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RESEARCH ARTICLE

Stable isotopic evidence for land use patterns in the Middle Euphrates Valley, Syria

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Abstract

Objectives: Stable carbon and nitrogen isotope ratios ($d^{13}C$ and $d^{15}N$) were used to reconstruct the history of subsistence strategies in the middle Euphrates valley, NE Syria, in six temporal subsets dating from the Early Bronze Age (c. 2300 BCE) to the Modern period (19th/20th century CE). The study aims to demonstrate that changes in political and social organization over time, for which the archaeological record suggests different goals of land use and modes of production, register through dietary patterns that are reflected in isotopic data.

Materials and methods: 173 dentin samples were taken from human individuals buried at three sites (Tell Ashara, Tell Masaikh and Gebel Mashtale) together with 15 animal bone samples. Distribution of the $d^{13}C$ and $d^{15}N$ values in collagen was interpreted in diachronic perspective, and with regard to lifetime shifts between childhood and adolescence.

Results: Diachronically, isotope signatures indicate a clear decrease in $d^{15}N$ values accompanied by a small shift in $d^{13}C$ values between the Old Babylonian (c. 1800–1600 BCE) and the Neo-Assyrian (c. 850–600 BCE) subsets. A major shift in $d^{13}C$ values occurred between the Early Islamic (c. 600–1200 CE) and Modern (c. 1800–1950) periods. Ontogenetic changes only occur in a few individuals, but these suggest change of residence between childhood and adolescence.

Discussion: The depletion in ^{15}N from the Neo-Assyrian period onwards is best explained in terms of a shift from intensive to extensive farming, triggered by the fall of regional city-states after the Old Babylonian period and the formation of large supra-regional polities in the Neo-Assyrian period and later. The enrichment in ^{13}C during the Modern period was most likely the effect of more widely utilizing the dry steppes, abundant in C_4 plants, as pasture.

KEYWORDS
carbon isotopes, diet, Mesopotamia, nitrogen isotopes, subsistence strategies

1 | INTRODUCTION

Over three decades ago, research on stable nitrogen and carbon isotopes in human and animal collagen became a principal tool in reconstruction of diet and subsistence in past human populations. Stable carbon isotope ratios ($d^{13}C$) allow for the estimation of the relative share of C_3 (most cereals and most grasses) and C_4 plants (maize, millet, sorghum, sugar cane, some grasses, and reeds) in human and animal diet. In addition, carbon values permit some distinction between terrestrial and marine diet, and potentially trophic level (De Niro & Epstein, 1978). Stable nitrogen isotope ratios ($d^{15}N$) are more strongly correlated to trophic level, mainly reflecting protein intake, but also help to characterize the environment, for example aridity due to water stress or human intervention and animal management due to manuring practices (DeNiro & Epstein, 1981).

Here we investigate temporal differences in $d^{15}N$ and $d^{13}C$ between several chronological subsets of human and animal collagen extracted from teeth and bones representing populations inhabiting the lower part of the middle Euphrates valley from the end of the Early Bronze Age (c. 2300 BCE) until the 19th or early 20th century CE

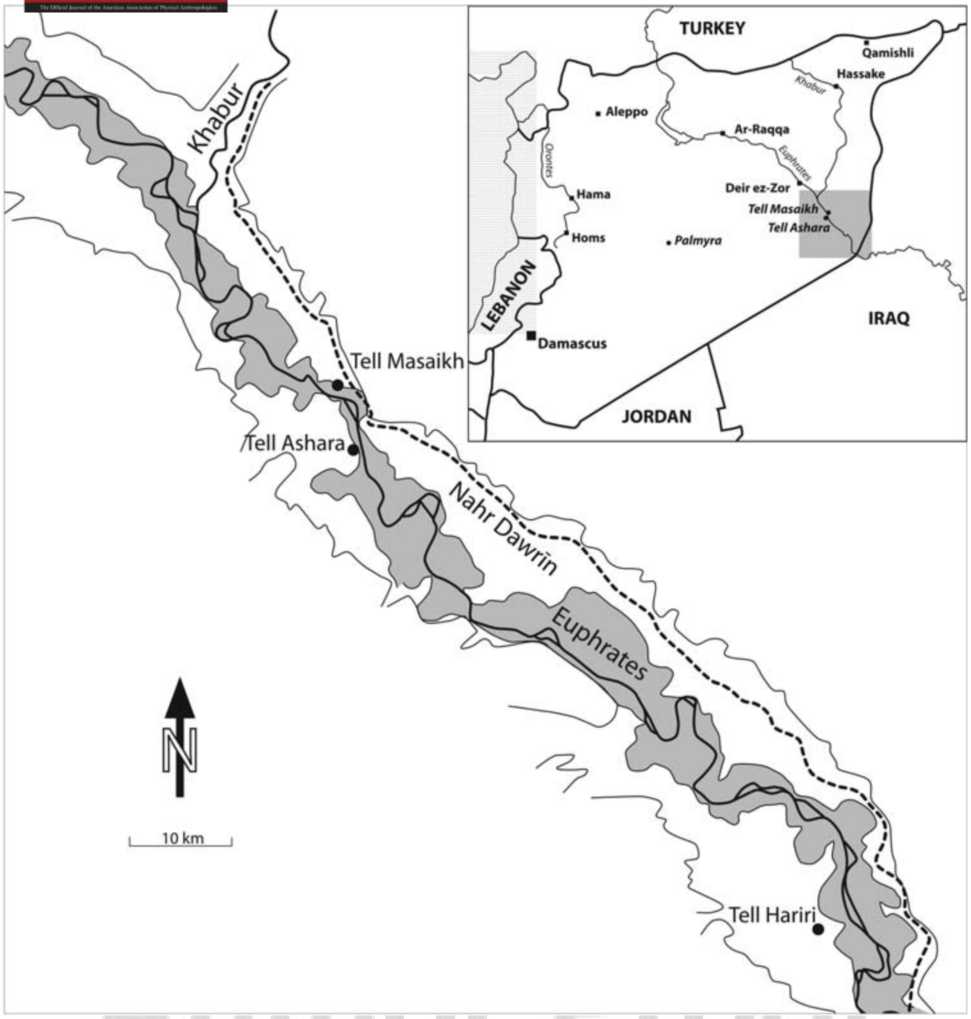

FIGURE 1 Map of the Euphrates valley between the Khabur confluence and Tell Hariri, showing the locations of major sites mentioned in the paper

(see Sołtysiak & Bialon, 2013 for the population history of this region). Isotopic data are used to trace the temporal changes in the interplay between subsistence strategies that may have been adopted separately or jointly in the region. Results of previous studies suggest a dichotomy between local and regional modes of production, namely farming based on higher water tables near the river, artificial irrigation, import of grains from dry farming zones, and herding inside the valley or outside in the dry steppe (see below). The political structures, independent smaller kingdoms of the Bronze Age, or supra-regional empires during the Iron Age, effected the implementation of such different land use strategies over time, for example, efforts to intensify agricultural production during the Bronze Age, as opposed to extending agriculture to larger areas of available and controlled land during the Iron Age.

