ to 16.7‰ for freshwater fish (Schoeninger and DeNiro, 1984; Walker and DeNiro, 1986; Katzenberg and Weber, 1999; Fuller et al., 2012; Robson et al., 2015). When C_3 plants are consumed, nitrogen isotope analysis is combined with carbon isotope analysis to distinguish between proteins derived from terrestrial, freshwater and marine resources. Other reasons for variability in $\delta^{15}N$ ratios of plants and animals include natural environmental conditions such as salinity and aridity or anthropogenic factors like manuring (Bogaard et al., 2007; Fraser et al., 2011). In general, human diet corresponds to a mixture of food with different isotope signatures. Plots of collagen $\delta^{13}C$ vs $\delta^{15}N$ values can be interpreted as mixtures of multiple components (Schwarcz and Schoeninger 1991; Phillips and Gregg, 2003) that do not yield unique solutions, but may outline the dominant components in the diet of the studied individuals. Besides, stable nitrogen isotope analysis can also be used to investigate breastfeeding and weaning practices. In fact, during breastfeeding, children exhibit $\delta^{15}N$ values enriched about 2-3‰ over that of their mothers (Fogel et al., 1989).

Strontium and oxygen isotopes are two independent isotopic systems in which strontium reflects local geology and oxygen reflects geography and can be used to reconstruct movements of past populations. The combination of these two isotopic systems is able to constrain possible areas and provide information about an individual's area of origin and thus determine mobility patterns (Bentley and Knipper, 2005; Evans et al., 2006a; Evans et al., 2006b).

Oxygen and strontium are fixed in phosphate in teeth and bones through ingested food and water. Strontium isotopes appear by substituting calcium in biogenic phosphate (Hillson, 1996; Price et al., 2002; Hoppe et al., 2003). After formation during infancy, tooth enamel does not incorporate other elements and thus will reflect the geological composition of the place of residence during childhood, assuming that childhood residence and food production area coincide, at least for the majority of the food intake (Hillson, 1996, Hillson, 1986). However, these patterns are not perfectly predictable at any level, because of vagaries in available food over time, and because the strontium ratio reflects an average value that synthesizes the geological composition of the different food provenances ingested during childhood. The average expected patterns are used to predict the most likely geographic links between tissue and location. In contrast, tooth dentine and bones are continuously remodeled throughout an individual's lifetime.

The radiogenic strontium isotopes are related to geology and vary according to the composition and age of bedrock. The strontium concentration in organisms varies according to the trophic level but the $^{87}Sr/^{86}Sr$ isotope signature of humans and fauna has negligible metabolic fractionation and will reflect the isotope signature of the underlying bedrock (Hoppe et al., 2003; Burton, 1996; Hoppe et al., 1999; Blum et al., 2000; Balter et al., 2001; Balter, 2004; Faure and Mensing, 2005; Hoppe and Koch et al., 2007). $^{87}Sr/^{86}Sr$ ratios in bedrock, soils, water and plants will be reflected in humans and animals that consume food and water from those sources (Bocherens and Drucker, 2003; Price et al., 2002; Bentley, 2006). Since the $^{87}Sr/^{86}Sr$ isotope ratio is inherited from the local environment, it is necessary to define the local bioavailable strontium isotope signature to evaluate residential mobility of individuals. There are several methods to establish the local baseline of the isotope signature by analyzing environmental samples including freshwater, soil leachates, ancient fauna and present-day small wild animals (Price et al., 2002; Bentley et al., 2004; Evans et al., 2010; Tütken et al., 2011). However anthropogenic activities such as the use of fertilizers could modify the strontium isotope ratios of modern ecosystems (Böhlke and Horan, 2000; West et al., 2009; Tichomirowa et al., 2010; Christian et al., 2011).

In contrast, the oxygen isotope reflects the isotopic composition of ingested water that is derived from meteoric water. The $\delta^{18}O$ in precipitation varies regionally according to temperature and other climatic parameters, such as distance from the coastline, altitude and latitude (Longinelli, 1984; White et al., 1998; Darling et al., 2006; Daux et al., 2008). Oxygen isotopes in the body are subject to several steps of metabolic fractionation. The fractionation mechanisms are relatively well known, allowing the calculation of approximate drinking water ($\delta^{18}O_w$) values from the $\delta^{18}O_p$ of biogenic phosphate by means of conversion equations (Longinelli, 1984; Daux et al., 2008; Luz et al., 1984; Luz and Kolodny, 1985; Levinson et al., 1987; Bryant and Froelich, 1995; Kohn, 1996; Iacumin and Venturelli, 2015). Despite complexities in the calculation of meteoric water isotope composition in the past, the oxygen isotope composition of human remains allows the identification of palaeomobility patterns.

The aim of this study was to reconstruct palaeomobility and palaeodiet patterns in the medieval Muslim population at Tauste. Tauste Muslim necropolis constitutes a suitable site to examine human mobility since it was located on the northern frontier during a very convulsive period of time. In addition, the palaoedietary pattern can illustrate the basic dynamics of medieval Muslim social life. For these purposes, stable isotopes ($\delta^{13}C$, $\delta^{15}N$, $\delta^{18}O$) and radiogenic strontium ($^{87}Sr/^{86}Sr$) were investigated to obtain information about nutrition and social stratification.

Archaeological setting

Tauste archaeological site is located in the town with the same name in the province of Zaragoza (northern Spain) (Fig. 1). Tauste is in the Ebro basin, on the River Arba, a tributary of the River Ebro. The Muslim archaeological site of Tauste is formed only by the cemetery, with a total absence of other vestiges of Islamic population. All the graves were aligned SW-NE and the human bodies were carefully placed on their right side, facing Mecca, indicative of a Muslim necropolis (Stutz and Tarlow, 2013) (Fig. 2).

All individuals were found in anatomical connection. Graves were dug in clay soil without any structure, or only a minimum structure formed by rammed earth on the sides according to Muslim burial rituals. More complex tombs corresponding to double grave burials (*shaq* or *ladj*) were found. A similar burial system has been documented in other Muslim necropolises in the Iberian Peninsula, such as Marroquíes Bajos (Jaen) (Serrano Peña et al., 2000), Tossal de Manises (Alicante) (Olcina et al., 2006) or the recent find at Valdeherrera (Calatayud). The human remains extended over an area of two hectares and the density of graves (0.25-0.30 individuals/m^2) indicates a minimum of 4.500 burials, excluding children (Gutierrez amd Pina, 2011; Gutierrez and Pina, 2016).

Only a simple bronze hoop ear-ring was found in a female's grave, and the lack of grave goods is also indicative of Islamic funeral rituals. The excavations have found at least two levels of burials, indicating this cemetery was in use during an extended period of time.

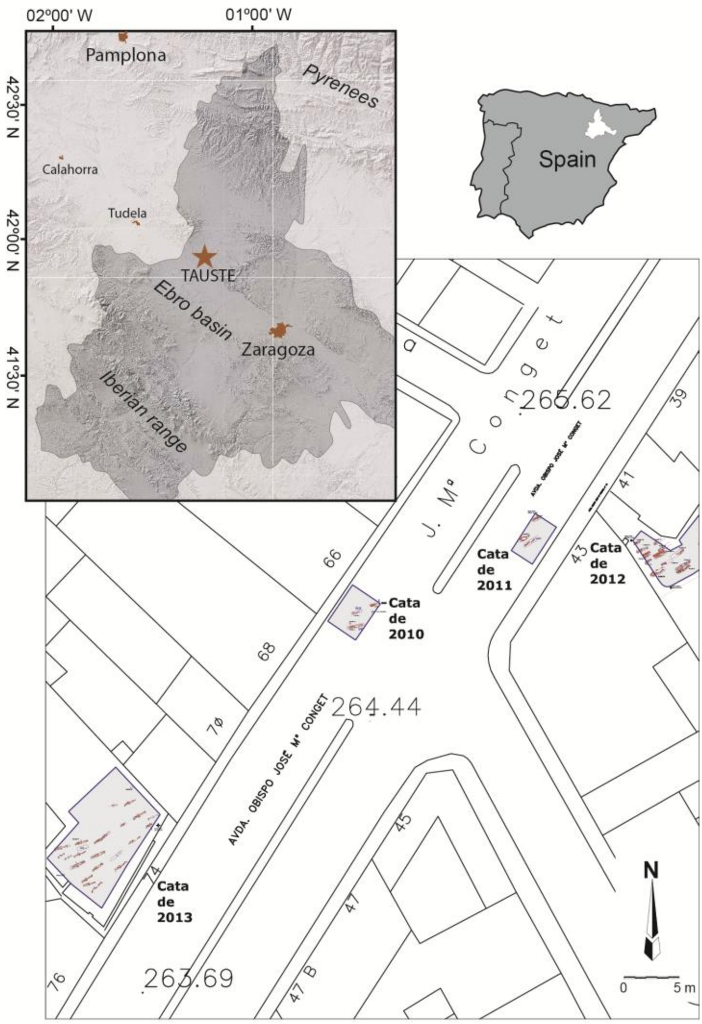

Figure 1. Location of Tauste archaeological site and the excavated areas. Reprinted from under CC by license, with permission from [Instituto Geográfico Nacional (IGN)], original copyright [2015].

Figure 2. Aerial view of some burials showing individuals placed in the graves following the Muslim burial rituals (facing east).

Radiocarbon dating of human bones dates the graveyard inthe 8th to 10th centuries and it could be one of the oldest Muslim necropolises in the Iberian Peninsula (Table 1, Fig. 3). Calendar ages were determined using the Oxcal v 4.2.4 program (Bronk Ramsey and Lee, 2013) with the latest IntCal13 calibration curve for atmospheric data (Reimer et al., 2013). Calibrated age ranges correspond to 95.4% probability (2σ) and are expressed in years cal AD. The age and extent of the necropolis suggest Tauste was a thriving village in the times of the Banu Qasi dynasty, when the northernmost limit of Al-Andalus was established (James, 2009; Safran, 2013).

Table 1. Calibrated radiocarbon dating of the Tauste site.

Sample	Lab. Code	Age BP	Age cal AD		
			from	to	%
Tomb 1	CSIC-2180	1072 ±32	895	929	22.7
			939	1021	72.7
Tomb 2	CSIC-2235	1286±31	664	772	95.4
			777	791	3.3
Tomb 3	CSIC-2234	1133±28	806	842	5.7
			861	986	86.4

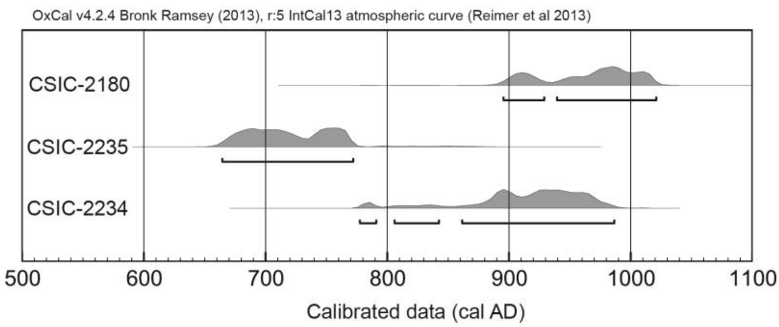

Figure 3. Radiocarbon dating of human bone samples from Tauste calibrated with OxCal v4.2.4 (Bronk Ramsey and Lee, 2013) and IntCal13 atmospheric data (Reimer et al., 2013).

Geologically, the Muslim necropolis of Tauste is located in the Ebro Basin, composed by Tertiary (Miocene) and Quaternary sedimentary rocks of continental origin (Fig. 4) (Rodríguez Fernández et al., 2015). Miocene materials consist of claystones and siltstones with subordinate sandstones and limestones, and interbedded gypsum layers and terrigenous episodes. These materials correspond to evaporite lacustrine facies, i.e. sediments deposited in the center of a continental sedimentary basin. Miocene deposits are overlaid by Quaternary materials consisting mainly of river terrace deposits and fluvial sediments. The evaporitic nature of the bedrock causes a large increment in salt contents in the environment. In fact, high levels of sodium chloride and sulphate ions have been found in the freshwater River Arba (Navas, 1988; Causapé Valenzuela, 2003).

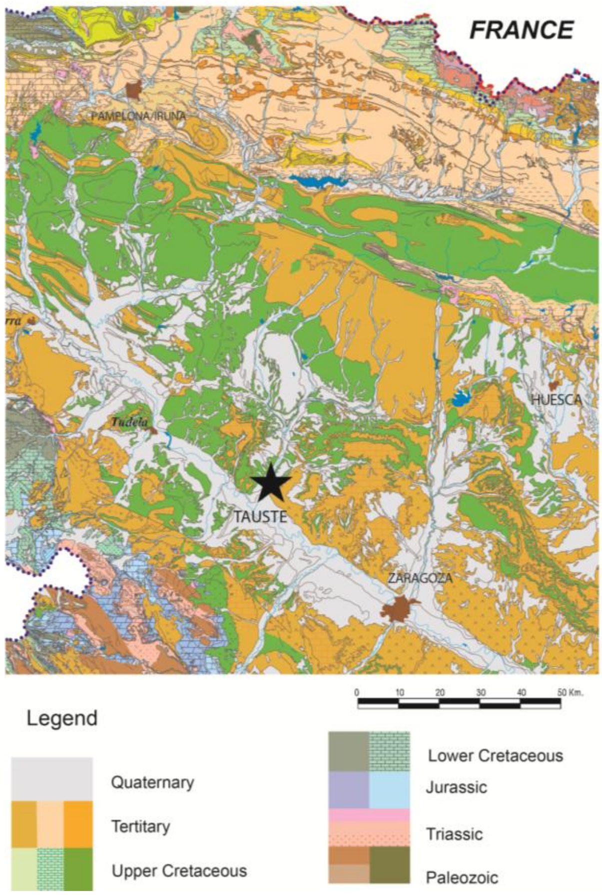

Figure 4. Geological map of the Tauste region showing evaporitic nature of the bedrock [taken from Rodríguez Fernández et al. (2015). Reprinted from under CC by license, with permission from [Instituto Geológico y Minero de España (IGME)], original copyright [2015].

Materials and Methods

This study deals with archaeological skeletal material and all necessary permits were obtained for the described study, which complied with all relevant regulations. The excavation licenses were issued by the General Director of Cultural Heritage of the *Gobierno de Aragon* (Spain) and are stored in its archives. Following excavation campaigns 2010/2013, the bones and teeth samples were transferred to the Heritage and Cultural Landscape Research Group (GIPyPAC) at the University of Basque Country-UPV/EHU, Spain for investigation. At present all archaeological remains, including the human bones, recovered at the site of Tauste are stored in the Museo de Zaragoza.

Carbon and nitrogen isotope measurements have been performed in bone collagen extracted from 31 human individuals corresponding to the sectors excavated in 2012 and 2013 (Fig. 5) and nine faunal bone samples. Additionally, 23 teeth and 8 bone samples were analyzed for strontium and oxygen isotope studies. In order to define the strontium isotope baseline, four soil samples and one freshwater sample were analyzed. The soil samples were collected in different parts of the cemetery, while the freshwater sample was collected from the River Arba near Tauste. Surface waters were collected from the banks of the river. Before analysis, water samples were filtered to remove suspended particles.

Water aliquot was filtered with disposable syringe filters (0.45 um) and 10 mL aliquot was transferred to a 15 mL Teflon (Savillex™) vial, evaporated down on a hot plate at 80ºC overnight and then dissolved in 1.5 mL of 2N HNO_3.

