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1 Lipid preservation in ceramics and bones from the Iranian Plateau: implications
2 for dietary and chronological reconstructions

3

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20

21 **Abstract**

22 This paper presents the first investigations of lipid preserved in bones and ceramics, from sites of
23 various ages and depositional environments in Iran with the aim of using them in palaeodietary and
24 chronological reconstructions. We also characterized modern dairy products from the region using
25 stable carbon isotope values to provide a local reference range for dairy lipids identification in
26 archaeological ceramics. The dataset recorded on pottery suggests lipids are recovered in
27 appreciable concentrations regardless of the environment or the age of settlements, but rather the
28 vessel function may play an important role. The results showed exploitation of ruminant adipose and
29 dairy products in pottery vessels from the Late Neolithic and Chalcolithic periods. Our dataset
30 suggests lipids in pottery are viable materials for palaeodietary and chronological reconstructions for
31 the Iranian region. Lipid preservation in bones, similar to collagen, is more variable. Lipid and
32 collagen preservation in bones are not directly related and one of the bones studied showed
33 diagnostic lipids in a context of low collagen preservation. This suggests lipids have some limited
34 potential to be used for chronological reconstructions as an alternative to collagen when the latter
35 does not survive.

36

37 **Key words**

38 Lipid residue analysis, collagen preservation, stable isotopes, dairy products

40 Introduction

41 Marking the Eastern wing of Fertile-Crescent with the Zagros Mountains, Iran is a key region for the
42 early emergence of agriculture and pastoralism. This high Plateau, limited in the North and the West
43 by two imposing chains of mountains, presents a large basin with deserts known as Dasht-e Kavir and
44 Dasht-e Lut. The country also presents two coastal areas, the Caspian Sea in the North and the
45 Persian Gulf in the South, with several large rivers system, highly threatened today by a growing
46 drought and dam constructions. Due to its geography and vast area, Iran shows diverse climates,
47 ranging from continental, humid, subtropical, semi-arid and arid with very large temperature
48 amplitude including extreme winter conditions in the West and Northwest of Iran and extreme heat
49 in the Lut desert (Lyons et al 2020).

50 Biomolecular archaeology, which studies organic molecules surviving in archaeological matrices
51 (Brown and Brown, 2011), is increasing substantially in terms of methodological development and
52 applications to various materials and regions. The study of bones, ceramics, seeds or coprolites with
53 a molecular and isotopic approach allows information to be gained on prehistoric population
54 behaviour, economy and interactions with the environment. The exploration of some aspects of
55 biomolecular archaeology, i.e. lipids, is nonetheless scarce for the Iranian region and potential exists
56 of further investigation. The various climatic environments existing in Iran can likely have effects on
57 the lipid preservation in archaeological artefacts across the country.

58 Developed about 30 years ago organic residue analysis of lipids preserved in pottery vessels has
59 imposed itself as a key method to study ancient diet, agriculture and culinary practices (Evershed,
60 2008; Evershed et al., 1990). Lipids in pottery and entrapped in the clay matrix remain protected
61 from contaminants in the burial environment (Evershed, 2008; Evershed et al., 1990). Based on
62 molecular and isotopic analyses at the molecular level, this method allows identification of specific
63 food products likely to have been processed in pottery vessels. Commonly recovered in pottery
64 vessels, degraded animal fats are identified by the presence of $C_{16:0}$ and $C_{18:0}$ fatty acids (FAs) with
65 their source being determined using the $\delta^{13}C$ values on individual FAs. The use of local $\delta^{13}C_{16:0}$ and
66 $\delta^{13}C_{18:0}$ values on FAs from reference animals, fed on controlled diet comparable to prehistoric ones
67 are necessary, as the $\delta^{13}C$ values are influenced by the diet and environment (Copley et al., 2003a;
68 Roffet-Salque et al., 2017). The use of $\Delta^{13}C$ ($= \delta^{13}C_{18:0} - \delta^{13}C_{16:0}$) values removes the environmental
69 effect and is known as the universal proxy. This proxy shows nonetheless some limits for the
70 identification of the mixing of food commodities within the same vessel. For this reason, recording
71 local ranges for a particular region would be beneficial for better interpreting the results, especially if
72 some mixing of commodities existed. There are to date, no modern references from Iran and the
73 Near East. Based on reference fats available from more arid environments such as Libya (Dunne et
74 al., 2012) and Kazakhstan (Outram et al., 2009), $\delta^{13}C$ values higher than the modern references of
75 adipose and dairy products from UK ruminants fed on a pure C_3 diet are expected for Iran.