We hypothesize that the ensuing significant regional socio-economic developments are reflected in diachronic patterns of dietary and subsistence change. This will provide both novel and corroborating proxy evidence of societal change, for which the archaeological and textual sources alone cannot offer comprehensive explanation. Adjustments to agricultural production in response to availability of land and resources facilitated the construction of artificial irrigation structures to increase water availability, or the exploitation of the dry steppe more abundant in C_4 grasses than the vegetation cover in the valley. Subsistence data directly ascertained from the populations that inhabited the region can thus test independently whether the political goals of changing modes of food production in support of regional and supra-regional polity formation find their expression in dietary information, as the concomitant small and larger scale societal changes can be expected to register in isotopic data. We also aim to trace these diachronic observations into modern times by incorporating data from contemporary Bedouin cemetery for comparison. Findings are discussed in the bioarchaeological context of the respective time periods.

1.1 | Ecological settings

The lower middle Euphrates valley (Figure 1) is not a very favourable place for agriculture. The average annual precipitation is too low (c. 140 mm) for dry farming and the valley is too deep and narrow for extensive irrigation, as opposed to the alluvial plain of southern Mesopotamia. For that reason, the textual evidence from the 3rd millennium BCE onwards shows that the local population consisted of relatively

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low number of settled farmers using narrow strips of arable land in the floodplains along the river bed and semi-nomadic pastoralists who operated with their ovicaprid herds in the dry steppes, sometimes far away from the Euphrates (Lyonnet, 2001, 2009). Such a dimorphic society with strong ties between two groups exercising different subsistence strategies is well documented especially in the early 2nd millennium BCE by the abundant cuneiform archives from Mari (Fleming, 2009; Pitard, 1996; Rowton, 1974). Trade was also important in the local economy, at least in those periods when the exchange of goods between southern Mesopotamia and Anatolia or the Levant was common and well organized (Klengel, 1983; Leemans, 1977; Zawadzki, 2008). However, the balance between plant- and animal-based food may be assumed to have been relatively stable in the middle Euphrates valley. Plant cultivation was less dependent on precipitation compared to areas further north (Wilkinson, 1997), nor endangered by soil salinization as in the southern alluvium (Artzy & Hillel, 1988). The efficiency of pastoralism was secured by abundant dry steppes.

Archaeobotanical research shows that from the 3rd millennium BCE to the 1st millennium CE the most important crops, as in other semi-arid areas of the Near East, were barley and wheat, with only a minor share of lentils or other legumes (Riehl, 2009; Samuel, 2001). The proportions of domesticated animals represented by bone remains are quite stable with 60–80% ovicaprids, and 20–40% cattle from the late 3rd millennium BCE onwards, except during the earliest phases of occupation at Tell Ashara (Early Dynastic and Akkadian periods, c. 2700–2300 BCE), which displays a greater number of ovicaprids. Most importantly, the remains of omnivorous animals such as pigs were absent or very infrequent in bone assemblages from all chronological contexts (Gręzak, 2015). Similarly, the economy of modern Bedouin tribes before their continuous sedentarization during the 20th century CE almost exclusively relied on nomadic ovicaprid herding (Bahhady, 1981).

1.2 | Artificial irrigation and temporal changes in agricultural strategies

The scale of artificial irrigation in the middle Euphrates valley is a highly discussed topic, and it usually focuses on the canal known as Nahr Dawrīn, located on the left (eastern) upper river terraces of the lower Khabur and middle Euphrates. Its total length is about 120 km and its width varies from eight to eleven meters (Figure 1), with large portions of this huge earthwork still clearly visible in the landscape (Figure 2). Both the dating and the function of Nahr Dawrīn are debated and the only commonly accepted fact is that the canal was used in the Early Islamic period, at least in the 7th/8th century CE, and was deserted before the 11th century as a consequence of political instability in the region.

Due to repeated conservation and repair work in antiquity, the construction of the canal cannot be dated through any archaeological method, and the textual evidence is sparse and unclear. For that reason, suggested dates vary by more than 3000 years (cf. Lafont, 2009). According to Margueron (2004:76–79), the canal was dug in the Early Bronze Age to secure a safer navigation on the major trade route along the Euphrates and Khabur (see also Geyer & Monchambert, 2003:212–213). Most other authors, however, point out that the location of the canal suggests gravity irrigation, but indications as to when it may have been constructed range widely, from Old Babylonian (Durand, 1997:580), through the Middle Assyrian or Neo-Assyrian period (Durand, 2002; Kühne, 1995; Kühne & Becker, 1991; Masetti-Rouault, 2008, 2010) to Early Islamic times (Berthier, 2001), with only limited evidence to support these proposals.

COLOR

FIGURE 2 Well preserved section of the Nahr Dawrīn canal near Tell Masaikh

Arguably, the existence of a large canal structure would provide a tangible link to the known efforts of the Neo-Assyrian and later empires to expand agricultural production on a large scale. Irrigation of a large part of the river valley could thus increase settlement density in the areas that were turned into agricultural land, but reported settlement patterns in the lower middle Euphrates valley are inconsistent and only suggest relative stability from the Early (EBA, c. 3000–2100 BCE) to Middle Bronze Age (MBA, c. 2100–1500 BCE), with a decline by the Late Bronze Age (LBA, c. 1500–1200 BCE) and some recovery in the Neo-Assyrian period (c. 850–600 BCE). Finally, after a decrease in settlement density in the Achaemenian period (c. 550–330 BCE), the number of sites gradually increased to an absolute peak in the Early Islamic period (Geyer & Monchambert, 1987; Simpson, 1984).

However, there is some evidence for a relationship between settlement pattern and the course of Nahr Dawrīn, mainly for the Neo-Assyrian (Masetti-Rouault, 2010) and Early Islamic periods (Berthier, 2001). In the former period, Kar-Assurnasirpal (modern Tell Masaikh), the largest known site on the left bank of the Euphrates, was established as the capital city of the Assyrian province of Rasappa. Since the Assyrians were interested in increasing agricultural production, the construction, reconstruction or extension of Nahr Dawrīn may have been an element of their policy (Masetti-Rouault, 2008, 2010).

Possible archaeobotanical evidence of irrigation would entail a shift from more to less drought resistant crops and the presence of weed species that grow in humid conditions. However, the dominant crop in all periods under investigation was barley, supplemented by small quantities of wheat, accompanied by some broomcorn millet from the Neo-Assyrian period onwards. From this time period, grass pea

(*Lathyrus sativus*) is present at Tell Masaikh, and its combination with millet and barley supports prevailing dry conditions associated with agriculture (Kubiak-Martens, 2013).

On the other hand, small-scale irrigation was always possible in the floodplain of the Euphrates including its oxbow marshes and, although agriculture based on such irrigation was risky due to the unpredictable seasonal amplitude of the river water table, intensive cultivation of areas along the river may have been exercised since the mid-4th millennium BCE (Masetti-Rouault, 2008). For example, there is clear evidence of small irrigation network in the neighborhood of Tell Hariri/Mari, dated to the mid-3rd millennium BCE (Geyer & Monchambert, 2003).