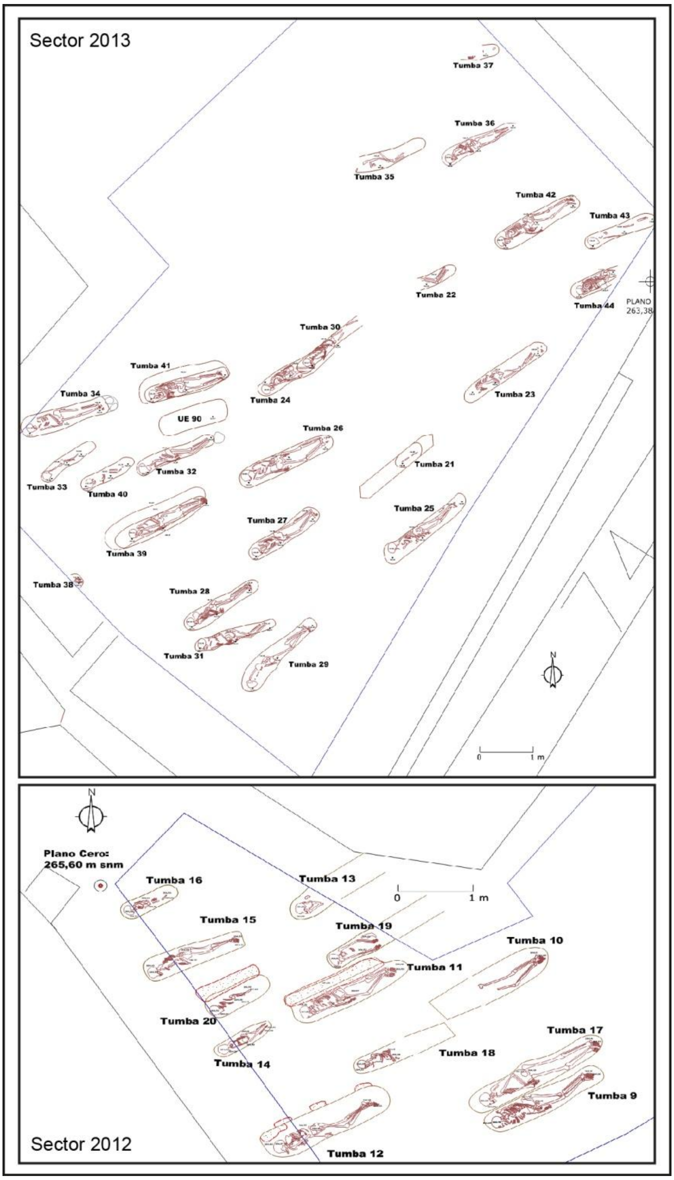

Figure 5. Detailed map of two excavated areas (sectors 2012 and 2013) showing studied individuals.

The measured individuals were corresponded to 12 males, 10 females and 9 of indeterminate sex (Gutiérrez and Pina, 2016). The individuals were categorized by age into infants (0-3 years), children (6-12 years), juveniles (12-17 years), young adults (18–34 years), middle-aged adults (35–50 years), and older adults (older than 50 years). According to these categories, 2 individuals are older adults, 11 are adults, 6 are juveniles, 7 are young adults, 4 are infants and 1 is of indeterminate age. Sex determination was carried out according to the classical patterns of dimorphism and age was defined by the most reliable markers: changes in auricular surface and pubic symphysis, epiphyseal closure, cranial sutures and dental eruption (White et al., 1991). The faunal samples corresponded to three wood mice (*Apodemus sylvaticus*), three shrews (*Crocidura russula*) and two common barbels (*Barbus barbus*) and a madrilla (*Parachondrostoma miegii*).

For carbon and nitrogen isotope analyses, bone collagen was extracted following the procedure in Bocherens et al. (1991). 300 mg of bone sample powder were demineralised in 1M HCl for 20 min at room temperature until the sample dissolved. To remove humic acid the samples were rinsed with distilled water and treated with 0.125 M NaOH. The resulting insoluble fraction after being rinsed again with distilled water was gelatinized in HCl solution at pH3 for 17 h at 90ºC. Then, samples were filtered with disposable syringe filters (5 um), freeze-dried and finally lyophilized. Lyophilized collagens (2.5-3.5 mg) were enclosed in tin capsules for isotopic analysis. Carbon and nitrogen isotope analyses were performed using an elemental analyzer on line with a continuous-flow isotope ratio mass spectrometer (EA-IRMS) at Iso-Analytical (Cheshire, UK). Replicate measurements of the liver standard NBS-1577B and ammonium sulphate IA-R045 working standard were run to confirm instrument accuracy. Replicate analysis of the NBS-1577B $\delta^{13}C$ standard during runs gave a $^{13}C/^{12}C$ of −21.62 ± 0.02 (1σ, n = 7) and

$^{15}N/^{14}N$ of 7.62 ± 0.13 (1σ, n = 7), and the IA-R045 working standard during runs gave a $^{15}N/^{14}N$ of −4.56 ± 0.17 (1σ, n = 4) and $^{13}C/^{12}C$ of −26.1 ± 0.03 (1σ, n = 4). Isotopic values are reported as δ values in per thousand (‰) relative to international defined standards for carbon (VPDB: Vienna Pee Dee Belemnite) and nitrogen (AIR: Ambient Inhalable Reservoir). The instrumental precision for $\delta^{13}C$ was ± 0.06‰ or better and for $\delta^{15}N$ was between ± 0.06‰ and ± 0.08‰, determined by replicated analyses of internal standards.

Tooth enamel and bones were used to determine strontium and oxygen isotope composition. The samples were washed in an ultrasonic bath to remove impurities and further cleaned by mechanical abrasion to remove the outer surface and avoid potential contamination.

For strontium isotope analysis a fraction of dental enamel was collected mechanically with a diamond-coated trepanation drill (MF-perfect, W & H Dentalwork, Bürmoos, Austria). The enamel sample was taken transversally. Enamel and bone samples (~10 mg) were dissolved in 7 ml Savillex® vials (Minnetonka, MN, USA) with 1.5 mL of 2N HNO_3 (analytical grade purified by subboiling distillation). In order to establish the local isotopic composition, two water samples and four soil samples were also analyzed. 15 ml of freshwater was evaporated to dryness and then dissolved in 2 mL HNO_3. A 1g aliquot soil sample was leached by adding 2.5 ml 1 M ammonium nitrate (NH_4NO_3) and shaking for 8 h to obtained the bioavailable Sr. After samples were centrifuged at 3000 rpm for 15 min, the supernatant was extracted (~1-2 l) and evaporated to dryness and then redissolved in 2 ml HNO_3. The solutions were loaded into cation exchange columns filled with Sr.spec® (ElChroM industries, Dariel, IL, USA), a strontium selective resin. The resin was used once to elute the sample and then discarded. Strontium procedural blanks were less than 100 pg and hence provided a negligible contribution.

The radiogenic strontium isotope samples were analyzed on a Neptune multi-collector inductively coupled plasma mass spectrometer (MC-ICP-MS) at the Advanced Research Facilities (SGIker) of the University of the Basque Country (UPV/EHU). $^{87}Sr/^{86}Sr$ measurements were corrected for krypton (Kr) and rubidium (Rb) interferences and normalized for instrumental mass bias using $^{87}Sr/^{86}Sr$ = 8.375209. Repeated analyses of NIST SRM-987 international standard yielded a value of $^{87}Sr/^{86}Sr$ = 0.710262 ± 0.000026 (2σ, n = 3). Long-term $^{87}Sr/^{86}Sr$ value, determined over a twenty-two month period, was 0.710266 ± 0.000021 (2σ, n = 47).

Tooth enamel was also prepared for oxygen isotope analysis following the procedure described in Stephan (Stephan, 2016). 60 mg of dental enamel powder was processed. The organic matter was removed with a solution of 2.5% NaOCl for 24 h at room temperature followed by a 48 h treatment in 0.125M NaOH at room temperature. The hydroxyapatite powder free of organic matter was dissolved in 2 ml of HF for 24 h. The phosphate solution and the residue composed of CaF_2 were separated by centrifugation, pipetted into a 100 ml glass tube and neutralized with 3 ml 2M KOH. Silver phosphate (Ag_3PO_4) was precipitated by adding 30 mL of a buffered silver amide solution (0.2 M $AgNO_3$; 1.16 M NH_4NO_3; 0.75 M NH_4OH) gradually warmed up to 70ºC, holding the temperature for 5-6 h and cooling down slowly. Silver phosphate crystals were filtered on a weighed 0.2µm filter and washed several times with double distilled water, then dried at 50ºC for 1-2 h. Subsequently, 0.3 mg of Ag_3PO_4 was mixed with 0.5-1 mg of AgCl and 0.3

mg of graphite in silver capsules. The capsules were transferred into the autosampler carousel of the Temperature Conversion Elemental Analyser (TCEA) and degassed for 30 minutes at 80°C in a vacuum. The oxygen isotope analyses were performed on a Thermo Finnigan TCEA coupled to a Delta Plus XP Spectrometer at the University of Parma. Isotopic compositions were given in the conventional δ-notation relative to V-SMOW (Vienna-Standard Mean Ocean Water). Normalization to the V-SMOW scale was based on four replicated international reference materials provided by the International Atomic Energy Agency (IAEA): IAEA-601, IAEA-602, IAEA-CH6, and IAEA-SO-6. The analytical precision of a single determination was better than ±0.4‰.

To identify outliers in $\delta^{18}O_{PO4}$ and in $^{87}Sr/^{86}Sr$ within the Tauste population two statistical techniques were used. Boundaries of intra-sample variation based on two measurements of scales were defined: ± 2 standard deviation (2SD) from the mean and Tuke's inter-quartile range method (IQR) considering 1.5xIQR and 3xIQR (Lightfood and O'Connell, 2006). Parametric statistics were used to describe isotope distribution and compare isotope values between groups. Differences between sample groups were analyzed by applying an unpaired Student's t-test. Statistical significance was accepted as $p < 0.05$. Statistical analysis was performed with SPSS v.20 (Statistical package for Social Sciences).

Results and discussion

Residential mobility

Strontium and oxygen isotope data for 23 tooth enamel samples, 8 rib bones and local geological materials to establish the strontium baseline signature at Tauste are shown in Table 2.

Table 2. Strontium, carbon, nitrogen and oxygen isotope results for human bones and teeth, and freshwater and soil samples from Tauste.

Sample	Sex	Material	Tooth Type	Age	% N	$\delta^{15}N$	% C	$\delta^{13}C$	C/N	$\delta^{18}O$	$^{87}Sr/^{86}Sr$	2SE
T-9	F	Rib		50-60	15.4	12.7	42.7	-19.3	3.23		0.70868	0.00001
		Enamel	M2							16.35	0.70855	0.00002
T-11	M	Rib		45	14.9	15.4	40.8	-19.5	3.19			
		Enamel	M2							16.72	0.70855	0.00002
T-12	M	Rib		40-45	16.7	15.7	45.8	-19.5	3.20			
		Enamel	I							16.78	0.70858	0.00001
T-13	M	Rib		40-50	15.6	16.3	42.8	-19.0	3.21			
		Enamel	M2							18.00	0.70857	0.00002
T-14	?	Rib		2-4	16.6	16.8	45.6	-18.9	3.21			
T-15	F	Rib		33-45	13.9	15.6	39.2	-18.5	3.29		0.70867	0.00001
		Enamel	M2							14.25	0.70860	0.00001
T-16	?	Rib		2	15.2	16.3	42.3	-17.0	3.25			

Table 2. Continued. Strontium, carbon, nitrogen and oxygen isotope results for human bones and teeth, and freshwater and soil samples from Tauste.

Sample	Sex	Material	Tooth Type	Age	% N	$\delta^{15}N$	% C	$\delta^{13}C$	C/N	$\delta^{18}O$	$^{87}Sr/^{86}Sr$	2SE
		Enamel	M2							17.03	0.70863	0.00001
T-17	M	Rib		33-45	15.5	15.3	42.5	-19.3	3.21		0.70868	0.00001
		Enamel	M2							16.65	0.70861	0.00002
T-18	M	Rib		25-35	15.1	13.7	42.1	-18.8	3.24		0.70869	0.00001
		Enamel	M2							17.17	0.70862	0.00001
T-19	F	Rib		33-45	14.5	14.1	40.2	-19.0	3.24			
		Enamel	M2							17.14	0.70850	0.00002
T-21	?	Rib		Indet	15.0	9.6	41.7	-19.3	3.25		0.70869	0.00001
T-22	?	Rib		>17	15.0	15.9	41.3	-19.1	3.22			
T-23	F	Rib		30-35	15.8	14.3	43.2	-19.1	3.20			
T-24	F	Rib		25-35	13.9	16.3	38.5	-19.3	3.23		0.70868	0.00001
		Enamel	M2							17.9	0.70837	0.00001

Table 2. Continued. Strontium, carbon, nitrogen and oxygen isotope results for human bones and teeth, and freshwater and soil samples from Tauste.

Sample	Sex	Material	Tooth Type	Age	% N	$\delta^{15}N$	% C	$\delta^{13}C$	C/N	$\delta^{18}O$	$^{87}Sr/^{86}Sr$	2SE
T-25	F	Rib		>20	15.3	16.0	42.4	-19.0	3.24			
T-26	M	Rib		25-35	15.9	16.0	43.6	-19.4	3.19			
		Enamel	M2							16.64	0.70862	0.00002
T-27	(m)	Rib		15-17	15.1	13.5	41.7	-19.3	3.23			
		Enamel	C							16.92	0.70857	0.00002
T-28	?	Rib		12-15	14.8	15.8	40.9	-19.3	3.22			
		Enamel	M2							17.8	0.70858	0.00002
T-29	?	Rib		>25	13.1	15.5	36.2	-19.1	3.23			
T-30	F	Rib		35-45	15.4	14.3	42.2	-18.4	3.21			
		Enamel	M2							18	0.70864	0.00001
T-31	F	Rib		16-20	15.8	14.2	43.7	-18.8	3.22			

Table 2. Continued. Strontium, carbon, nitrogen and oxygen isotope results for human bones and teeth, and freshwater and soil samples from Tauste.

Sample	Sex	Material	Tooth Type	Age	% N	$\delta^{15}N$	% C	$\delta^{13}C$	C/N	$\delta^{18}O$	$^{87}Sr/^{86}Sr$	2SE
		Enamel	M2							14.48	0.70859	0.00002
T-32	F	Rib		45-55	15.8	10.8	43.6	-19.0	3.23		0.70883	0.00002
		Enamel	M2							17.9	0.70867	0.00002
T-33	?	Rib		4-6	15.7	14.6	43.7	-19.5	3.24			
		Enamel	M2							17.0	0.70864	0.00002
T-34	M	Rib		12-15	15.8	15.5	43.9	-18.7	3.25			
		Enamel	M2							16.9	0.70853	0.00002
T-35	?	Rib		40-50	15.1	16.9	41.9	-19.2	3.25			
T-36	F	Rib		50-65	14.9	13.9	41.4	-19.1	3.24			
T-39	M	Rib		40-50	14.1	16.5	39.2	-19.5	3.23		0.70867	0.00001
		Enamel	M2							17.4	0.70860	0.00002
T-40	?	Rib		3-5	16.1	17.5	44.1	-19.9	3.20			

Table 2. Continued. Strontium, carbon, nitrogen and oxygen isotope results for human bones and teeth, and freshwater and soil samples from Tauste.

Sample	Sex	Material	Tooth Type	Age	% N	$\delta^{15}N$	% C	$\delta^{13}C$	C/N	$\delta^{18}O$	$^{87}Sr/^{86}Sr$	2SE
		Enamel	M2							17.06	0.70866	0.00002
T-41	M	Rib		20-30	16.3	15.0	45.3	-19.5	3.24			
		Enamel	M2							17.0	0.70860	
T-42	M	Rib		35-45	16.7	17.0	46.4	-19.0	3.23			
		Enamel	M2							19.1	0.70851	
T-44	M	Rib		25-35	16.7	14.0	46.2	-18.9	3.23			
		Enamel	M2							19.3	0.70856	
T-39*		Soil									0.70868	0.00002
T-41*		Soil									0.70869	0.00002
T-42*		Soil									0.70867	0.00001
T-44*		Soil									0.70867	0.00001
W-Arba		Water									0.70843	0.00001

m = male; (m) = probably male; M= molar; C=canine; I=incisor; ? = undetermined; f = female. *= Soil samples. 2SE=standard error.

To establish local bioavailable strontium, bedrock, fauna, soils and surface water are used but archaeological microfauna or snail shells are considered the most appropriate material (Philips and Gregg, 2003; Price et al., 2002; Blum et al., 2000; Balter et al., 2002; Maurer et al., 2012: Voerkelius et al., 2010). Since the site is a Muslim necropolis, no fauna remains are associated with the burials. Therefore, to define the Tauste bioavailable strontium isotope baseline, surface water and soils were considered. Local $^{87}Sr/^{86}Sr$ isotope composition determined by the local soils varies between 0.70867 and 0.70869, while the freshwater composition is 0.70843. The $^{87}Sr/^{86}Sr$ ratios of enamel vary between 0.70837 and 0.70867 and human bone samples range from 0.70867 to 0.70883 (Fig. 6).

According to the defined local baseline, most individuals buried in Tauste have a $^{87}Sr/^{86}Sr$ signature consistent with local origin. Only two individuals plot outside the estimated local compositional range (1σ) according to outliers indentifying by 1.5xIQR and 2SD method.

Enamel of young adult female T-24 displays a lower radiogenic strontium isotope value ($^{87}Sr/^{86}Sr$ = 0.70837) and the rib of adult female T-32(b) presents a higher strontium value ($^{87}Sr/^{86}Sr$ = 0.70883) (Fig. 6). These compositions indicate two different mobility patterns for these females. The female T-24's strontium value suggests she was born in another place and died in Tauste. Whereas female T-32(b)'s isotope value suggests that she spent her childhood in Tauste, during adulthood moved to another location and came back to Tauste a few years before she died.

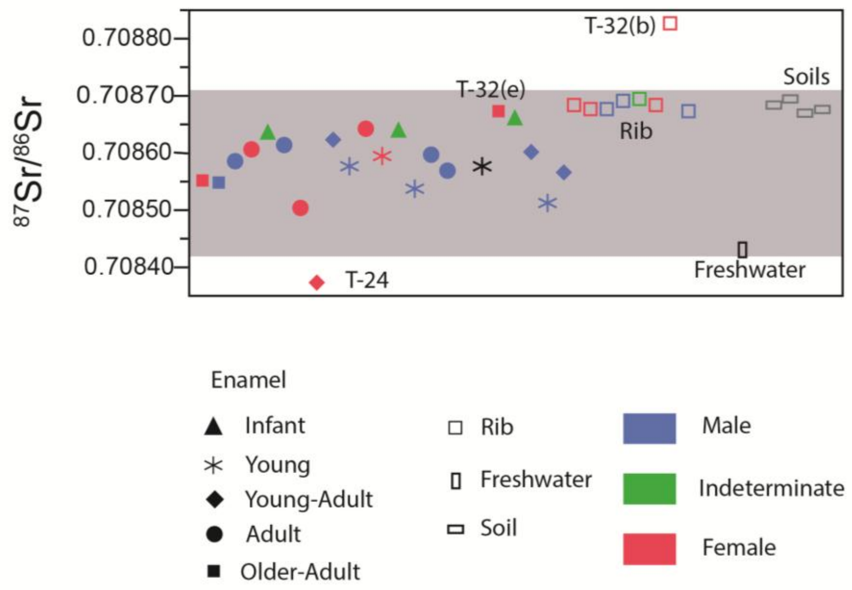

Figure 6. Strontium isotope variation of studied samples. Grey area corresponds to local strontium background defined by local freshwater and soils. Abbreviations (e): enamel sample, (b): bone sample.

The enamel phosphate oxygen ratios ($\delta^{18}O_p$) cover a broad range of values from 14.25‰ to 19.30‰. Based on kernel density estimations (Fig. 7), data can be split into three groups: a larger group (n=18) with $\delta^{18}O_p$ ratios between 16.4‰ and 18‰ and two smaller groups, one formed by two males with $\delta^{18}O_p$ signature higher than 19.2‰ and the other formed by two females with $\delta^{18}O_p$ signature lower than 14.4‰. Local meteoric water $\delta^{18}O_{dw}$ is -5.6‰ (average values from 2000 to 2006) considering data from Zaragoza airport station (Global Network of Isotope in Precpitation, 2015). $\delta^{18}O_{dw}$ values for humans from Tauste were calculated using the available phosphate/drinking water equations (White et al., 1998; Daux et al., 2008; Luz et al., 1984; Levinson et al., 1987; Iacumin and Venturelli, 2015), and comparing them with expected local water values derived from IAEA/WISER data set (Global Network of Isotope in Precpitation, 2015). The equation by Iacumin and Venturelli (2015) was used to estimate the drinking water isotope value. The larger group of individuals show calculated $\delta^{18}O_{dw}$ ranging from -3.7‰ to -6.9‰, consistent with local meteoric water which ranges between -4.1‰ and -6.3‰ as annual average (Global Network of Isotopes in Precipitation, 2015).

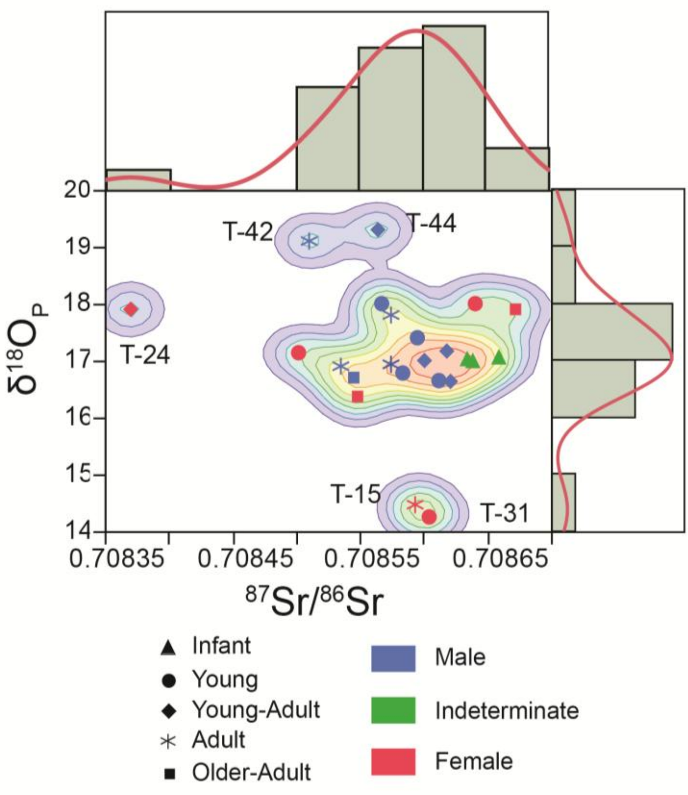

Figure 7. $\delta^{18}O$ versus $^{87}Sr/^{86}Sr$ in the tooth enamel of Tauste individuals. Kernel density contour lines represent 10%.

The number of outliers identified was determined using 1.5xIQR and 2SD statistical methods. The two males and two females whose isotope values fall outside the larger group may come from a warmer, more coastal or possible more arid climate, and from a colder or higher altitude region, respectively (Fig 8). When oxygen isotope data are compared with $^{87}Sr/^{86}Sr$ ratios of the same teeth the individuals in the three groups display isotopic values compatible with bio-accessible strontium measured to establish the local signature. Only the female T-24 falls strictly outside the expected strontium range for local origin although she falls into the expected range of the calculated drinking water values for Tauste (Fig. 7). Similar values of $\delta^{18}O$ for Tauste meteoric water showed a broad geographic distribution (Fig. 8) overlapping different geological areas. The lack of a strontium isotope composition database in the Iberian Peninsula prevents a determination of the regional provenance of the non-local female T-24.

During Muslim period the Ebro valley was a trade route between Mediterranean coast towards north of the Iberian Peninsula and trans-Pyrenean Europe. Taking on account both oxygen and strontium isotope data the origin areas for these two outliers groups would be confined to the Ebro Valley. Though, during the Early Middle Ages few people regularly travelled because it was simply too difficult and too dangerous. Muslim female were subject to inbreeding marriages so that Tauste outlier females would move by patrilocal marriages. Males should move to get better economic opportunities and possibility of improving status. Tauste non-local males would move from farms to a large village to find new kinds of works. The political instability of the Upper March frontier region also favored displacement of people towards larger and safer urban centers (Bittel, 2002).

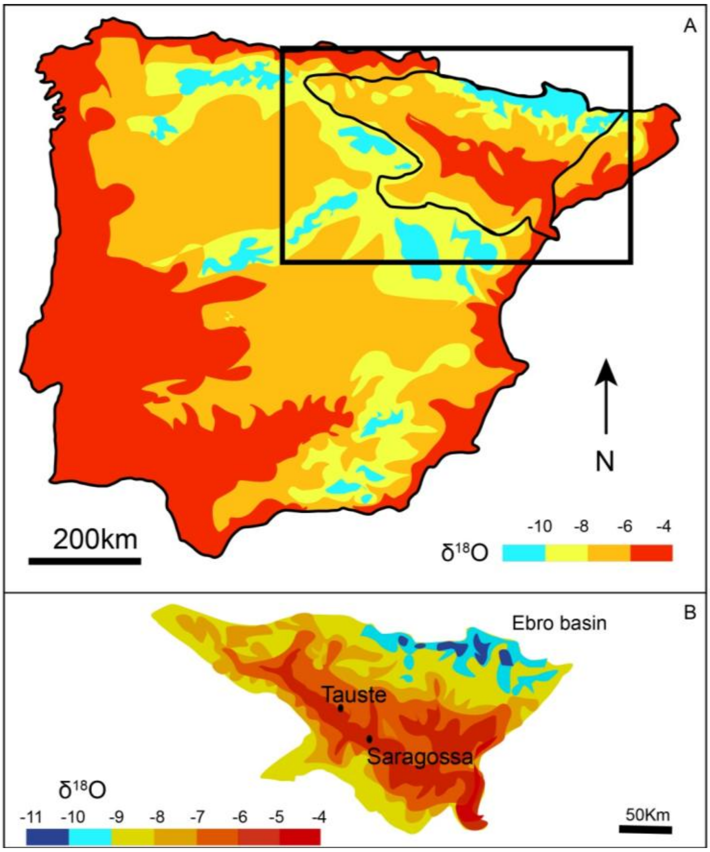

Figure 8. A. Large-scale map of oxygen isotope signatures in the Iberian Peninsula (Bol et al., 2005; White, 2013). **B.** Detailed map of oxygen isotope signature of the Ebro Valley (Bol et al., 2005; White, 2013).

Dietary reconstruction

The bone collagen obtained was very well preserved with an average yield of 6.27±4.19%wt (1s.d.). The content of carbon and nitrogen in bone collagen was about 13.1-16.8% and 25.6%- 36.2%, respectively, so well-preserved bone collagen should display a carbon/nitrogen molar ratio, based on the content (in %) of these elements in the sample between 2.9 and 3.6 (DeNiro, 1985; Ambrose, 1990). The individual data are given in Table 2.

The $\delta^{13}C$ ratio for human bone samples ranges between -17.0‰ and -19.9‰ with a mean value of -19.1±0.50‰, and $\delta^{15}N$ ratios range between 9.6‰ and 17.5‰ with a mean value of 14.9±1.74‰. The rather strong $\delta^{15}N$ signal of nearly all individuals is noteworthy, with values more than 5‰ over the terrestrial ecosystem baseline (Fig. 7), an offset unusually large for a single trophic level effect (Barret et al., 2002; Müldner and Richards, 2007; Bocherens and Drucker, 2013).

Individuals from Tauste were compared with broadly coetaneous Muslim and Christian populations at several locations in the Iberian Peninsula (Fig. 9). There are no significant differences in $\delta^{13}C$ ratios between Tauste (-19.1±0.5‰) and most neighboring Muslim populations ($\delta^{13}C$ -19.0±0.3‰ in Zaragoza and -19±0.2‰ in Albarracín), or between Tauste and Christian populations ($\delta^{13}C$ -18.4±1.1‰ in Jaca, -18.4 ±0.6‰ in Valencia, -19.0±1‰ in Aistra, -19.8±0.7‰ in Zaballa, -18.1±1.1‰ in Zornoztegi and -19.6±0.7‰ in Treviño (Quiros Castillo, 2013; Mundee, 2010; Alexander et al., 2014) (Table 3). However, the Benipeixcar Muslim population showed enrichment in $\delta^{13}C$ (-16.36±0.9‰) attributed to marine resource consumption (Alexander et al., 2014). In contrast, the $\delta^{15}N$ ratios at Tauste (average 15.0±1.7‰) are unusually high compared with contemporaneous Christian and Muslim populations in the Iberian Middle Ages, whose average values are lower 11‰ (Table 3).

For these reasons, two local species of small mammals and freshwater Faunal samples are required to strengthen conclusions about human diet. Establishing the local isotope composition baseline was problematic since the Islamic burial ritual forbade any objects being buried with the body. Additionally, there is no evidence of Muslim settlement in Tauste to provide coeval fauna. Thus it was not possible to obtain the local baseline with archaeozoological data. Furthermore, present-day local faunal must be discarded because livestock are fed with non-local resources and the isotopic signal will not correspond to local plant resources. Pasture-fed livestock will also exhibit nitrogen isotope depletion due to the widespread use of mineral fertilizers (Bol et al., 2005; White, 2013). In addition to the fodder and fertilizer effect, livestock trade is another factor affecting isotope composition. All these factors prevented the use of present-day macro-mammals to establish the carbon and nitrogen isotope local baseline.