Archaeobotanical data from Tell Ashara and Tell Masaikh include some weed species that indicate good moisture conditions at least since the 3rd millennium (Kubiak-Martens, 2015). During the Neo-Assyrian (9th to 7th century BCE) and later periods these weeds, for example, amaranth (*Amaranthus* sp.) and Bermuda grass (*Cynodon dactylon*) known to grow especially on the banks of canals, were quite common. In the Neo-Assyrian samples from Tell Masaikh there are also several rare crops that need good watering, such as coriander (*Coriandrum sativum*), celery (*Apium graveolens*), cumin (*Cuminum cyminum*), possibly also dill (*Anethum graveolens*) and cultivated grape (*Vitis vinifera*) (Kubiak-Martens, 2013). However, they were recovered from the governor's palace and, therefore, may not be representative for the general agriculture of that period. Late Roman samples as well included some more water-demanding species, such as free-threshing wheat, grapes, figs (*Ficus carica*), and melon (*Cucumis melo*) (Kubiak-Martens, 2015).

Therefore, the archaeobotanical evidence is ambiguous. But also the assemblages of animal bones reveal only minor differences in the proportions of domesticated animals. In all periods sheep and goats were the predominant species (from 85% during the 3rd millennium BCE to 60% in the Islamic period), but in the Neo-Assyrian period and especially in the Islamic period (but not in the Late Roman period) the share of cattle bones clearly increased. Pigs were present only in the Neo-Assyrian and Late Roman periods when they represented c. 5% of the entire assemblage (Grężak, 2015). Cattle and pigs need much more water than ovicaprids, but a direct comparison of species representation is difficult as only the number of identified specimens (NISP) is available and moreover the dominance of ovicaprids—in concert with a predilection for barley—may be a cultural rather than ecological choice. Especially the absence of pigs in the Islamic period was likely related to their cultural perception as impure animals (cf. Kassam & Robinson, 2013).

The textual evidence is also ambiguous. Several sources from Mari mention irrigation works that were undertaken under the rule of Yahdun-Lim (c. 1810–1794 BCE) and his son Zimri-Lim (c. 1775–1761 BCE). Apart from accounts of two canals called Hubur and Isim-Yahdun-Lim, located most likely on the right bank of the Euphrates north of the Khabur confluence, there is also information provided in a letter by the governor Yaqqim-Addu that another canal transported the waters of the Khabur and was an important part of the irrigation system (Durand, 1997:580; Heimpel, 2003; Viollet, 2004:56). However, neither the length nor a more precise location of this canal are clearly described here or in other contemporary documents. No known texts refer explicitly to any irrigation canals during the Neo-Assyrian period (Masetti-Rouault, 2010).

A few hundred years later, Xenophon mentiones the river Maskas (*Anabasis* 1.5.4), perhaps subsequently referred to by Ptolemy as Saocoras (*Geographia* 5.18.3), which has been located as a left-hand tributary of the Euphrates 35 parasangs (about 100–140 km) below the confluence of the Khabur (Lempriere, 1836:199; Ainsworth, 1888:432–433). According to Xenophon, the river Maskas surrounded a large deserted city named Korsote, which may be identified as one of two modern archaeological sites, either Baghouz or ed-Diniyye (Barnett, 1963:3–5). On the other hand, there is also a possibility that Maskas and Saocoras were nothing but ancient names of the Khabur itself (cf. Gawlikowski, 1992).

Islamic sources explicitly mention one canal in the area between Deir ez-Zor and Al Bukamal, referred to as Nahr Sa'id after its creator, prince Sa'id ben 'Abd al-Malik in the early 8th century CE. Its location is not completely fixed, but it most likely used waterwheels to irrigate a relatively large right bank floodplain of the Euphrates north of the Khabur confluence, in the approximate location as the earlier canals Hubur and Isim-Yahdun-Lim (Genequand, 2009; Rousset, 2001). However, small archaeological test trenches in Nahr Dawrīn revealed Early Islamic pottery and the distribution of 26 small sites along the canal dated to the Umayyad period (661–750 CE) suggests that Nahr Dawrīn was used in the 7th/8th century CE (Berthier, 2001; Hont, 2005:208), and likely was abandoned not much later. Thus, the last date for possible irrigation using the Nahr Dawrīn may be safely estimated between the 8th and 10th century CE.

1.3 | Dry steppe pastures

Abundant archives from the royal palace of Mari, dated to 18th century BCE, show a specific social organization with urban dwellers of the major cities (Mari and Terqa), farmers engaged in plant cultivation along the river and small-scale stationary animal husbandry, as well as semi-nomadic pastoralists that operated in the dry steppes beyond the upper terrace. These mobile herders were tied to the settled population by mutual exchange of various products, but also by kinship (Liverani, 1997; Rowton, 1974, 1977).

While state authorities sought to keep mobile herders under control (cf. Bonneterre, 1995), they nevertheless occasionally invaded lands under cultivation and disrupted agricultural production. However, for most of the time, co-existence of herders and settled population was peaceful and the network of relation between two socioeconomic entities operating in different ecological zones has been labeled as dimorphic society (Rowton, 1977).

It is unclear whether such a close relationship between mobile pastoralists and farmers was typical only for the kingdom of Mari during the Early and Middle Bronze Age, or whether it was present also in later periods. The social and economic crisis of the 12th century BCE forced herders to become more mobile and previous kingdoms of the Near East based on agriculture collapsed (Neumann & Parpola, 1987). It is likely that during that time some farmers joined Aramean tribes of herders, but in general during the beginning of the Iron Age the mobile herders were perceived as a major threat by urban dwellers. In times of the Neo-Assyrian Empire (9th–7th century BCE) the agricultural potential of northern Mesopotamia was restored and the state was powerful enough to strictly control herders (Sołtysiak, 2016). However, in later periods some autonomy of Aramean and Arab nomadic tribes was attested and the mobility of herdsmen was increased due to domestication of camels (Rosen & Saidel, 2010).

The most rapid and drastic change in the history of North Mesopotamia was caused by the invasion of Mongols in the second half of the 13th century CE. They destroyed many cities, killed large parts of the local population, and devastated lands under agriculture. After their raids and other conflicts in the area, the part of the Euphrates valley not suitable for dry farming was largely abandoned and eventually taken by Bedouin tribes that moved in from the Arabian Peninsula during the 17th century. In contrast to previous periods, Bedouins operated mainly in the dry steppe, and had much lower interaction with the scarce remaining permanent sites along the valley (Raswan, 1930).

1.4 | Stable nitrogen and carbon isotopes: The model

The general principles underlying the representation of dietary signals by stable isotope values of carbon and nitrogen in past populations are established and the processes of fractionation and trophic level spacing well understood (e.g., Lee-Thorp, 2008 for overviews; Katzenberg, 2008). In the context of the present study, several more specific aspects are particularly pertinent and will be addressed for their relevance as outlined above.

Archaeological populations of the Old World are frequently associated with a subsistence strategy that provided them with a mainly C_3-based terrestrial diet consisting of varying proportions of plant and animal-derived foodstuffs. Ecological circumstances and cultural choices govern the dietary mix that is eventually reflected in the isotope values of human bone and teeth. In areas characterized by low precipitation, such as the middle Euphrates valley, crops that are less demanding are expected to form the main-stay of plant matter supply, but irrigation broadens the range of cultivated food items to dietary staples that may be grown or consumed. In addition, there were certain time periods when more arid-adapted C_4 crops, such as broomcorn millet may have been grown. C_4 crops follow a biochemical pathway that reflects adaptation to an arid climate, where there is pronounced discrimination against ^{13}C, whose incorporation into the diet would result in more positive d^{13}C values of the human end user (Nesbitt & Summers, 1988). However, even if present, millets were never an important part of human or animal diet in the middle Euphrates area (cf. Kubiak-Martens, 2015). Diversification of agricultural strategies, for example the expansion of pastoral activities into the dry steppe, exposes livestock to a variety of C_3 and C_4 shrubs and grasses. When humans subsequently consume these animals or their products, the C_4 component would result in more positive d^{13}C values in humans as well and thereby indicate an adaptation in subsistence activities.