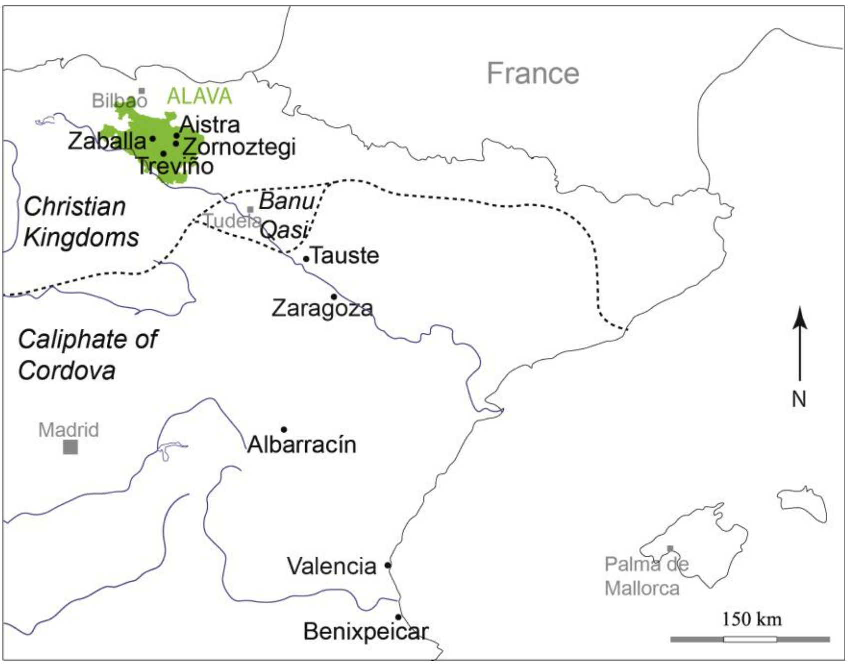

Figure 9. Location of Muslim and Christian archaeological sites and the Upper March or Muslim northern frontier during the 9th century.

Table 3. Carbon and nitrogen isotope average, SD, max and min values and subgroup (sex and age group) of individuals at Tauste. The comparative coeval dataset of Christian sites: Zornoztegi, Aistra, Zaballa, Treviño, Jaca, Valencia; and Islamic sites: Albarracín, Benipeixcar and Zaragoza (Quiros Castillo, 2013; Mundee, 2010; Alexander et al., 2014).

Site	Religion	N	$\delta^{13}C$ (‰)				$\delta^{15}N$ (‰)			
			Mean	Std Dev	Max	Min	Mean	Std Dev	Max	Min
Tauste (8th-10th)	I	31	-19.1	0.5	-17.0	-19.9	15.0	1.7	9.6	17.5
Male	I	11	-19.2	0.3	-18.7	-19.5	15.5	1	17.0	13.7
Female	I	10	-19.0	0.3	-18.4	-19.3	14.2	1.6	17.5	10.8
Infant	I	4	-18.8	1.3	-17.0	-19.9	16.3	1.2	17.5	14.6
Juvenile	I	6	-19.0	0.3	-18.7	-19.3	15.1	1.0	16.0	13.5
Young adult	I	7	-19.2	0.3	-18.8	-19.5	15.0	1.0	16.3	13.7
Adult	I	11	-19.1	0.4	-18.4	-19.5	15.3	1.8	17.0	10.8
Older adult	I	2	-19.2	0.2	-19.1	-19.3	13.3	0.9	14.0	12.7
Alava (8th-15th)	C	71	-19.1	0.8	-18.1	-19.8	8.7	0.8	9.6	7.9
Zaballa (10th-15th)	C	14	-19.8	0.7	-18.8	-21.3	9.0	0.8	10.4	7.6
Zornoztegi (12th-14th)	C	7	-18.1	1.1	-16.7	-19.9	8.3	0.6	9.2	7.5
Aistra (8-9th)	C	35	-19.0	1.0	16.7	-22.0	7.9	1.0	12.1	6.8
Treviño (12th-14th)	C	15	-19.6	0.7	-18.7	-22	9.6	1.2	12	7.5
Jaca (13th-15th)	C	25	-18.4	1.1	-15.3	-19.6	10.0	0.8	12.2	8.6
Valencia (14th-15th)	C	18	-18.4	0.6	-16.8	-19.3	10.5	1.1	11.7	8.0
Zaragoza (10th-12th)	I	36	-19.0	0.3	-18.2	-19.6	10.9	1.4	14.1	9.0
Albarracín (10th-12th)	I	31	-19.0	0.2	-18.5	-19.4	10.8	0.6	12.1	9.4
Benipeixcar (15th-16th)	I	20	-16.4	0.9	-14.2	-18.0	10.7	0.6	11.9	9.2

C., Christian; I., Islamic

fish were analyzed to establish the dietary baseline for Tauste medieval population. Small mammals were selected since present low mobility with restricted home ranges more accurately reflects the local isotopic composition baseline. The analyzed species were wood mice (*Apodemus sylvaticus*), as they are herbivorous (seed eaters), and shrews (*Crocidura russula*), which are one level higher than wood mice in the trophic chain.

To address this issue, the isotope enrichment of one trophic level has to be established. For this purpose, both faunal and human values from the nearest coeval Christian and Muslim settlements were compared (Table 4, Fig. 10). Livestock from Alava archaeological sites and Benipeixcar Muslim site displayed different average $\delta^{13}C$ and $\delta^{15}N$ values. The nitrogen values of plants and fauna are strongly influenced by local climate conditions (Ambrose, 1991). Fauna at the Alava sites, located in the northern Iberian Peninsula, displayed lower nitrogen isotope values due to a more humid climate than Benipeixcar, on the warmer and drier Mediterranean coastline.

Comparison of human values with fauna revealed the average offset between human and livestock of c. 1.5‰ in $\delta^{13}C$ and 4-4.5‰ in $\delta^{15}N$ (Fig. 11) typical of one trophic level (Drucker and Bocherens, 2004).

Table 4. Present-day fauna carbon and nitrogen isotope data (average, SD, max and min) from Tauste region and archaeofauna data from ther coeval sites of Alava and Beneipeixcar (Quirós Castillo, 2013; Mundee, 2014; Alexander et al., 2014).

Site	Specie	N	δ^{13}C (‰)				δ^{15}N (‰)			
			Mean	Std Dev	Max	Min	Mean	Std Dev	Max	Min
Tauste	Common barbel	2	-22.7	0.3	-22.5	-23	12.7	0.1	12.8	12.7
	Madrilla	1	-22.7	-	-	-	12.9	-	-	-
	Shrew	3	-19.1	2.8	-16	-21.5	12.4	2.2	14.5	10.1
	Wood mouse	3	-18.4	0.9	-18	-19.4	8.7	0.7	9.3	7.9
Alava	Cow	6	-21.3	1.1	-19.8	-22.7	4.7	1.1	6.1	3.5
	Sheep/goat	2	-19.3	0.9	-18.7	-19.9	4.6	2	6.1	3.2
	Pig	3	-21.5	0.6	-20.8	-22.0	4.1	1.9	6.1	2.3
	Horse	1	-20.1	-	-	-	6.3	-	-	-
	Dog	2	-19.2	0.7	-18.6	-19.7	8.4	1.4	9.4	7.4
	Poultry	2	-17.5	2.4	-15.8	-19.1	7.4	0.1	7.5	7.4
Benipeixcar	Cow	5	-17.8	2.8	-14.3	-20.1	7.0	1.2	8.5	5.7
	Sheep/goat	9	-19.3	0.2	-19.1	-19.5	4	0.8	5.6	2.9
	Pig	1	-17.8	-	-	-	6.6	-	-	-
	Poultry	4	-17.8	2.0	-13.3	-17.5	7.0	4.0	9.7	1.0
	Cat	3	-16.0	0.5	-15.5	-16.3	8.7	0.5	9.1	8.1
	School shark	1	-12.4	-	-	-	9.9	-	-	-

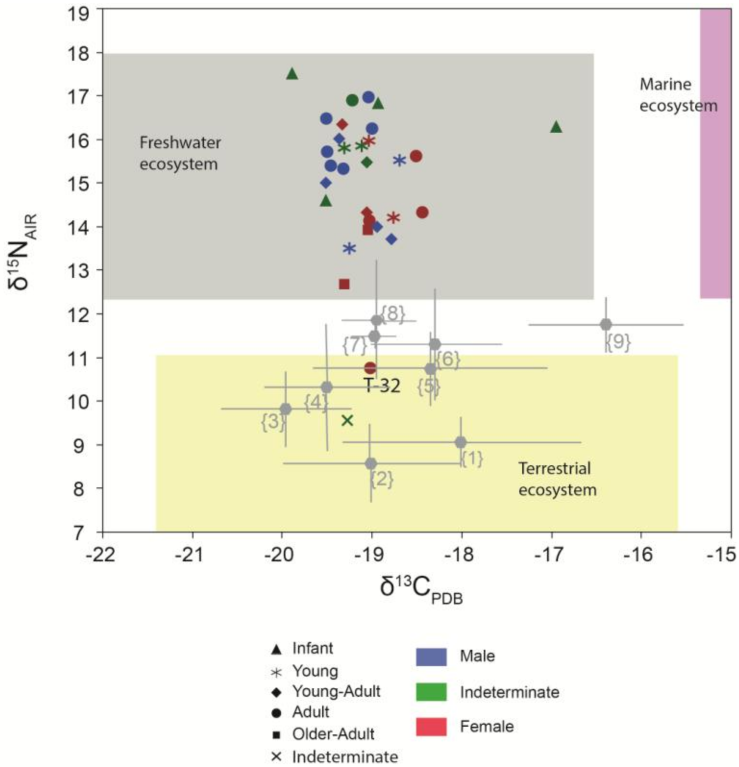

Figure 10. Plot of $\delta^{13}C$ and $\delta^{15}N$ values for the individuals at Tauste. Ecosystem boxes are based on faunal data reported by (Ambrose, 1990) and (Müldner and Richards, 2007). Field values are corrected for trophic level effect. 1-9 corresponds to coetaneous Muslim and Christian populations in the Iberian Peninsula: , (1) Zornostegui 12th-14th centuries, (2) Aistra 8th-9th centuries, (3) Zaballa 10th-15th centuries, (4) Treviño 12th-14th centuries, (5) Jaca 13th-15th centuries (6) Valencia 14th-15th centuries (7) Albarracín 10th-12th centuries (8) Zaragoza 10th-12th centuries; (9) Benipeixcar 15th-16th centuries (1)-(4) (Quirós Castillo, 2013), (5)-(8) (Mundee, 2014), (9) (Alexander et al., 2014).

In an attempt to establish the trophic level offset in Tauste, small mammal isotope values were considered. The average of wood mice values (-18.4±0.9‰ in $\delta^{13}C$ and 8.7±0.7‰ in $\delta^{15}N$) and shrew values (-19.1±2.8‰ in $\delta^{13}C$ and 12.4±2.2‰ in $\delta^{15}N$) exhibited an offset of c. -1‰

in $\delta^{13}C$ and 4‰ in $\delta^{15}N$. Considering the predictable trophic level offset, the isotope values of the Tauste population should fall within the average of c. -21±1‰ in $\delta^{13}C$ and 11±0.5‰ in $\delta^{15}N$, which is the enrichment typical of one trophic level. Such an enriched $\delta^{15}N$ baseline can be explained by regional environmental conditions. Regional aridity and the local high relative salinity led to this $\delta^{15}N$ enrichment (Heaton, 1987). In fact, chemical analysis performed in teeth from Tauste individuals showed the log (Ba/Sr) = -2.35; similar high concentrations of strontium are found in both arid/semiarid region soils and in marine food sources (Guede et al., 2017; Burton and Price, 1990). Tauste site is located far from the coastline and the possibility large-scale consumption of marine food can be discarded. Thus Ba/Sr values and the $\delta^{15}N$ enrichment can be explained by the local bedrock (gypsum and salt) and environmental factors in the Tauste region.

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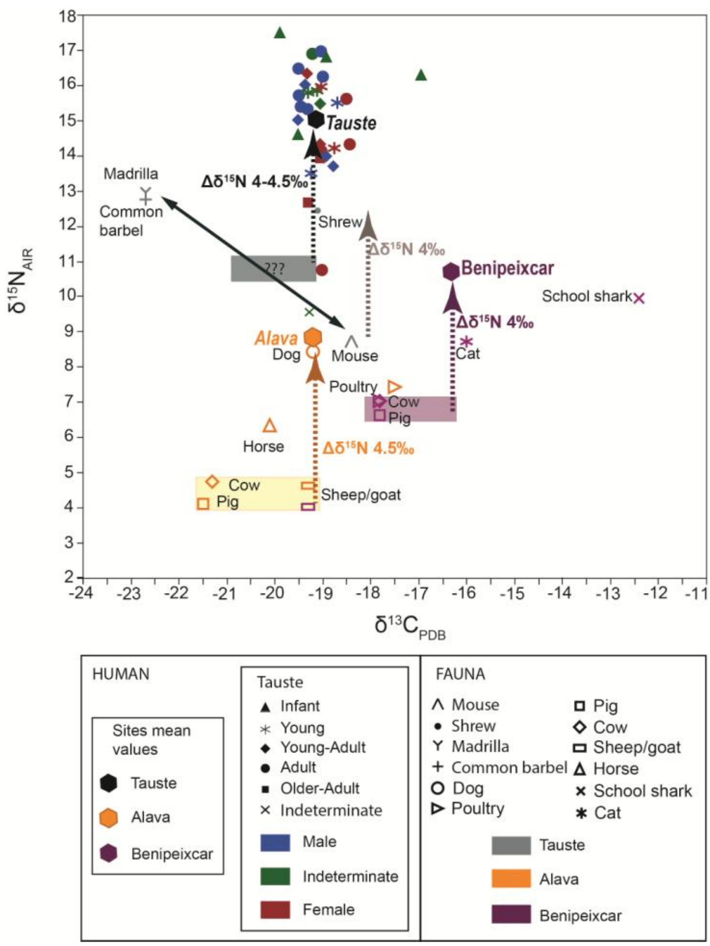

Figure 11. Plot of $\delta^{13}C$ and $\delta^{15}N$ values of present-day fauna and Medieval human at Tauste and the comparative Alava and Benipeixcar archaeofauna and human dataset (Barrett et al., 2002; Quirós Castillo, 2013). Colored rectangles indicate local carbon and nitrogen baseline. Doted arrows show the offset between local baseline and human $\delta^{15}N$ due to one trophic level enrichment. Grey rectangle indicates estimated local carbon and nitrogen baseline at Tauste.

The offset between the isotope ratios of the mice collagen (as equivalent to livestock) and the average of the expected range for human diet suggested a mixture of protein from terrestrial and freshwater sources in their foodstuffs. The relative proportion of terrestrial and freshwater resources in diet can be estimated c. 50-50% using a simple linear mixing model.

However, a particularly high consumption of freshwater fish does not appear justified from either historical or anthropological points of view. Islamic texts about the daily diet in Muslim medieval Spain indicate it was based mainly on cereals: wheat (*Tritium*), barley (*Hordeum vulgare*) and rye (*Secale cereale*), together with such C_4 grains as millet (*Pennisetum*) and sorghum (*Sorghum*), vegetables and pulses, such as chickpeas (*Cicer arietinum*), lentils (*Lens culinaris*), and peas (*Pisum sativum*), with some regional differences (García Sanchez, 1995; Salas-salvadó, 2006; Garcia Sanchez, 2002). The main sources of proteins were meat and pulses, and the type and quantity of protein consumed varied according to social status and gender. The most highly regarded meats were lamb and poultry. Pork and any animal not slaughtered in a way considered halal were excluded from the diet because Islamic law forbade it. People from lowly backgrounds consumed little meat and often made do with offal because it was cheap. Pulses such as broad beans, chickpeas and lentils constituted another source of protein and were classified as medical food. Although fish was not considered food of great dietetic value, it was part of the diet of the people from more humble backgrounds, particularly in river or coastal areas (Anderson, 2006).

Another possible interpretation for the high $\delta^{15}N$ enrichment in the Tauste population is to consider the manuring effect on plants. Since the use of manure was an advanced agricultural method introduced by Muslims in the Iberian Peninsula (Glick, 2005) all plants from manured soils showed a $\delta^{15}N$ enrichment that can be about 5‰ in cereal $\delta^{15}N$ (Fraser et al., 2011). Consequently, domestic animals foddered with manured chaff and grains will present higher $\delta^{15}N$ values. Assuming the consumption of plants enriched in $\delta^{15}N$ by the manure effect, the contribution of freshwater resources may decrease and become less than 10% of dietary protein intake.

Another interesting aspect of the reconstruction of the Tauste inhabitants' palaeodiet is the variation in isotope composition by sex and age. For the comparison, female T-32 was excluded because her strontium isotope composition indicates a return to Tauste in the last years of her life preventing $\delta^{15}N$ and $\delta^{13}C$ remodeling to the local isotope signature. The mean values of the male samples are 15.5±1.00‰ for $\delta^{15}N$ and -19.2±0.31‰ for $\delta^{13}C$. In female samples, the mean values are 14.6±1.15‰ for $\delta^{15}N$ and -19.0±0.32‰ for $\delta^{13}C$. Comparison of the results with the Student's t-test for males and females confirms the significant differences in $\delta^{15}N$ (t_{18}, $p=0.0259$).These differences suggest that females and males had different access to animal protein, probably due to the sexual division of labor (Coope, 2010). In fact, written evidence suggests the diet of Muslim females and males differed. Medical treatises make recommendations about diet, and special recommendations were made for females during pregnancy or lactation (Garcia-Sanchez, 2006). Two meals a day were recommended but in practice a smaller meal as breakfast was taken by working males. Lower dietetic needs were expected of females since female labor was restricted to domestic tasks and other activities in the household (Shatzmiller, 1994). However, the differences in nitrogen composition are related to the individuals' age rather than to their sex. When comparing $\delta^{15}N$ by age, both sexes show a statistically similar (p=0.042) mean nitrogen isotope composition, if older adult females (older than 50 years) and middle-aged adult males (ages 35–50 years) are excluded. Middle-aged adult males show higher $\delta^{15}N$ values**.** Variations between adult and younger males indicate a different dietary intake that may be related, for instance, to a lesser consumption of legumes or greater meat protein consumption.

Finally, the highest $\delta^{15}N$ values were found in children younger than 3 years, whereas children older than 4 years old have similar $\delta^{15}N$ values to those of adults. In younger infants, higher $\delta^{15}N$ values are due to a "nursing effect" indicating a diet based mainly on maternal milk.

However considering the significantly wide chronological span and the small number of radiocarbon dates, it is not possible to rule out temporal dietary differences, which is to say that there may have been variations in the diet in the course of time.

Conclusions

Isotope signatures in archaeological human remains have been used to investigate palaeomobility and the palaeodiet of the medieval Muslim population at Tauste, on the changing frontier between Muslim and Christian kingdoms in the 8th to 10th centuries.

The combination of strontium and oxygen isotope analyses was able to discriminate non-local and local populations. Although Tauste was located on the northern al-Andalus frontier most individuals were of local origin and only three females and two males were non-locals. Establishing the provenance of incoming individuals is difficult as strontium isotope ratios indicate a similar geological region. According to the oxygen isotope composition, two males would come from a warmer region while two females would come from a more mountainous geographical area. Also T-24 was of non-local origin since the different strontium values indicate a different geological provenance. Within the local population, the female T-32 stand out because she was born in Tauste, some years later she moved and lived in another geological region and then returned to Tauste a few years before she died.

As regards the medieval Muslim diet, the $\delta^{13}C$ and $\delta^{15}N$ results illustrate not only differences in diet according to sex and age but also the environmental conditions. The extremely high $\delta^{15}N$ values in Tauste population ($\delta^{15}N$ =15.0‰ on average) indicate an anomalously high $\delta^{15}N$ baseline that can be explained by the concurrence of (1) geological and environmental conditions, (2) the manuring effect on vegetables, cereals and livestock and (3) the consumption of freshwater fish. The amount of fish in the diet varies from 50% to <20% as the manuring effect increases. Significant differences were observed in $\delta^{13}C$ and $\delta^{15}N$ by sex, indicating different diets that may be related to the sexual division of labor since Muslim female work was restricted to the household. The main dietary differences between males and females were amongst adult individuals, suggesting adult males were differentially valued in medieval Muslim society and consumed more animal protein than females and young males. The lower $\delta^{15}N$ values of the elder females indicated lower protein consumption due to lesser dietary needs. In contrast, the significant higher $\delta^{15}N$ values in the children younger than 4 years could be related to the “nursing effect”.

Chapter V:

Tauste site-Trace elements study

"Analyses of human dentine and tooth enamel by laser ablation-inductively coupled plasma-mass spectrometry (LA-ICP-MS) to study the diet of medieval Muslim individuals from Tauste (Spain)"

Microchemical Journal 2017, 130: 287–294

Abstract

Trace elements have been analysed in 23 tooth enamel and dentine samples from a Muslim population in Tauste (North Spain) to investigate health and palaeodietary patterns during the 8th–10th centuries. LA-ICP-MS technique was used to determine the chemical composition of teeth. Post-burial alteration was established by REE and U high content (1 N µg/g) and several samples, mainly of deciduous teeth, were discarded. Trace elements show different behaviour in dentine and enamel related to the composition of tissues. Five individuals had high Pb contents (ranging between 2 and 30 µg/g) suggesting intoxication by occupational exposure to anthropogenic lead. Considering the period of time, individual lead intoxication could be attributed to working activity. Young individuals vs adults, and males vs females show different food intake, probably due to sexual division of labour or social status. The palaeodietary pattern of the Tauste population provides insights into this Muslim community.

Introduction

Research on residentialmobility or diet has been performed in bones and teeth through the development of macroscopic and chemical methods of analysis. In recent decades the use of laser ablation-inductively coupled plasma-mass spectrometry (LA-ICP-MS) has enabled direct analysis of solid samples using only a small amount of material and minimal sample destruction (Kang et al., 2004; Speakman and Neff, 2005). This minimal destructive nature of the laser ablation technique is essential in archaeological materials and even more so when remains are limited and must be preserved.

LA-ICP-MS provides the distribution of trace elements throughout the skeleton samples. Several factors could affect the elemental composition in bones and teeth, especially in buried samples. In addition to diet, another factor affecting the composition is the remodelling process. Bones are mainly composed of hydroxyapatite and at structural level are remodelled continuously during the individual's life. Teeth are divided into inner pulp cavity, dentine and outer enamel tissues. Dentine is a much softer, has a less mineralized structure and regenerates constantly; whereas tooth enamel is the hardest material in the human body and is not renewed after forming during childhood, thus fixing the elemental composition in an earlier stage of the individual's life (Hillson, 1996; Parfitt, 1983; Dolphin and Goodman, 200; Humphrey et al., 2008; Richards et al., 2008). In archaeology, enamel is preferred to determine the chemical composition because it is less likely to be affected by diagenesis and environmental conditions, (e.g. Khon and Cerling, 2002; Trueman and Tuross, 2002).

In archaeological samples, burial conditions can cause changes in the mineral composition leading to exchange of ions with the environment (Malleson, 1990). The diagenesis effect is crucial in an interpretation of the elemental composition of bones and teeth. The analysis of soils associated with human remains is the best approach to evaluate diagenesis. When soil composition is unknown, a different approach is used to evaluate the diagenesis effect, as the content of rare earth elements and U and Th in teeth and bones are sensitive indicators of diagenesis (Lambert et al., 2003; Zlateva et al., 2003; Carvalho et al., 2004). When diagenesis is not a problem, the chemical analysis of enamel and dentine can be directly related to the diet. Thus some elements such as sodium, magnesium, zinc, strontium, barium and lead are directly related to food consumption (Curzon and Cutress, 1983; Grupe 1998; Sandford and Weaver, 2000; Djingova et al., 2004). In a multi-elemental approach, barium, strontium, manganese and magnesium are prevalent in vegetables while zinc and copper indicate a meat-based diet. Additionally tooth chemical composition allows identification of long-term heavy metal exposure and may provide information about the exposure of an individual to toxic metals (e.g. Cd, Pb) (Chew et al., 2000; Tvinnereim et al., 2000).

This study analyses the chemical composition of human teeth to determine palaeodietary patterns of a Middle Age Muslim population in Tauste. The samples come from a Muslim necropolis where all the human skeletons face Mecca. The necropolis is the only vestige of Muslim presence in this village. The Tauste human remains were analysed in absence of any other vestiges of Islamic population, such as the Muslim village associated with the necropolis or written sources. The dietary point of view might be able to shed some light on nutrition, life history and migration during the Islamic period in the Iberian Peninsula.

Experimental methods

Material and sample preparation

Twenty-three individuals' teeth from the Muslim necropolis in Tauste were analysed. Individuals were characterized by sex and age. Thus 13 individuals are men, 7 women and 3 of indeterminate sex (Gutierrez and Pina, 2011). According to age, individuals were categorized as: 3 infants (aged younger than 12 years), 3 juveniles (ages 12–17 years), 5 young adults (ages 18–34 years), 10 adults (ages 35–50 years), and 1 older adult (older than 50 years).

Samples consisted of molar teeth with the exception of one canine tooth. One tooth from each individual was analysed to evaluate the elemental composition of enamel and dentine. Prior to analyses, teeth were repeatedly washed an ultrasonic bath for 10 min in distilled water and rinsed in ultrapure water to remove impurities from burial. Then the teeth were sliced longitudinally with a diamond disc mounted in epoxy resin to expose the whole dental structure and polished. Only sample T-12 was mounted on a polished thin-section.

Instrumentation

Analyses were carried out using quadrupole-inductively coupled plasma-mass spectrometer (Q-ICP-MS) model Thermo ×7 updated to

XSeries2 coupled to the UP213 laser ablation system equipped with Xt interface unit. To improve equipment sensitivity, a second vacuum pump at the interface of the system was used. Laser ablation system was New Wave Nd:YAG operating at a wavelength of 213 nm. Calibration of LA-ICP-MS was achieved using a standard reference glass material NIST SRM 612 (Jochum et al., 2011; Günther et al., 2001; Herwartz et al., 2013; Kowal-Linka et al., 2014; Kowal-Linka et al., 2015). Measurements were taken for the spot size of 100 µm at a distance of 300 µm from each other. Time delay between the end of LA of one spot and the initiation of LA of the next spot was 15 s. LA was performed with a laser spot diameter of 100 µm, laser fluency 4.5 J/cm2, and a repetition rate of 10 Hz. Table 1 summarizes the operating parameter used for the LA-ICP-MS measurements.

The elements analysed were Na, Mg, Al, Sc, Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, Rb, Sr, Y, Ba, Pb, Th, U and the rare earth elements (La-Lu). Data collection was performed by rapid peak-hopping (5–30 ms) dwell time (Table 1) between selected isotopes of each analyte elements for a period of 60 s. A background signal was collected during the first 30 s of analysis. The laser was fired for 60 s from which the middle 30 s were used for signal integration. For data reduction, Iolite 3 data processing software package for time esolved mass spectrometry data was used (Paton et al., 2011; Paul et al., 2012). The background from the "gas blank" is used to background subtract the gross analyte data. The concentration of element, even though calculated directly with the Iolite 3 software, can also be obtained by defining the intensity integration point for the background and the sample analysed by laser. The 43Cawas used as an internal standard to account for variation in ablation efficiency, which is caused by variations in the mass of the material ablated. The internal standard was used to correct for multiplicative effect of matrix and more importantly the different volume of teeth tissues ablated. Calcium concentration was assumed from the stoichiometry of biogenic hydroxyapatite. The estimated calcium content is 252 mg/g for the dentine and 360 mg/g for the enamel tissues (Kohn et al., 1999).

Table 1. Laser ablation and ICP-MS operating conditions.

ICP-MS	
Q-ICP-MS	Thermo Fisher Xseries-I
Forward Power	1400 W
Gas Flows	
Coolant (plasma)	Ar: 14 L/min
Auxiliary	Ar: 0.5 L/min
Carrier Gas	He: 0.8 L/min
Make up gas	Ar: 0.9 L/min
Analysis protocol	
Scanning mode	Peak hopping, 1 point per peak
Acquisition mode	TRA (Time Resolved Analysis)
Analysis duration	90 s (30 s background, 60 s signal)
Dwell times:	
^{43}Ca	5 ms
^{23}Na, ^{24}Mg, ^{88}Sr, ^{137}Ba	10 ms
^{27}Al, ^{45}Sc, ^{47}Ti, ^{51}V, ^{52}Cr, ^{55}Mn, ^{56}Fe, ^{59}Co, ^{60}Ni, ^{65}Cu, ^{66}Zn, ^{85}Rb, ^{89}Y, ^{139}La, ^{140}Ce, ^{141}Pr, ^{146}Nd, ^{147}Sm, ^{153}Eu, ^{157}Gd, ^{159}Tb, ^{163}Dy, ^{165}Ho, ^{166}Er, ^{169}Tm, ^{172}Yb, ^{175}Lu, ^{208}Pb, ^{232}Th, ^{238}U	30 ms
LA	
Model and manuacture´s name	UP213
Wavelength	213nm
LA system	New Wave, Nd:Yag
Laser energy	0.353 mJ
Laser frequency	10Hz
Spot size	100 µm
Average fluence	4.5 J/cm^2

The mean and standard deviation of repeat analyses of a matrix matched (Durango apatite), in-house standard indicate that analytical reproducibility was 2–6% (Table 2). The accuracy for the analysis was further indicated by the close agreement between results for the Durango apatite and determination made by ICPMS (typically <5–10%; see Table 2). The analytical accuracy is similar to reported by Trotter and Eggins (2006).

Detection limits (LOD) for each element and spot was determined based on Longerich et al. (1996) and are reported in supplemental file S1. The equation used to calculate these detection limits was the following:

$$OD = \frac{3\sigma_{BCG}}{SxY}\ x\ \sqrt{\frac{1}{N_{BCG}} + \frac{1}{N_{PK}}}$$

where σBCG is the standard deviation of replicate analysis of the pre-ablation background identification; NBCG and NPK are the number of replicate determinations used for background and peak signal integration, respectively; S is the normalized sensitivity in cps per unit of concentration for the reference material; y the ablation yield relative to the reference material, determined from the counting intensity measurement and the known concentration of the integral standard.

Table 2. Analysed elements and isotopes, NIST 612 values and results obtained for Durango apatite.