Nitrogen stable isotope values largely reflect consumption of animal-derived protein in temperate climates (Hedges & Reynard, 2007). In arid regions, elevated d^{15}N values were long thought to be a result of water stress, either through consumption of water-stressed animals or as a direct effect on humans, with the underlying mechanism relating to the increased excretion of urea, which is ^{14}N-depleted relative to the diet and thus ^{15}N is enriched in the body (Ambrose & DeNiro, 1987). Isotope data from controlled feeding experiments (Ambrose, 1983) and, especially, herbivores from arid areas (Hartmann, 2011) now suggest that d^{15}N values are rather determined by the isotopic composition of their diet. AQ1

In agricultural systems where intensive cultivation is maintained by high levels of manuring, d^{15}N values are likely to be elevated (cf. Fraser et al., 2011; Styring et al., 2016) and this effect may be high enough to differentiate between intensive (small area, high manuring) and extensive (large area, low manuring) agriculture in North Mesopotamia (Styring et al., 2017). In the specific case of the middle Euphrates valley, transition from more intensive to more extensive agriculture may have been enabled mainly by introduction of the large-scale irrigation system that dramatically increased the area suitable for plant cultivation and at the same time decreased pastures within the valley.

2 | MATERIALS

Human and animal remains used for the present study were excavated at three archaeological sites located c. 70 km south of Deir ez-Zor (Figure 1). Tell Ashara (ancient Terqa) was a major city during the MBA and LBA, and the capital city of the Khana kingdom that followed the destruction of Mari by Babylonians in the mid-18th century BCE. Several burials dating to this period were found in the domestic areas. During 19th and early 20th century, local Bedouin tribes used the top of the ancient site as a regular cemetery.

Tell Masaikh (ancient Kar-Assurnasirpal) was a small settlement during the Early Chalcolithic and in the MBA, and a large regular town during the Neo-Assyrian period. A dozen burials were recovered here in domestic contexts. During Classical Antiquity and later it was also used as a cemetery. Gebel Mashtale is a much smaller settlement with an Islamic cemetery on top. All three sites were excavated by the French-Syrian mission directed by Olivier Rouault and Maria Grazia Masetti-Rouault. Human remains excavated from 1996 to 2006 were studied by Sołtysiak (cf. Sołtysiak & Bialon, 2013) and are curated in the Department of Bioarchaeology, University of Warsaw, Poland. Animal remains were studied by Anna Grȩzak.

Human collagen was extracted from root dentin of second and third permanent molars. For four random skeletons from Tell Masaikh collagen yield was also checked in bone. The source for animal collagen was bone. Human samples were taken from all individuals with at least one root preserved, amounting to a total of 173 specimens covering most of the regional history from the Early Bronze Age until the beginning of the 20th century CE. The chronological distribution of human samples is shown in Table 1. To interpret human collagen data against T1 a broader ecological background, faunal material from later occupations at both sites was also included that comprised three dogs, two bovids,

TABLE 1 Chronological distribution of human samples

Acronym	Period	Dates	M2 samples	M3 samples
SHA	Shakkanakku	c. 2300–1900 BCE	11	7
OB	Old Babylonian	c. 1900–1700 BCE	18	9
NA	Neo-Assyrian	c. 900–600 BCE	4	1
CLA	Classical Antiquity	c. 600 BCE–600 CE	31	25
ISL	Early Islamic	c. 600–1200 CE	26	16
MOD	Modern Islamic	c. 1800–1950 CE	15	9
Total			105	67

one sheep, one goat, three camels, one equid, two gazelles, one fox, and one rodent. They represent later periods of occupation at both sites.

3 | METHODS

Dentine and bone samples were taken in duplicate. After cleaning the surfaces with aluminium oxide powder air abrasion to remove adhering soil particles, the samples were subjected to a modified Longin method (Brown, Nelson, Vogel, & Southon, 1988) for collagen extraction, which consists of the following steps: demineralization in 0.5M HCl at 2–58C, followed by gelatinization at 728C for 48 h in deionized water adjusted to pH 3, with 0.5M HCl. Insoluble materials of the extraction mix were removed using Ezee filter separators (Elkay Laboratory Products, Basingstoke) and further purified using Amicon Ultra-4 centrifugal filters (Millipore) to remove contaminants lower than 30,000 nominal molecular weight limit (Brown et al., 1988). The filtered products were lyophilized, a subsample of 0.4 6 0.1 mg combusted and analyzed by Isotope Ratio Mass Spectrometry (Finnigan Delta Plus XL) in the School of Archaeological Sciences, University of Bradford, UK. The analytical precision of the instrument was estimated as 60.2& for nitrogen and 60.05& for carbon isotopes.

At regular intervals, methionine standard reference material, with known $d^{13}C$ (–26.6&) and $d^{15}N$ (–3.0&) values (Elemental Microanalysis, Devon, UK) was measured in tandem with samples of bone collagen to determine the accuracy and precision of analytical methods. In addition, internal and external certified laboratory standards (e.g., IAEA standards, bovine liver, fish gel) were used for quality assurance. To control for possible effects of diagenetic processes (Ambrose, 1993), collagen yield, the %-carbon and %-nitrogen, and the C/N ratio were recorded. Collagen yields of 1% have been considered sufficient to indicate preservation of authentic collagen (van Klinken, 1999), but occasionally in this study samples with lower collagen yield (>0.2%) were accepted if a C/N ratio was between 2.9 and 3.6, which is the known atomic C:N range for bone collagen (Ambrose, 1993).

Standard parametric and nonparametric statistical tests (Kruskal–Wallis ANOVA with post hoc test, Mann–Whitney U test, Pearson's product–moment correlation coefficient r, Pearson's v^2 test) have been performed using STATISTICA version 12. Distribution bimodality has been tested using likelihood ratio test for bimodality (Holzmann & Vollmer, 2008) with a package 'bimodality test' for R (Schwaiger, Holzmann, & Vollmer, 2013).

4 | RESULTS

No collagen could be extracted from four samples of human bone and the collagen yield in dentine was variable between sites and periods

TABLE 2 Collagen yield depending on site and chronology

		Collagen >0.2%		Median coll. yield	
Site	Chronology	n/N	(%)	M2	M3
Tell Ashara	Shakkanakku	15/18	83%	10.1%	8.9%
Tell Ashara	Old Babylonian	15/19	79%	11.2%	10.6%
Tell Masaikh	Old Babylonian	7/8	88%	5.6%	5.5%
Tell Ashara 1 Tell Masaikh	Neo-Assyrian	3/5	60%		
Tell Masaikh 1 Gebel Mashtale	Classical Antiquity	35/56	63%	14.6%	13.3%
Tell Masaikh	Early Islamic	19/50	38%	15.1%	14.2%
Tell Ashara 1 Gebel Mashtale	Modern Islamic	23/24	96%	15.3%	14.8%
Total (humans)		117/180	68%	13.3%	13.5%
Tell Ashara 1 Tell Masaikh	All periods (animals)	11/15	73%		

Median collagen yield calculated only for teeth where any collagen was preserved.