Element (µg/g/*wt.%)	Isotope (amu)	NIST 612	Durango	Std. dev. (n=43)	LOD
Na_2O*	23	13.70	1059.23	52.96	4.87
Mg	24	68.00	138.25	3.71	1.94
Al_2O_3*	27	2.03	nd	nd	4.79
Sc	45	39.90	0.70	0.32	0.49
Ti	49	44.00	62.92	6.70	2.83
V	51	38.80	40.26	1.79	0.43
Cr	52	36.40	nd	nd	0.57
Mn	55	38.70	72.62	2.43	0.38
Fe	57	51.00	251.00	30.37	0.99
Co	60	35.50	0.07	0.02	0.08
Ni	60	38.80	1.84	0.75	1.52
Cu	63	37.80	0.65	0.06	0.89
Zn	66	39.10	0.52	0.36	0.35
Rb	85	31.40	0.04	0.01	0.07
Sr	86	78.40	461.20	11.23	0.21
Y	89	38.30	560.89	28.15	0.20
Ba	137	39.30	1.49	0.13	0.05
La	139	36.00	3741.09	161.17	1.01
Ce	140	38.40	4096.70	97.48	1.15
Pr	141	37.90	311.24	8.85	0.09
Nd	146	35.50	1024.51	40.32	0.35
Sm	147	37.70	141.83	6.47	0.06
Eu	153	35.60	14.69	0.57	0.01
Gd	158	37.30	126.66	7.22	0.06
Tb	159	37.60	15.37	0.80	0.01
Dy	163	35.50	90.35	4.88	0.04
Ho	165	38.30	17.82	0.98	0.01
Er	166	38.00	48.75	2.84	0.02
Tm	169	36.80	6.17	0.35	0.01
Yb	174	39.20	34.21	1.95	0.02
Lu	175	37.00	4.33	0.26	0.00
Pb	208	38.57	0.47	0.02	0.01
Th	232	37.79	196.41	12.26	0.06
U	238	37.38	7.98	0.20	0.00

Analyses were performed in both dentine and tooth enamel. Elemental composition of teeth varies in concentration of ppm or µg/g. Spot ablation experiments consisted of spots arranged in line scanning covering the enamel and dentine (Fig. 1)

Pre-ablation analyses in order to explore tooth enamel and dentine structure in several samples were examined using a scanning electron microscope (JEOL JSM-6400) fitted with a backscatter detector operating at 15 kV. Specimens were sputter coated with a thin layer of carbon. The analyses were performed in the Materials and Surface Unit of the Advanced Research Facilities (SGIker) in the Basque Country University (UPV/EHU).

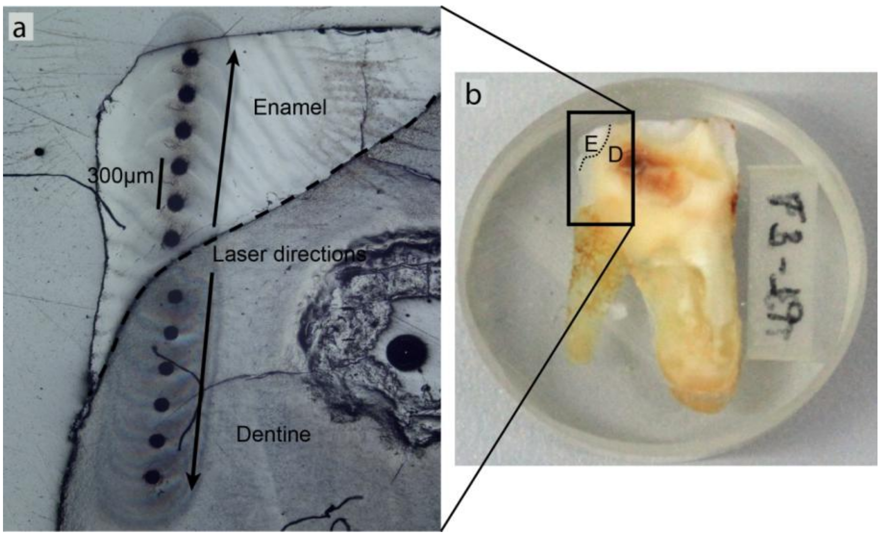

Figure 1. Transverse tooth section showing analytical locations: (a) Microphotographs showing LA-ICP-MS laser spots locations. Black dots indicate laser ablation spots in the enamel and dentine. (b) Photograph of teeth showing outer enamel (E) and dentine (D) tissues. Square delimits the analytic area.

The correlation matrix of dental tissues was examined in order to distinguish differences in the tooth contents. The data were compared using SPSS v.20 software (Statistical package for Social Sciences). Differences between sample groups were determined by means of an unpaired Student's t-test. Statistical significance was accepted as $p < 0.05$. The multivariate analysis principal component analysis (PCA) was used for discrimination of whole tooth samples and for the discrimination of the different tooth tissues separately.

Results and discussion

Rare earth elements (REE) and uranium (U) contents are regarded as sensitive tracers of diagenetic modification. During burial, U and REE substitute OH and Ca in bioapatite attesting alteration to other chemical constituents (Kohn et al., 1999; Kolodny et al., 1996; Reynard et al., 1999; Trueman, 2004). Diagenetically unmodified modern teeth have REE and U contents lower than 1 µg/g. Samples T-16, T-33, T-34 and T-39, with REE and U concentrations above 1 µg/g, were excluded for the data discussion. Only sample T-16 has high REE and U contents both in dentine and enamel whereas samples T-33, T-34, T-39 and T-40 have high U contents. Samples T-16, T-33 and T-40 correspond to deciduous teeth and samples T-34 and T-39 how defects of enamel as carious lesions and external signs of erosion (Fig. 2). High contents of REE and U in deciduous teeth indicate that infant teeth must be excluded in palaeonutrition studies.

Table 3 summarizes mean contents of quantified elements in the Tauste individuals' teeth including the samples discarded for the discussion. All analytical results are included in supplementary file S2. In dentine the quantified elements were Na, Mg, Al, Sc, Ti, V, Cr, Mn, Fe, Co, Cu, Zn, Rb, Sr, Ba and Pb whereas in enamel only Na, Mg, Sc, Ti, Mn, Fe, Zn, Rb, Sr, Ba and Pb have contents above the quantifying limit. Differences in element content and chemical composition result from differences in the structure and composition of enamel and dentine tissues. Enamel is formed by 96% calcium hydroxyapatite, 0.5% organic material and water while dentine consists of 70% inorganic hydroxyapatite, 20% organic material (mainly collagen) and 10% water (Cate, 1998). Enamel has a strongly mineralized structure preventing element migration whereas dentine is more porous and more permeable to element diffusion favouring chemical remodelling (Castro et al., 2010). Most elements exhibit a concentration in enamel and dentine below 1 µg/g, including most transition elements REE, Th and U. Elements with concentrations above 1 µg/g include Fe, Pb and Ba with contents of 2 to 5 µg/g on average, Ti and Zn ranging between 40 and 70 µg/g, and Na and Mg with contents between 3300 and 5800 µg/g. Mg, Ti and Zn concentrations are slightly enriched, Sr, Ba and Pb are highly enriched whereas Na and Fe are depleted in entine more than in enamel.

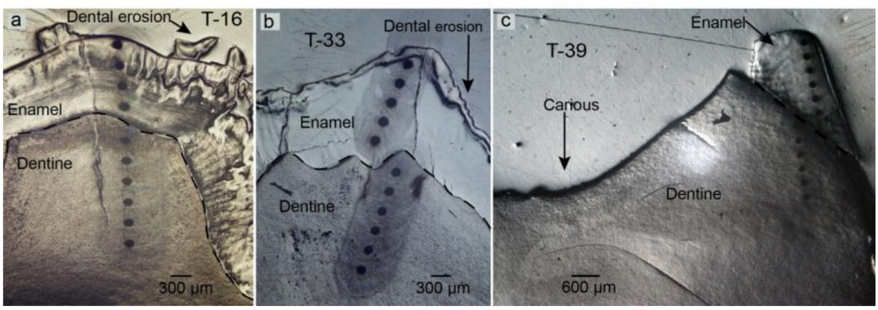

Figure 2. (a, b) Microphotographs of teeth showing external signs of erosion and (c) microphotographs of teeth showing carious lesions.

Table 3. Average of element concentration (in µg/g) of dental tissues determined by LA-ICP-MS.

Sample	Tissue	n	Sex	Age	Na	Mg	Al	Sc	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	Rb	Sr	Y	Ba	Pb	U
T9	Enamel	4	F	Mature	5097.0	3611.8	<16.8*	<1.6*	50.1	<1.5*	<1.1*	<1.4*	<1.6*	<0.2*	<3.3*	<1.6*	23.1	<0.2*	338.8	<0.03*	1.6	0.6	<0.004*
T11	Enamel	3	M	Adult	4856.7	3135.7	<11.9*	1.1	53.3	<1.2*	<1.0*	1.5	4.1	<0.1*	<1.9*	<1.6*	87.7	<0.1*	177.1	<0.04*	1.1	1.8	<0.001*
T12	Enamel	6	M	Adult	5433.0	3364.0	<4.3*	0.7	77.5	<0.4*	<0.6*	0.6	2.7	<0.1*	1.0	<1.0*	66.7	0.1	315.5	<0.01*	1.3	5.9	<0.001
T15	Enamel	6	F	Adult	5451.7	3689.7	<6.8*	<0.8*	70.8	<0.5*	<10*	0.6	2.1	<0.1*	<1.6*	<1.4*	25.4	<0.1*	350.5	<0.02*	0.6	0.1	<0.001*
T16	Enamel	13	Inde	Infant	6411.9	3035.5	<4.6*	0.5	52.6	0.6	0.7	1.7	73.8	0.3	1.7	2.8	58.5	<0.1*	952.4	0.2	9.5	1.5	14.2
T17	Enamel	5	M	Adult	5403.4	3578.4	<15.1*	1.1	56.0	<1.2*	<0.7*	<1.1*	<2.2*	<0.2*	<5.4*	<2.6*	24.1	<0.1*	277.4	<0.03*	0.6	1.2	<0.001*
T19	Enamel	6	F	Adult	5651.3	3981.3	<9.5*	<1.3*	64.9	<0.9*	<1.1*	<0.8*	40	<0.2*	4.1	<2.0*	24.9	0.1	137.1	<0.03*	1.1	0.1	<0.01*
T24	Enamel	6	F	Young adult	4221.0	2187.8	<4.9*	0.6	61.5	<0.4*	<0.5*	1.1	4.5	<0.1*	<1.1*	<0.9*	307.5	0.1	300.4	<0.01*	0.5	0.2	<0.002*
T26	Enamel	6	M	Young adult	5597.2	3149.7	<7.1*	0.8	65.2	<0.5*	<0.9*	0.7	1.8	0.1	<2.1*	<2.1*	39.8	<0.1*	337.9	<0.02*	0.2	0.2	<0.001*
T28	Enamel	6	M	Young	5648.3	3536.0	<8.1*	1.2	72.4	<0.6*	<0.8*	0.4	1.9	<0.1*	<2.9*	0.5	21.4	<0.2*	317.9	<0.04*	0.5	0.3	<0.001*
T30	Enamel	6	F	Adult	5878.3	3442.2	<5.7*	1.0	70.0	<0.4*	<0.5*	0.5	1.5	0.1	1.7	<1.1*	20.4	0.1	320.3	<0.02*	0.6	15.7	<0.001*
T31	Enamel	7	F	Young	6710.0	3015.1	<5.3*	<0.4*	59.2	<0.4*	<0.6*	1.1*	3.7	<0.1*	<1.7*	<1.0*	46.6	0.1	332.6	<0.01*	0.4	0.1	<0.001*
T33	Enamel	5	Inde	Infant	5641.2	3900.8	<3.9	<0.3*	65.9	0.5	<0.5*	0.8	3.4	<0.1*	<1.3*	<0.8*	31.9	<0.1*	382.6	<0.01*	2.2	0.3	0.9
T34	Enamel	7	M	Young	6418.6	3944.6	<5.2*	0.7	64.4	<0.4*	<0.5*	<0.4*	1.6	0.1	<2.4*	0.5	21.9	0.1	286.2	<0.02*	2.2	6.8	<0.003*
T39	Enamel	6	M	Adult	5901.5	3121.2	<5.8*	<0.5*	47.9	<0.5*	<0.6*	0.6	2.9	<0.1*	<1.6*	<1.0*	56.4	0.1	316.1	0.1	0.5	0.2	<0.001*
T13	Enamel	6	M	Adult	5831.5	3359.7	<6.0*	0.5	66.3	<0.5*	<0.8*	2.2	1.8	0.1	<1.8*	<1.0*	23.9	<0.1*	392.1	<0.02*	2.0	0.2	0.3
T18	Enamel	6	M	Young adult	6750.0	4002.0	<5.3*	0.3	59.6	<0.3*	<0.5*	0.4	1.2	<0.1*	<1.5*	<0.8*	25.9	<0.1*	360.8	<0.01*	1.2	0.3	<0.01*
T27	Enamel	5	M ?	Young	4607.4	2820.2	<11*	0.9	57.6	<0.7*	<1.3*	0.8	5.5	0.1	<2.1*	<1.3*	57.7	<0.1*	282.1	<0.02*	0.8	0.8	<0.001*
T32	Enamel	6	F	Adult	6022.2	3604.3	<6.7*	1.1	65.4	<0.6*	<0.9*	1.1	1.9	<0.1*	<2.3*	<1.2*	23.8	0.1	410.5	<0.02*	1.4	0.1	<0.001*
T40	Enamel	5	Inde	Infant	5316.0	3091.2	<3.9*	<0.3*	59.1	<0.3*	<0.5*	0.7	2.3	<0.1*	<1.7*	<0.8*	40.2	<0.1*	248.4	<0.01*	0.8	0.5	0.1
T41	Enamel	6	M	Young adult	6038.0	3119.5	<3.2*	<0.4*	56.4	<0.3*	<0.4*	<0.3*	2.8	<0.1*	<1.0*	<0.8*	30.0	0.1	288.7	<0.01*	0.3	0.1	<0.001*
T42	Enamel	7	M	Adult	6189.7	3280.3	<4.8*	<0.4*	60.0	<0.4*	<0.6*	<0.3*	2.4	<0.1*	<1.5*	<1.0*	24.8	<0.1*	387.3	<0.01*	0.4	0.5	<0.001*
T44	Enamel	7	M	Young adult	6596.7	3264.0	<5.3*	<0.3*	66.6	<0.4*	<0.5*	<0.3*	4.9	<0.1*	<1.6*	<1.0*	28.4	0.1	391.8	<0.01*	0.9	0.1	<0.001*

Elements content in µg/g. M: male; F: female; Inde: indeterminate. * Element is not determined and reported the LOD

During the analyses of sample T-12, dentine was completely perforated by the laser and caused chemical contamination in some elements. Hence most elements show outlier anomalous contents and sample T-12 dentine contents were excluded from the discussion

Of the analysed elements, only biofunctional elements were chosen because they are commonly found in teeth tissues at concentrations about the detection limit. Na, Mg, Sr, Zn, Fe, Ba are considered biofunctional elements because they are indispensable to maintain the physiological functions of the human body (Fraga, 2005). Moreover biofunctional elements have applications in the monitoring of paleodiet patterns. Laser spots of biofunctional elements are compared in Fig. 3. Line traverses illustrate pronounced zoning between enamel and dentine. In general, dentine has lower Zn, Ba and Sr contents and a higher Na content than enamel. Mg displays random behavior between samples. In most cases (61% of samples) Mg content is higher in enamel whereas in 39% it is lower. This variable trend was not related to either age or sex of individuals and the reasons still remain unclear.

The concentration of biofunctional elements is related to food products and therefore human dietary intake, allowing the determination of palaeodietary patterns. The average Zn content in Muslim teeth from Tauste was low: 72 ± 14 µg/g in dentine and 28 ± 13 µg/g in enamel, indicative of possible nutritional Zn deficiencies. The Zn concentration < 90 µg/g in children's teeth could be due to borderline Zn bioavailability (Tvinnereim et al., 1999).