TABLE 3 Basic statistics for temporal subsets

Chronology	N	$d^{13}C$ Mean	SD	Median	$d^{15}N$ Mean	SD	Median
SHA	12	219.60	0.44	219.66	14.11	2.53	14.40
OB (Tell Ashara)	12	219.75	0.46	219.71	14.48	2.48	14.38
OB (Tell Masaikh)	5	220.20	0.15	220.24	14.43	1.43	14.36
NA	3	219.21	0.54	218.93	11.89	1.78	11.03
CLA	23	218.69	0.66	218.39	11.68	1.28	11.95
ISL	15	219.19	0.76	219.27	12.11	1.03	12.47
MOD	15	216.44	1.81	217.12	11.96	1.29	12.37
Total	85	218.77	1.48	219.04	12.71	2.02	12.68

(Table 2). If the whole number of samples is divided into three broad chronological categories, enough collagen was detected in 82% of Bronze Age samples from deep strata, in 54% samples from shallow strata representing Neo-Assyrian to Early Islamic periods and in 96% of Modern samples. The total average collagen yield for all periods was slightly higher (by 0.5–1.5%) for M2 than for M3, perhaps because of the greater average robustness of M2 roots.

In total, 117 samples representing 85 individuals passed the quality control (collagen yield >0.2% with C/N ratio between 2.9 and 3.6) (Table 2). For 31 individuals two samples taken from both M2 and M3 were measured and there was a high correlation between $d^{13}C$ (r 5 .93) values and between $d^{15}N$ values as well (r 5 .90), and therefore average values were taken for further analyses to represent chronological subsets. The preservation of collagen in animal bone was sufficient in 11 cases representing all taxa except cattle, although most are represented by one individual only. All human and animal individual measurements are available in the Supporting Information Tables 1 and 2, and the basic statistics for temporal subsets are provided in Table 3.

4.1 | Ecological background

The distribution of isotopic values for humans is quite broad, and in the case of nitrogen ranges from 8& to almost 18&. On the other hand, most humans fall within a narrower range of $d^{13}C$ between 221 and 217&. This supports a diet rich in C_3 plants, with a limited share of C_4 plants. Faunal isotopic values both in $d^{15}N$ and in $d^{13}C$ (Figure 3) are within the expected trophic level positions, but there is some distinction between domesticated and wild species. Domesticated ungulates (goat, sheep, and camel) and dogs overlap with humans, although in some cases less negative $d^{13}C$ can be observed. On the other hand, wild ungulates (gazelles and equid) display less negative $d^{13}C$ and lower $d^{15}N$ values than humans and domestic animals. A rodent with very low $d^{15}N$ and relatively high $d^{13}C$, as well as a fox with a very high $d^{13}C$ value, feature clearly outside the general isotope value distribution.

4.2 | Diachronic patterns

The whole sample was divided into six chronological units covering more than four millennia from c. 2300 BCE to the 20th century CE.

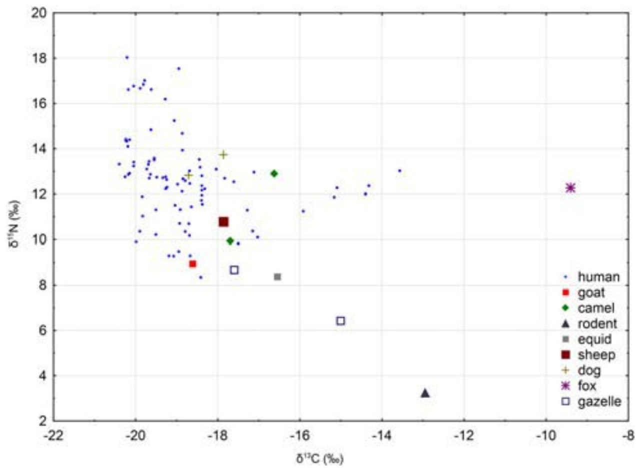

FIGURE 3 Distribution of $d^{13}C$ and $d^{15}N$ values in human and animal samples from the middle Euphrates valley; second and third molars combined

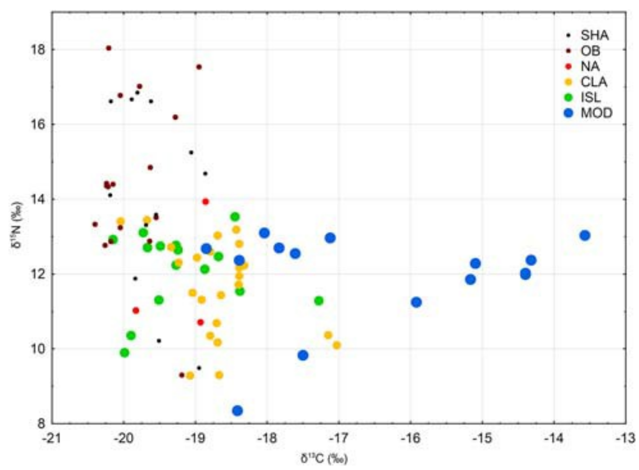

FIGURE 4 Distribution of $d^{13}C$ and $d^{15}N$ values in human samples from the middle Euphrates valley; temporal subsets are marked by different dot size and color; second and third molars combined

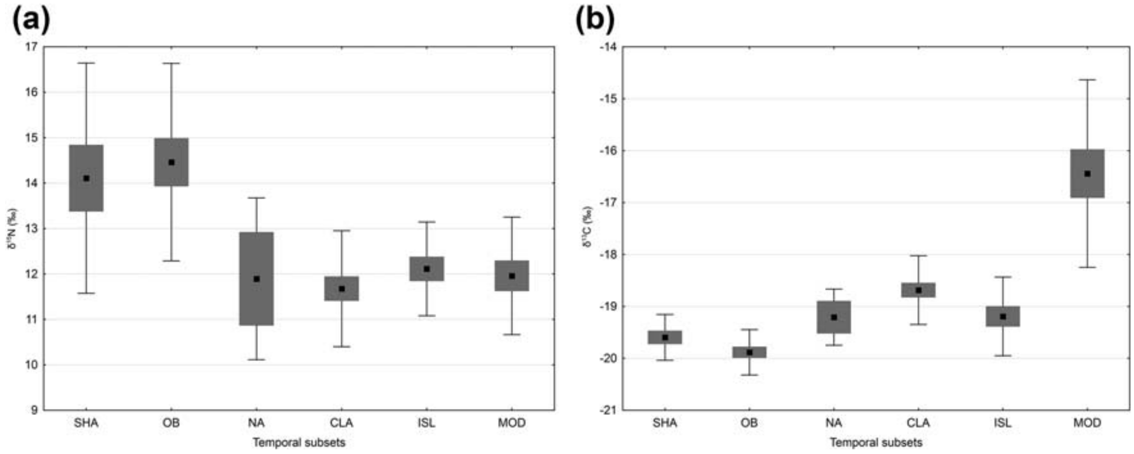

FIGURE 5 Temporal changes in average d13C (a) and d15N values (b). The box-and-whisker plots display mean, SE, and SD; second and third molars combined

The number of individuals per subset is variable, but for most periods (except NA) it is >12, allowing some insight into temporal trends in d13C and d15N values. The data for all temporal subsets are shown in F4 Figure 4, where some differences between subsets may be observed. First of all, SHA and OB distributions overlap in the upper range of the d15N values, while those for all later periods overlap in the lower range of the d15N values. There is also a small shift towards less negative d13C values in the later periods, although the difference is much more evident between MOD and all earlier periods. Some individuals from the Modern cemeteries were clearly enriched in 13C and also the range of d13C values in this temporal subset is much broader than in the earlier periods, ranging from 219& to 213.5&.