The young adult female T-24 has an anomalously high Zn content in enamel (308 µg/g) while the Zn content in dentine is similar to the average. High Zn content denotes a diet rich in marine products (like fish and/or shellfish) and according to Sr isotope data suggests nonlocal origin (Guede et al., 2016). The average Fe content in dentine and enamel is similar, 2.4 ± 0.8 µg/g and 2.8 ± 1.4 µg/g, respectively. However females have a lower Fe content in dentine than males (2.2 ± 0.6 µg/g and 2.7 ± 0.9 µg/g, respectively). The iron concentration was found to be lower in females as a result of the burden of blood loss during menstruation (Tanaka et al., 2004). The average Na content is higher in enamel (5813 ± 886 µg/g) than in dentine (3584 ± 666 µg/g), whereas the average Mg content is lower in enamel (3394 ± 510 µg/g) than in dentine (4411 ± 2435 µg/g). Similar Mg values were found in modern human teeth while Na contents are two folds lower in the Tauste individuals (Cakir et al., 2011; Teruel et al., 2015).

Four individuals (samples T-30, T-34, T-17 and T-11) exhibited anomalously high contents of lead (Table 3). Such high concentrations suggest poisoning that may be due to continuous exposure to anthropogenic lead. In fact in the Middle Ages, Islamic potters introduced lead-glazed pottery into Muslim Spain (Al-Andalus) and developed the Hispano-Moresque style (Mery, 2006). Examples of lead-glazed pottery were found in the nearby Muslim village of Tudela (Al-Tutili) (Zuluaga et al., 2012). Thus high lead values for these five individuals could suggest poisoning related to pottery making

Na, Mg, Sc, Ti, V, Cr, Mn, Fe, Co, Cu, Zn, Rb, Sr, Ba and Pb content was subjected to correlation analysis to determine relationships within enamel and dentine. Table 4 shows significant ($p < 0.05$) correlation coefficients between element concentrations in dentine and enamel. Since the chemical composition of dentine is remodeled during an individual's lifetime (Parfitt, 1983), correlations between element concentrations would reflect the dietary patterns of the Tauste population. Significant positive correlation ($p < 0.01$) was observed between Mg, Zn, Na, Ba and Sr concentrations. In contrast, in enamel, significant positive correlation ($p < 0.01$) was observed between Na, Mg, Sr, Ba, Mn, Fe, Co and Zn. These chemical differences are due to the teeth recording two different times in the life of individuals. Enamel records diet during adolescence when enamel is formed whereas dentine reflects the diet in the last years of an individual's life.

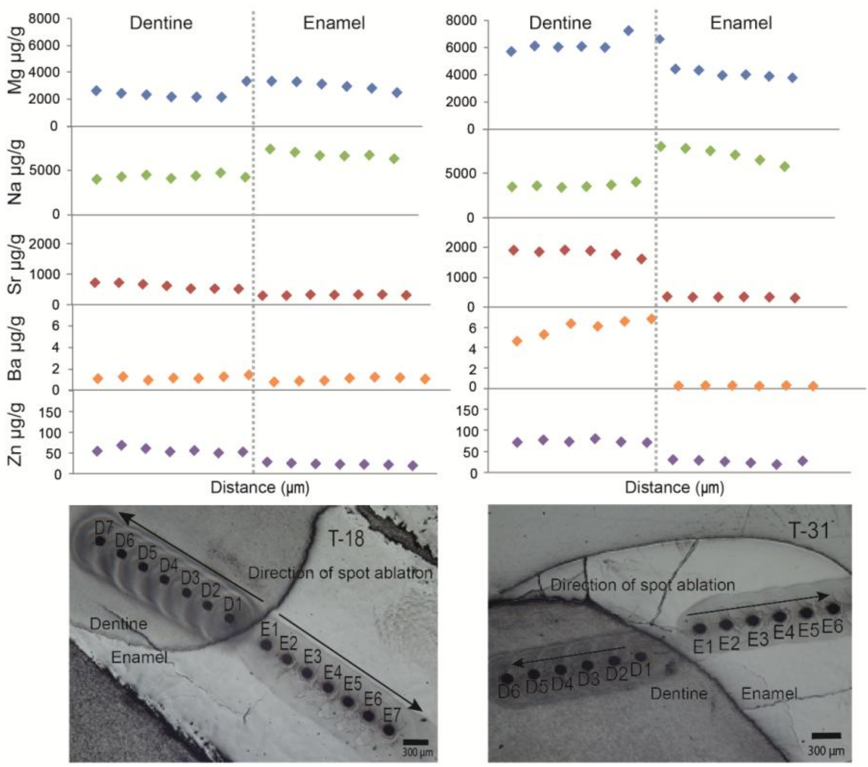

Figure 3. Laser spot ablation of two representative teeth showing biofunctional element content variations within and between tissues. The microphotographs of teeth showing the laser spot directions and positions.

Principal component analysis defines new variables (or principal components) as linear combinations of the original variables in order to explore the variance of the data and ignore the grouping of the samples (non-supervised analysis). Principal component analysis of previously evaluated variables and 17 tooth samples (excluding 3 infant samples and 5 diagenetically modified samples) was performed considering only the following biofunctional elements: Mg, Zn, Na, Ba, Sr, Fe, and Cu to discriminate dietary patterns between individuals. Mixing values are excluded according to the excluded cases pairwise option in SPSS package program. Since dentine and enamel record different times of life, the chemical data were grouped according to tissue.

A PCA model for enamel tissue was not built due to the lack of analytical data for these elements to infer a robust explanation.

Combining these variables in dentine samples the first four components accounted for 77.4% of the total variance. The first component (PC1) was defined by Mg and Na (positive loading) and Ba and Sr (negative loading), located at both ends of the factor and explains 45% while Zn, Cu and Fe show positive loading on the second component (PC2) explaining 18.5% of the total variance. Fig. 4 shows score and loading plots for the two principal components (63.5% explained data).

Table 4. Correlation coefficients for chemical elements in enamel (upper-right, in light blue boxes) and dentine (lower-left, in white boxes).

	Na	Mg	Al	Sc	Ti	V	Cr	Mn	Fe	Co	Cu	Zn	Rb	Sr	Y	Ba
Na		0.527**(104)		-0.484**(29)					-0.669**(47)			-0.368**(99)		0.494**(99)		
Mg	0.556**(102)								-0.561**(47)			-0.390**(99)	0.589*(16)			0.292**(99)
Al																
Sc	-0.309**(98)	-0.419**(98)														
Ti	0.750**(102)	0.304**(102)														
V	-0.266*(67)	-0.279*(67)	-0.643*(10)		-0.380**(67)											
Cr	-0.724**(27)	-0.503**(27)			-0.732**(27)	0.647**(24)										
Mn							-0.391*(27)									0.789**(99)
Fe					-0.226*(102)							0.667**(47)		-0.485**(47)		
Co	-0.464**(84)				-0.447**(84)	-0.438**(59)			0.309**(84)			0.990**(5)				
Cu						0.362*(37)										
Zn		0.270**(102)	0.698**(16)		-0.208*(102)	-0.344**(67)	-0.548**(27)							-0.250*(99)		
Rb		0.346*(40)							-0.489**(40)		0.682**(15)			-0.814**(16)		
Sr	-0.420**(102)	-0.870**(102)		0.243*(98)	-0.318**(102)	0.530**(67)	0.616**(27)					-0.249*(102)	-0.542**(40)			0.207*(99)
Y	0.406**(47)	0.649**(47)						-0.432**(43)						-0.627**(47)		
Ba	-0.573**(102)	-0.820**(102)		0.507**(98)	-0.265**(102)	0.315**(67)	0.430*(27)					-0.332**(102)	-0.375*(40)	0.736**(102)	-0.420**(102)	

** Correlation coefficients significant at the 0.01 level

* Correlation coefficients significant at the 0.05 level.

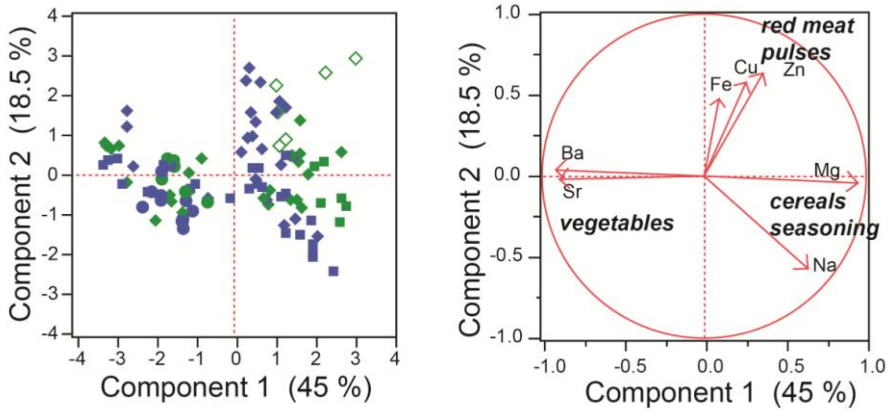

Figure 4. Score and loading plots for the PCA model of biofunctional elements (Mg, Na, Ba, Sr, Zn, Cu) in both tooth tissues. Symbols correspond to the individuals' age. Circle: young, square: young adult, diamond: adult and, empty diamond: mature. Colours correspond to the individuals' sex. Green: female and blue: male. The score plot with imputation for the mixing values.

The loading plot shows the correlated variables, and the biofunctional elements display a strong correlation between groups of elements that might be attributable to different food groups. Sodium is an important component of seasoning and can be attributed to the addition of edible salt during cooking. Apart from seasoning, Na is an important component in bread. Magnesium is abundant in cereals. Strontium and barium are considered indicators of a vegetal contribution in the diet. Zinc and iron are used as markers of a red-meat biased diet (Leblanc et al., 2005; Avegliano et al., 2011).

An interesting point emerged from the score plot, when samples were grouped according to the diet and age of individuals (Fig. 4). The score plot of PC1 versus PC2 displayed distinct clustering containing samples characterized by similar dietary pattern. Young individuals were grouped in the left part of the diagram showing enrichment in Sr and Ba, indicative of a large vegetable component in their diet. Most adults are grouped in the right part of the diagram indicating a diet based on cereals and red meat. Within the adults group, two trends can be identified according to the sex of individuals. Females shift towards Cu and Zn components. A conventional interpretation of these data suggests that females had a diet with a larger intake of meat than men. High contents of Zn and Cu can also be found in legumes (Leblanc et al., 2005) and thus a large intake in vegetal protein and cereals could explain the female scatter. In general, during the Middle Ages, in both Christian and Muslim communities, women's nutrition was worse than men's, probably due to sexual division of labour or related to social status (Reitsema et al., 2010). To assume women consumed more meat than men would lead to an erroneous interpretation of the diet. A relatively greater consumption of cereals and vegetables by women may explain the observed female shift towards the Cu and Zn component in the PCA plot. Moreover, larger consumption of meat by females is not in accord with the unpublished nitrogen isotope results that indicate lower consumption of animal protein with respect to males (Guede et al., 2016).

In order to elucidate whether the observed differences between females and males are significant and indicative of different diet, linear discriminant analysis has been performed. The score plot for the samples in Fig. 5 demonstrates discrimination according to sex and diet. These dietary differences between Tauste populations suggest unequal access to animal protein between males and female.

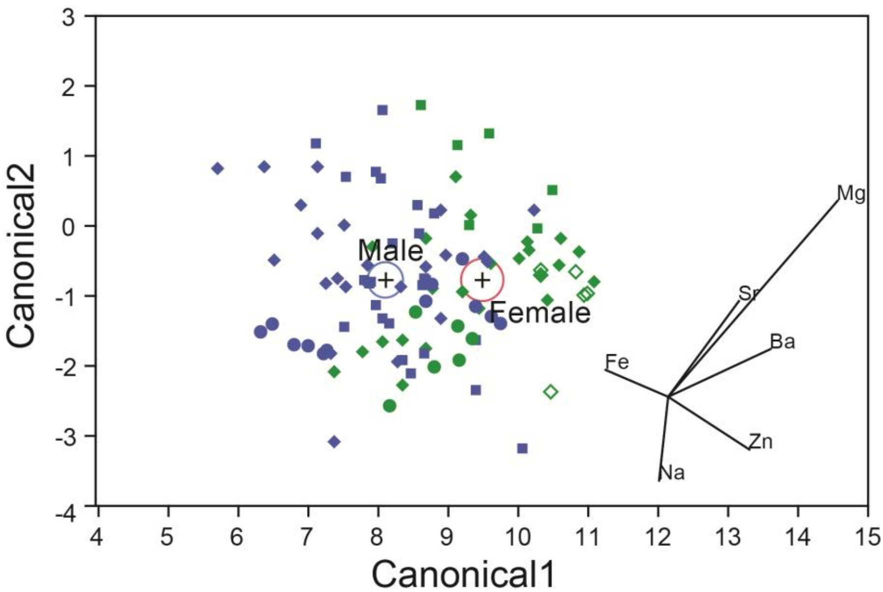

Figure 5. Scores plot of discriminant analysis for Tauste adult individuals based on concentration of biofuntional elements and using as input a priori male and female groups. Colours correspond to the individuals' sex. Green: female and blue: male. Symbols correspond to the individuals' age. Circle: young, square: young adult, diamond: adult and, empty diamond: mature.

Conclusion

LA-ICP-MS is an effective technique for the direct analysis of ancient human teeth and is especially recommended to investigate palaeodiet. In addition, its capacity for multi-elemental analysis offers an excellent power of discrimination to classify samples.

The chemical composition of infant teeth shows high REE and U contents indicative of burial modifications so these samples have to be excluded for the palaeonutritional studies.

The chemical composition of the teeth of Tauste individuals highlights significant differences between the dentine and enamel. These chemical differences are due to the different composition of the tissues. Only dentine is remodelled during the individual's lifetime and reflects dietary habits and health.

Four individuals have extremely high contents of lead indicating poisoning due to continuous exposure to anthropogenic lead.

Variations in chemical composition were observed according to sex and age and are directly related to food intake. Adults enjoyed a larger consumption of animal proteins and males more than females.

Dietary differences were probably due to sexual division of labour or related to the social status of the Tauste Muslim population.

Conclusions

1. Strontium and oxygen isotope composition of human bone and tooth remains has been used to determine local and non-local individuals and thus to reconstruct residential mobility in some rural populations in the north of the Iberian Peninsula.

   After the collapse of the Roman Empire, during the Early Medieval period a profound transformation affected territorial organization and large numbers of individuals are supposed to have migrated. Nevertheless, the isotope composition suggests that residential mobility was limited at all the studied archaeological sites. Only few individuals were of non-local origin and the provenance area was para-local from nearby areas, or was impossible to determine. Males moved to obtain better economic opportunities and the possibility of improving their status and females would move for patrilocal marriages.

2. In order to reach robust conclusions about human diet, the local isotope baseline must be established because the carbon and nitrogen isotope composition depends greatly on the climate. Nevertheless, it has seen that establishing the local baseline can sometimes be problematic either because of livestock trade and transhumance, as in Las Gobas site, or because of the absence of archaeozoological data as at the Tauste site.

   The results of the present study show that the local baseline differs between the studied archaeological sites, which reveals the importance of establishing the baseline. In the case of Tauste, two local species of small mammals and freshwater fish were analysed to establish the dietary baseline and present-day large mammals were discarded because livestock are fed with non-local resources.