Temporal differences are statistically significant for both elements (Kruskal–Wallis ANOVA, H 5 53.86, p < .0001 for carbon and H 5 29.30, p < .0001 for nitrogen) and, if NA is excluded for its small sample size, the most significant transition in d15N is between OB and CLA (z' 5 4.50, p < .0002) and for d13C two transitions are observed, again between OB and CLA (z' 5 4.62, p < 0.0001) and between ISL and MOD (z' 5 4.04, p < .001). These transitions are well illustrated in Figure 5b for nitrogen and in Figure 5a for carbon. F5

The discrimination between early (SHA and OB) and late subsets in d15N values is clear. No late individual has d15N values higher than14&, and only four early individuals have d15N values below 12&. Therefore, it may be safely stated that the d15N values decreased between the Old Babylonian and the Neo-Assyrian period from approximately 12–17& to 9–13&. On the other hand, the temporal transitions in the d13C values, although no less statistically significant, are less clear (Figure 4). There is an evident bimodal distribution in d13C in the Modern subset, within the top cemetery at Tell Ashara. For this subset, the likelihood ratio for bimodality is 4.7 (p < .02, N 5 13) and the unrestricted estimation of the mean d13C values in two hypothetical subpopulations is 217.96 and 214.73&. Other temporal subsets show unimodal distributions of d13C.

Sex assessment was available for 55 individuals from all periods, but no clear differences between males and females are observed. Interestingly, d15N values of males in the Early Bronze Age deviate

COLOR

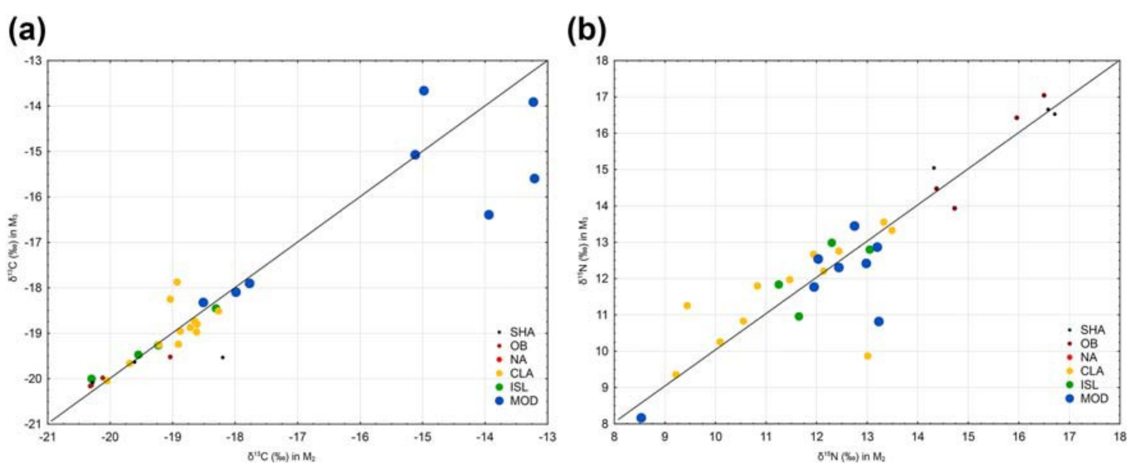

FIGURE 6 Differences between d13C (a) and d15N values (b) in second and third molars; temporal subsets marked by different dot size and color

from a unimodal distribution, and among eight individuals there are three small, yet distinct clusters around 9.5, 12, and 16&, the last one representing four individuals (cf. Figure 4 and Supporting Information Table 1).

4.3 | Lifetime dietary shifts

For 31 individuals d^{13}C and d^{15}N values were measured in both M2 and M3 and therefore possible dietary shifts between late childhood (10–13 years, M2 root formation time) and adolescence (15–18 years, M3 root formation time) may be detected. Considerable differences between molars in d^{13}C values (Figure 6a) occurred in four individuals from the Modern period, in three cases there was a shift towards more negative values (by c. 1–2&), and in one case towards a less negative value (by c. 1.5&). Slighter, but nevertheless evident shifts can be observed in two individuals from Classical Antiquity (c. 1& towards less negative d^{13}C) and in one individual from the Shakkanakku period (c. 1.5& towards more negative d^{13}C).

The d^{15}N values are less correlated between molars, and there are less clear outliers than in the case of carbon isotopes (Figure 6b). Only two individuals dated to Classical Antiquity show a shift in two opposite directions (one c. 13 to c. 10&, the other c. 9.5 to c. 11&), and one Modern individual displays a shift from c. 13 to c. 11&.

5 | DISCUSSION

Although isotopic analyses have contributed widely to the bioarchaeology of various geographical areas and time periods (cf. Lee-Thorp, 2008), they rarely have been adopted in research on human and animal remains from ancient Mesopotamia. This is in part the result of political instability in the region that limited availability of suitable samples at a time when the research on diet became more refined (Sołtysiak, 2006). Climatic conditions of the Near East are also frequently thought to be less favourable for collagen preservation (Bocherens, Mashkour, & Billiou, 2000; Plug, van der Plicht, & Akkermans, 2014; Weiner & Bar-Yosef, 1990). Therefore, the list of sites with any published studies of carbon and nitrogen isotopes so far includes just a few names, for example, Tell Umm el-Marra on the western border of Mesopotamia (Batey, 2011), Tell Barri (Sołtysiak & Schutkowski, 2015) and Tell Sheh Hamad in the Khabur drainage (Höring & Jungklaus, 2010) and finally Tell Bakr Awa in the Shahrizor plain near the Zagros Mountains (Fetner, 2016). Among them, only studies on Tell Barri and on Bakr Awa produced results suitable for diachronic interpretations. In the middle Euphrates valley, while, indeed, the collagen yield in a few analyzed bone samples was very low, most dentin samples retained considerable amount of organic matter and therefore the potential of gathered data is much higher than at other sites in the region.