3. The studied rural populations consumed an omnivorous diet consisting of vegetables and cereals mainly of C3 plants, such as wheat, barley and legumes, and a low animal protein intake from livestock, mainly pigs and sheep/goats. In the case of the Muslim population at Tauste, the consumption of pork was forbidden by religion and the main sources of proteins were lamb and poultry, and pulses. The consumption of freshwater fish or marine resources cannot be discarded as part of the diet of the population. Additionally, in Las Gobas and San Juan de Momoitio some millet consumption has been observed although in specific times. Millet was generally regarded as a low-status foodstuff by medieval society, particularly in rural settlements, but the results may be complicated by the fact that the consumption of millet could be direct or indirect through consumed fauna.

4. Trace element analyses in teeth were used to investigate palaeodietary patterns, additionally to carbon and nitrogen isotope composition. The laser ablation-inductively coupled plasma-mass spectrometry (LA-ICP-MS) technique allows direct analysis, obtaining the distribution of trace elements throughout the teeth samples, and offers an excellent power of discrimination to classify samples. Na, Mg, Sr, Zn, Fe, Ba are considered biofunctional elements because they are indispensable to maintain the physiological functions of the human body. The concentration of biofunctional elements is

related to food products and therefore human dietary intake, allowing the determination of palaeodietary patterns.

It was only possible to perform the LA-ICP-MS analyses on the Tauste samples and the results show a strong correlation between groups of elements that might be attributable to different food groups. Magnesium is abundant in cereals. Strontium and barium are considered indicators of a vegetal contribution in the diet. Zinc and iron are used as markers of a red-meat biased diet. LA-ICP-MS analyses and isotope composition indicate similar palaeodietary patterns. However, trace element analysis also allows ancient individuals' health to be investigated and within the Tauste population five individuals had high Pb contents, suggesting intoxication attributed to working activity.

5. Palaeodietary patterns provide information about the food consumed but can also illustrate the basic dynamics of medieval social life. Not only C3 vs C4 plant consumption, which represents social status and structuration, but also animal protein consumption.

   In the same way as in other coetaneous Peninsular and European sites, the studied sites do not show the same pattern of consumption habits with respect to the age and sex of the individuals. The rural communities at Las Gobas and San Juan de Momoitio do not display dietary differences according to the individuals' sex or age, while in Tauste significant differences observed between males and females may be related to the sexual division of labour since Muslim female work was restricted to the household. Adult males consumed more animal protein than females and young males, which suggests that adult males were the most privileged members of society in the medieval Muslim world.

The repetitive pattern of an increase in $\delta^{15}N$ values in children younger than 4 years of age at all sites reflects the nursing and weaning effect.

6. Multi-isotope studies including large number of individuals from several communities are able to attain a better understanding of the way of life in the rural world in the Middle Ages. The foodstuffs and dissimilarities in consumption habits according to sex and age inform about social dynamics within the communities (Christian and Muslim) and allow the reconstruction of regional identities.

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Anexo 1: Published Papers

LA Ortega, I Guede, MC Zuluaga, A Alonso-Olazabal, X Murelaga, J Niso, M Loza, JA Quirós Castillo. Strontium isotopes of human remains from the San Martín de Dulantzi graveyard (Alegría-Dulantzi, Álava) and population mobility in the Early Middle Ages. Quaternary Internacional (2013) 303: 54-63.

I Guede, LA Ortega, MC Zuluaga, A Alonso-Olazabal, JLSolaun, A Azkarate, Iban Sanchez. Isotopic evidences for reconstruction of diet and mobility during village genesis during Medieval Ages in northern Spain: Las Gobas site (Burgos, north Spain). Archaeological and Anthropological Sciences (2017)

I Guede, LA Ortega, MC Zuluaga, A Alonso-Olazabal, X Murelaga, M Pina, FJ Gutierrez, P Iacumin. Isotope analyses to explore diet and mobility in a medieval Muslim population at Tauste (NE Spain). PlosOne (2017) doi.org/10.1371/journal.pone.0176572.

I Guede, MC Zuluaga, LA Ortega, A Alonso-Olazabal, X Murelaga, M Pina, FJ Gutierrez. Analyses of human dentine and tooth enamel by laser ablation-inductively coupled plasma-mass spectrometry (LA-ICP-MS) to study the diet of medieval Muslim individuals from Tauste (Spain). Microchemical Journal 130 (2017) 287–294.

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Strontium isotopes of human remains from the San Martín de Dulantzi graveyard (Alegría-Dulantzi, Álava) and population mobility in the Early Middle Ages

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ABSTRACT

Strontium isotope analysis of human remains from San Martín de Dulantzi (Alegría-Dulantzi, Álava, Spain) graveyard has been used to establish mobility patterns during the Early Middle Ages. Some archaeological human remains had Germanic grave goods. Through radiogenic strontium isotope analysis, local origin individuals and immigrants were differentiated. Archaeological human bone samples exhibit $^{87}Sr/^{86}Sr = 0.70779–0.70802$ values similar to domestic fauna isotope composition, indicating local origin of individuals or long residence time in the region. Comparing these data with tooth enamel values, two groups of immigrants from distinctive geological environment were established. The Dulantzi population constituted mainly a local society with influxes of immigrants. The foreign individuals are distributed through the studied period of time, suggesting that migration movements were limited in number. Isotopic signatures indicating mainly local individuals, linked to grave goods with archaeological attribution to Germanic origin, question the previous ethnic paradigm.

1. Introduction

The study of the role of German migrations during the disintegration of the Roman Empire and the transformation of Roman society during the Early Medieval period has seen renewed interest in recent decades, because the implementation and generalization of new analytical techniques has transformed interpretative frameworks. In recent years, revisions carried out by some scholars downplayed the impact of the Germanic migrations, even extending to denying them, whereas some central European researchers had revisited the ethnogenesis paradigm and redefined the historic events in terms of systemic transformation processes (Pöhl, 1998; Gillett, 2006; Castellanos, 2007; Hakenbeck, 2008; Heather et al., 2010; Hakenbeck, 2011; James, 2011). However, in the archeology of southern Europe the disruptive approach that gives a remarkable role to Germanic migrations prevails (Valenti, 2009). This explains the effect of cultural historicism which defined ethnic groups as historical subjects identifiable based on cultural material, thereby allowing the definition of migration flows due to the spatial distribution of the diagnostic objects. Therefore, the grave goods found in cemeteries and other markers, such as architectures and wares, allow identification of immigrants (Brogiolo and Chavarria Arnau, 2008).

In recent years, several research projects have been started up across Europe, seeking to verify the real impact of migration and the historical role of Germanic elites by means of strontium isotope analysis of archaeological human remains, in particular those individuals buried with Germanic grave goods. However, these studies do not assist in the cases of individuals buried without Germanic grave goods, a situation corresponding to most burials even in so-called "Germanic cemeteries" (Quirós Castillo and Vigil-Escalera, 2011). Therefore the study of rural communities with "Germanic cemeteries" in a broad time frame will allow establishment of mobility patterns and detect the presence or absence of mass migrations in the Middle Ages.

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ORIGINAL PAPER

Isotopic evidence for the reconstruction of diet and mobility during village formation in the Early Middle Ages: Las Gobas (Burgos, northern Spain)

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Abstract Strontium, carbon, and nitrogen isotopes of human bone and tooth remains have been used to reconstruct residential mobility and diet of early medieval populations at Las Gobas from the sixth to eleventh centuries. Most non-local individuals correspond to the tenth to eleventh centuries and were mostly women and infants. This residential mobility coincided with the formation of Laño village and the abandonment of artificial cave settlement. Carbon and nitrogen isotope ratios of bone collagen indicate an omnivorous homogenous diet based on terrestrial plant resources, with few animal-derived proteins from livestock. Millet consumption was restricted to an earlier period of time (seventh to ninth centuries); and in later periods (tenth to eleventh centuries), mainly C_3 plants such as wheat and barley were consumed. In general, there were no dietary differences between individuals according to sex or age. Sex-related dietary differences have only been observed in the tenth to eleventh centuries, when females consumed a more vegetarian diet and less animal protein. The higher δ^{15}N values in infants reflect the weaning effect, while the differences in δ^{15}N values between young adult men and young adult women can be explained as a physiological factor related to pregnancy or different origins. In a comparison with contemporaneous medieval populations in the northern Iberian Peninsula, both δ^{13}C and δ^{15}N values suggest similar foodstuff resources and diet among Christian and Muslim populations.

Keywords Palaeodietary patterns · Human migration · Rock-hewn dwelling · Middle Age · Northern Iberian Peninsula

Introduction

A profound transformation affecting territorial organization occurred after the collapse of the Roman Empire. In the Cantabrian region of north Spain, the post-Roman landscape showed a high degree of territorial fragmentation with a lack of villages or rural structures. In this historical context, small and dispersed farmsteads and a few rock-hewn dwellings dated in the sixth and seventh centuries have been identified. In the course of the eighth century, a profound transformation of the Cantabrian region landscape started with the creation and gradual expansion of a network of villages (Quirós Castillo, 2009; Quirós Castillo 2011). In the ninth century, the former peasant settlement densification occurred with the creation of true village networks. Unlike in other regions, churches in the Cantabrian region never played a significant role in the formation of village networks. Early medieval churches were constructed once the villages were created and the construction of a church implied new ways of social organization and the exploitation of the territory.

The Las Gobas site consists of cave settlement and adjoining farmsteads. The use of artificial caves has been subject to the most varied interpretations. Traditional historiography has explained such occupations as a phenomenon related to different variables of Christian asceticism (e.g., González Blanco 1993; Monreal 1997; Castellanos 1998; Espinosa 2006). Alternatively, they have been interpreted as farming

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RESEARCH ARTICLE

Isotope analyses to explore diet and mobility in a medieval Muslim population at Tauste (NE Spain)

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Abstract

The Islamic necropolis discovered in Tauste (Zaragoza, Spain) is the only evidence that a large Muslim community lived in the area between the 8th and 10th centuries. A multi-isotope approach has been used to investigate the mobility and diet of this medieval Muslim population living in a shifting frontier region. Thirty-one individuals were analyzed to determine $\delta^{15}N$, $\delta^{13}C$, $\delta^{18}O$ and $^{87}Sr/^{86}Sr$ composition. A combination of strontium and oxygen isotope analysis indicated that most individuals were of local origin although three females and two males were non-local. The non-local males would be from a warmer zone whereas two of the females would be from a more mountainous geographical region and the third from a geologically-different area. The extremely high $\delta^{15}N$ baseline at Tauste was due to bedrock composition (gypsum and salt). High individual $\delta^{15}N$ values were related to the manuring effect and consumption of fish. Adult males were the most privileged members of society in the medieval Muslim world and, as isotope data reflected, consumed more animal proteins than females and young males.

Introduction

Muslims invaded most of the Iberian Peninsula in the Early Middle Ages (AD 711) and remained for the next seven centuries, until 1492 when the Christian Kingdoms totally reconquered the peninsula. The northern frontier of the country captured by the Muslims, known as al-Andalus, extended eastward on the southern slopes of the Cantabrian range from the present Galicia to Catalonia. Following the Muslim conquest, al-Andalus was at first (711–750) a province of the Umayyad Caliphate centered on Damascus. From 740 a series of civil wars between various Muslim groups resulted in the breakdown of the Arab empire and the Emirate of Cordova (c. 750–929) emerged. In 929 the emir of Cordova proclaimed himself Caliph and the period of the Caliphate of Cordova was established (929–1031). The Cordova

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Analyses of human dentine and tooth enamel by laser ablation-inductively coupled plasma-mass spectrometry (LA-ICP-MS) to study the diet of medieval Muslim individuals from Tauste (Spain)

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ABSTRACT

Trace elements have been analysed in 23 tooth enamel and dentine samples from a Muslim population in Tauste (North Spain) to investigate health and palaeodietary patterns during the 8th–10th centuries. LA-ICP-MS technique was used to determine the chemical composition of teeth. Post-burial alteration was established by REE and U high content (1 > μg/g) and several samples, mainly of deciduous teeth, were discarded. Trace elements show different behaviour in dentine and enamel related to the composition of tissues. Five individuals had high Pb contents (ranging between 2 and 30 μg/g) suggesting intoxication by occupational exposure to anthropogenic lead. Considering the period of time, individual lead intoxication could be attributed to working activity. Young individuals vs adults, and males vs females show different food intake, probably due to sexual division of labour or social status. The palaeodietary pattern of the Tauste population provides insights into this Muslim community.

1. Introduction

Research on residential mobility or diet has been performed in bones and teeth through the development of macroscopic and chemical methods of analysis. In recent decades the use of laser ablation-inductively coupled plasma-mass spectrometry (LA-ICP-MS) has enabled direct analysis of solid samples using only a small amount of material and minimal sample destruction [1,2]. This minimal destructive nature of the laser ablation technique is essential in archaeological materials and even more so when remains are limited and must be preserved.

LA-ICP-MS provides the distribution of trace elements throughout the skeleton samples. Several factors could affect the elemental composition in bones and teeth, especially in buried samples. In addition to diet, another factor affecting the composition is the remodelling process. Bones are mainly composed of hydroxyapatite and at structural level are remodelled continuously during the individual's life. Teeth are divided into inner pulp cavity, dentine and outer enamel tissues. Dentine is a much softer, has a less mineralized structure and regenerates constantly; whereas tooth enamel is the hardest material in the human body and is not renewed after forming during childhood, thus fixing the elemental composition in an earlier stage of the individual's life [3,4,5,6,7]. In archaeology, enamel is preferred to determine the chemical composition because it is less likely to be affected by diagenesis and environmental conditions, e.g. [8,9].

In archaeological samples, burial conditions can cause changes in the mineral composition leading to exchange of ions with the environment [10]. The diagenesis effect is crucial in an interpretation of the elemental composition of bones and teeth. The analysis of soils associated with human remains is the best approach to evaluate diagenesis. When soil composition is unknown, a different approach is used to evaluate the diagenesis effect, as the content of rare earth elements and U and Th in teeth and bones are sensitive indicators of diagenesis [11,12,13].

When diagenesis is not a problem, the chemical analysis of enamel and dentine can be directly related to the diet. Thus some elements such as sodium, magnesium, zinc, strontium, barium and lead are directly related to food consumption [14,15,16,17]. In a multi-elemental approach, barium, strontium, manganese and magnesium are prevalent in vegetables while zinc and copper indicate a meat-based diet. Additionally tooth chemical composition allows identification of long-term heavy metal exposure and may provide information about the exposure of an individual to toxic metals (e.g. Cd, Pb) [18,19].

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Anexo 2: Summited papers

I Guede1, MC Zuluaga, LA Ortega, A Alonso-Olazabal, X Murelaga, I Garcia-Camino, P Iacumin. Social structuration on medieval rural society based on stable isotope analysis of dietary habits and mobility patterns: San Juan de Momoitio (Biscay, North Iberian Peninsula). American Journal of Physical Anthropology (Under review).

SOCIAL STRUCTURATION IN MEDIEVAL RURAL SOCIETY BASED ON STABLE ISOTOPE ANALYSIS OF DIETARY HABITS AND MOBILITY PATTERNS: SAN JUAN DE MOMOITIO (BISCAY, NORTH IBERIAN PENINSULA)

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Diet and mobility patterns in a medieval peasant community

Keyword stable isotopes, diet, mobility, middle age, rural life, northern Iberian Peninsula.

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