At a most general level, the diet of people inhabiting the middle Euphrates valley was based mainly on C_3 plants, although some C_4 resources were also available. Archaeobotanical data are quite limited, but it seems likely that for all periods the most important cultivar was barley, accompanied by much smaller quantities of wheat, millet, lentil and pea. Barley is known to be drought and salt resistant, although its cultivation and consumption was perhaps also a cultural choice (Kubiak-Martens, 2013). Quite important is the limited presence of broomcorn millet, a C_4 cereal, at least since the Neo-Assyrian period (Kubiak-Martens, 2015). However, the most significant source of C_4 biomass may have been the dry steppe, which in the area around the middle Euphrates valley is covered mainly by Artemisia (C_3), Chenopodiaceae (mainly C_4) and Poaceae (both C_3 and C_4) (Kubiak-Martens, 2013). The distribution of d^{13}C values in wild animals suggests that ungulates (gazelle, equids) occasionally grazed on C_4 plants, and rodents even exhibited a preference for C_4 plants, which is clear both from direct and indirect (a fox feeding on rodents) evidence. No direct data from the middle Euphrates valley are available, but rodents living in other arid areas of the Near East feed primarily on chenopods (Kami & Yadollahvand, 2014; Shenbrot, 2004) and this is consistent with the isotopic signal observed here.

The average d^{15}N values are high in humans and animals from the middle Euphrates valley and this may be mainly a result of the effect of low precipitation in the region, only c. 140 mm on average, and the concomitant ^{15}N-enriched diet. Humans living at other sites in Northern Mesopotamia, located in more humid areas, show a much lower average d^{15}N, from 6.8& on average at Bakr Awa on the very humid foothills of the Zagros mountains (Fetner, 2016) to 9.9& at Tell Barri in the Khabur triangle (Sołtysiak & Schutkowski, 2015) and 11.1& at the semi-arid Umm el-Marra site near Aleppo (Batey, 2011).

5.1 | Shift from intensive to extensive agriculture

There are at least four factors that may influence the d^{15}N values in human tissues. Firstly, the availability of water. The marked differences in average d^{15}N values between Mesopotamian sites (see above) are likely the result of differences in average annual precipitation (cf. also Gröcke, Bocherens, & Mariotti, 1997) and its resulting universal effect on nitrogen values in species consumed by humans. The second factor includes agricultural practices; especially manuring with animal dung may increase d^{15}N by more than 6& in cereals (Fraser et al., 2011). Thirdly, floodplains are enriched in ^{15}N (Finlay & Kendall, 2008). The fourth factor relates to the association between d^{15}N and trophic level in that a higher input of animal products in diet is reflected in higher d^{15}N values of the end consumer (Hedges & Reynard, 2007).

The isotopic data from the middle Euphrates valley show a quite clear and consistent picture: there was a shift in d^{15}N values between the Old Babylonian period and the Neo-Assyrian period, by c. 22.5& on average. No sample representing the later periods had d^{15}N above 14&, and in earlier periods the d^{15}N values even reached 18&. On the other hand, several samples representing earlier periods overlapped the range of d^{15}N for later periods and three of them even approached the lower limit of d^{15}N in the whole dataset. However, the shift between earlier and later periods with no major differences within earlier (i.e., Bronze Age) and later temporal subsets is evident.

For growing urban centres that appeared in the middle Euphrates valley during the Early Bronze Age to control trade traffic along the river, supply of grain was a crucial element of their policy. Although the state of Mari occasionally controlled some dry farming areas in the

Khabur drainage and could import grain using this partially navigable river or from upstream Euphrates valley (Chambon, 2011), maximum possible exploitation of narrow strips of arable land available along the Euphrates was necessitated by high cost and uncertainty of grain supply from more distant parts of Mesopotamia on one side, and growing demands of increasing urban population on the other (cf. van Koppen, 2001). In this context, high average d^{15}N values during the periods when the state of Mari flourished as the regional power controlling traffic along the Euphrates may be interpreted as evidence of intensive plant cultivation with high levels of manuring.

Such interpretation is consistent with the dimorphic model of economy suggested by textual sources (cf. Rowton, 1977). During the growing season of cereals ovicaprid herds grazed in the dry steppe and, after the harvest, cleared the stubble and left their dung on the fields. When arable fields were limited, the area was additionally fertilized using waste from the cities or mud from the river (Wilkinson, 1989).

The situation changed when population density decreased after the decline of local states during the Middle and Late Bronze Age. Intensive plant cultivation was no longer supported by centralized state administration and when large empires took control of the land, their policy supported an increase of the economic potential through extension of arable lands (e.g., by large-scale irrigation) rather than through efforts to increase productivity per hectare. The construction of the Nahr Dawrīn canal may be an example of such policy as it dramatically extended the area of fields suitable for agriculture and secured high cereal yield with no need of intensive manuring. Such large-scale and labor demanding investment is quite consistent with the policy of the Assyrian state, which sought to increase agricultural production after the social collapse in the Early Iron Age (cf. Sołtysiak, 2016) using human resources available through mass deportations (cf. Oded, 1979).

Therefore, the shift in the d^{15}N values between the Bronze Age and the later periods was most likely the effect of changing agricultural policy: from intensive exploitation of limited areas suitable for plant cultivation to more extensive use of land, with lower level of manuring, but also with some actions aimed at the increase of available farmlands. A similar transition from more intensive to more extensive agriculture has been observed in the dry farming zone of Northern Mesopotamia and was explained in terms of growing urban population during the Late Chalcolithic and Early Bronze Age (Styring et al., 2017). In the middle Euphrates area, however, this effect was later in time and was even more pronounced because it was triggered by a dramatic re-orientation of subsistence strategies following the collapse of regional independent kingdoms and the establishment of supra-regional empires, rather than by gradual and intrinsic evolution of the local economy.

The results show an interesting threshold in d^{15}N values at around 14&. Most individuals from the Bronze Age and no individual from later periods have higher d^{15}N values than this threshold, while several Bronze Age samples overlap with this range for later periods, sometimes close to the lower limit of this range (cf. Figure 4). Such an effect is consistent with the proposed interpretation, as during the Bronze Age some grain may have been transported from the north, from areas with much higher average precipitation and therefore lower expected d^{15}N values in imported grain; possible migration of people from the north, feeding on local resources in their adolescence, is also a possibility. Moreover, the level of manuring in intensive agriculture may have varied from time to time and from place to place, and therefore higher variability of d^{15}N values is expected with this kind of subsistence strategy.

5.2 | Dry steppe exploitation

As the middle Euphrates valley is located far away from the sea, the most important factor differentiating d^{13}C values is the proportion of C_3/C_4 plants in the human diet, either directly or indirectly through consumed animals or their products. There are also some minor factors, such as the trophic level spacing (Bocherens & Drucker, 2003) and climatic fluctuations, i.e. differences in precipitation (Riehl, 2008). However, the scope of the trophic level effect is relatively small when compared to differences between C_3 and C_4 plants (Bocherens & Drucker, 2003) and in the arid area where any plant cultivation must rely on ground water and not on rain, the climatic effect may be also neglected.

Mesopotamian agriculture was based on C_3 crops, especially barley (which was also dominant in the middle Euphrates valley) and wheat (Riehl, 2009), with some share of pulses. The only C_4 crop present in ancient Mesopotamia was millet, but it always seemed to have been a marginal cultivar, detected in small quantities mainly at Pre-Pottery Neolithic (Hunt et al., 2008) and Neo-Assyrian sites (Nesbitt & Summers, 1988). It should be noted, however, that broomcorn millet was present in the soil samples from the middle Euphrates valley dated to the Shakkannakku, Neo-Assyrian and Late Roman periods (Kubiak-Martens, 2013). It is therefore possible that this drought resistant C_4 cereal was permanently cultivated in this arid area as an alternative summer crop.

On the other hand, macroremains of some plants present in the dry steppe (e.g., *Salsola* sp., a C_4 chenopod, and the Syrian mesquite, *Prosopis farcta*, a C_3 plant) have been found in the 3rd millennium strata at Tell Ashara, and they most likely derived from animal dung (Kubiak-Martens, 2013). Therefore, use of the dry steppe for pasture may have also increased the d^{13}C values in ovicaprids and (indirectly) in humans feeding on their meat and dairy products.

Among six temporal subsets analyzed in the present paper, two shifts in d^{13}C were detected. The first enrichment in ^{13}C by c. 1& on average occurred between the Old Babylonian and Late Roman periods, with the very small Neo-Assyrian sample in an intermediate position. Although small, this enrichment is not only statistically significant, but also paralleled by a similar effect observed at two other sites in Northern Mesopotamia: Tell Barri, where the chronology of this small shift is better specified as the transition from the Middle to Late Bronze Age (Sołtysiak & Schutkowski, 2015) and Bakr Awa, where the dating is even less precise than in the middle Euphrates valley (Fetner, 2016). At all these sites the shift was statistically significant and less than 1& (from 219.43 to 219.07& on average at Tell Barri, from 219.90 to 219.17& at Bakr Awa and from 219.76 to 218.91& in the middle Euphrates valley; all early and late subsets except the Modern one).

At Tell Barri this shift coincided with a dramatic regional decrease in the proportion of pigs among domesticated animals and therefore is was interpreted as the result of greater reliance on ovicaprid grazing in the dry steppe (Sołtysiak & Schutkowski, 2015). However, at Tell Ashara and Tell Masaikh, a completely different pattern is observed: low quantities of pig remains are present only in bone assemblages from later periods. No samples from the Late Bronze Age were analyzed here and it is not clear whether this enrichment in ^{13}C in the middle Euphrates valley occurred before or after this period. There are therefore two possible explanations of this effect. Either there was a greater share of millet among consumed plants, a possibility that seems to be supported by available archaeobotanical evidence (Kubiak-Martens, 2015). Alternatively, a larger emphasis on cattle and pig husbandry after large-scale irrigation of the Euphrates valley may have pushed ovicaprid herding from more humid zones below the upper terrace to the dry steppes surrounding the settlements and forced animals to graze on pastures with a higher proportion of C_4 plants.

A much more pronounced shift in $d^{13}C$ values occurred between the Islamic period (i.e., 7th–13th c. CE) and the Modern period (i.e., 19th–20th c. CE). On average, this enrichment in ^{13}C was much higher than the previous one (c. 2.5& on average), but taking into account the bimodality of $d^{13}C$ distribution in the Modern period, the actual enrichment displayed a range of 1.2 and 4.5& on average, respectively, for two identified modes.

There is an obvious culture-historical explanation of this effect, since between these periods a major disruption in the history of Mesopotamia occurred due to invasion of the Mongols and fall of the Caliphate in 1258 (May, 2016). During that time, the agricultural population of Northern Mesopotamia was decimated (Ashtor, 1976) and the area of the middle Euphrates remained very sparsely populated until 17th century when Bedouin tribes from the Arabian Peninsula moved their flocks to the steppes around the valley (Raswan, 1930). In early 20th century, the French authorities settled some farmers from southeastern Anatolia and western Syria in the valley and also forced sedentarization of Bedouins, which however was only partially successful (Velud, 2000).

The cemetery at the top of Tell Ashara belonged to this Bedouin population that based their subsistence on ovicaprid herding both in the river valley and on the dry steppes around. The bimodal distribution may reflect the process of sedentarisation and the difference between individuals that still exercised the nomadic life of sheepherders and those that settled and became sedentary farmers. Also frequent differences in $d^{13}C$ values between M2 and M3 in this temporal subset, and especially three individuals with $d^{13}C$ decreasing over time, suggest (small numbers acknowledged) relatively rapid lifetime changes in diet that may be the consequence of this transition from nomadic pastoralism to agriculture or a mixed subsistence strategy.

5.3 | Diet, mobility, and social structure

Differences in diet between males and females have been proposed for many societies with males often preferring animal-related food rich in proteins and females preferring plant-related food rich in carbohydrates (Wansink, Cheney, & Chan, 2003). In the studied sample no such differences were observed and it is likely that the diet of both sexes was similar, at least during childhood and adolescence.

The three-modal distribution of the $d^{15}N$ values among eight males from the Shakkanakku and Old Babylonian periods reveals an interesting pattern: half of them cluster together above the maximum value for later periods, but there are two data pairs within the $d^{15}N$ range for later periods, one close to the maximum limit and one close to the minimum limit of this range. It is not very likely that such differences were related to diet, but clear differences in average $d^{15}N$ values between Tell Ashara/Tell Masaikh, Tell Barri and Bakr Awa suggest that these individuals may have migrated from areas with higher annual precipitation (e.g., the dry farming zone north and north-east of the middle Euphrates valley) or consumed large amounts of cereals imported from the north, even though independent corroboration from other isotope systems is not available. During the Early and Middle Bronze Age Tell Ashara-Terqa was one of centers controlling the major trade road along the Euphrates (Liverani, 2014) and therefore high mobility of people living in this place and at that time may be expected.

Although no data about the social status of individuals buried at Tell Ashara and Tell Masaikh are available, the isotopic evidence allows some insight into the social complexity of the middle Euphrates valley. Apart from possible evidence of a transition from mobile to sedentary life in the Modern period (see above), there are also a few other examples of lifetime shifts in $d^{13}C$ values; one dated to the Late Roman period indicating a higher share of C_4 plants, and one dated to the Shakkanakku period reflecting a shift in the opposite direction. They perhaps witness possible changes from farming to husbandry and inversely, but their low number before the Modern period rather suggest stability of subsistence, in spite of textual evidence from the Old Babylonian period reporting movement between the sedentary and mobile segments of the local population (Matthews, 1978).

In the Modern subset, apart from the observed bimodality in $d^{13}C$ values, there are two individuals showing not only relatively low $d^{13}C$ values, but also very low $d^{15}N$ values. It is possible that they were immigrants from more humid regions, relocated to the middle Euphrates valley within the re-settlement program of the early 20th century.

6 | CONCLUSION

Stable carbon and nitrogen isotope values in dentine of populations inhabiting the region of Tell Ashara-Terqa has produced new data that allow a better understanding of the economic history in this part of Northern Mesopotamia. First of all, as a proxy for prevailing subsistence strategies, they provide evidence of a dramatic shift, as witnessed by the clear decrease of average $d^{15}N$ values, from more intensive to more extensive plant cultivation between the Old Babylonian and Neo-Assyrian period, an effect most likely related to different agricultural policies in the Bronze Age regional states and in the supra-regional empires following the establishment of the Neo-Assyrian state.

Temporal shifts in the $d^{13}C$ values between the Bronze Age and later periods are not easy to interpret and it is equally possible that they were related to a moderately higher share of millet in crops or to more intensive exploitation of dry steppes as pastures. For the Modern period, however, they provide clear evidence of mobile ovicaprid herding in the dry steppe and also evidence of sedentarization of Bedouin tribes at the beginning of the 20th century.

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SUPPORTING INFORMATION

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