76 Lipid residue analyses in pottery vessels are widespread across Europe, Africa and western Asia
77 particularly for the Neolithic period (e.g. Casanova et al., 2020a; Chakraborty et al., 2020; Courel et
78 al., 2021; Cramp et al., 2019; Debono Spiteri et al., 2016; Dunne et al., 2012; Evershed et al., 2008;
79 Grillo et al., 2020; Lucquin et al., 2016; Oras et al., 2017; Outram et al., 2011; Stojanovski et al.,
80 2020). With the exception of Turkey, Syria and Israel for which substantial pottery assemblages have
81 been studied (Evershed et al., 2008; Nieuwenhuys et al., 2015), the Near East and Southwest Asia
82 have been less studied than other regions. So far studies of lipid from Iran are limited to one
83 Chalcolithic potsherd from Tepe Sofalin where beeswax lipids were identified (Mayyas et al., 2012)

84 and an assemblage of 20 pottery vessels from the Iron Age site of Kani-Zirin (Qanbari-Taheri et al.,
85 2020) where plant and animal lipids were identified; the study does not provide stable carbon
86 isotope analyses to distinguish the source of animal fats. The scarcity of analyses from this region
87 means Iran and surrounding areas remain an unexplored ground for lipid residue analyses in
88 ceramics and their use in palaeodietary reconstructions.

89 The analysis of organic matter in bones relies mainly on collagen for radiocarbon dating or
90 palaeodietary reconstructions. Previous studies on archaeological bones from Iran showed collagen
91 preservation is not affected by age but rather climate (Bocherens et al., 2000). The dry and cold
92 environments are favourable for collagen preservation compared to the warmer regions of the
93 country (Bocherens et al., 2000). Aridity is therefore affecting the preservation of collagen and
94 impacting paleodietary and dating programs based on collagen in the most arid areas of the country.
95 While the mechanisms of degradation of bone collagen are understood, the degradation of other
96 biomolecules in bones such as lipids still require investigations (Collins et al., 2002). The study of
97 lipids preserved in bones is uncommon compared to collagen. Early studies showed cholesterol and
98 fatty acids to be the main lipids preserved in human bones thus are considered diagnostic lipids the
99 palaeodietary determinations (Evershed et al., 1995; Stott and Evershed, 1996; Stott et al., 1999).
100 Stable carbon isotope analyses of cholesterol showed values be representative of the last year of diet
101 compared to collagen or apatite that reflect the last 10-25 years of the adult life (Stenhouse and
102 Baxter, 1979; Hedges et al., 2007). More recent attention to lipid extraction and contamination
103 removal protocol for the study of archaeological bone lipids have been undertaken (Colonese et al.,
104 2015). These studies on human and animal bones from the UK, Scandinavia, Spain and Brazil
105 highlighted the potential of bone lipids to be used as a palaeodietary indicator complementary to
106 bone collagen analysis (Evershed et al., 1995; Stott and Evershed, 1996; Stott et al., 1999; Colonese
107 et al., 2015). The use of bone lipids biomarker in palaeodietary reconstructions remain overall scarce
108 and not a common practice.

109 Bone collagen is one of the most targeted fractions for radiocarbon dating of archaeological sites.
110 The aridity of the Iranian region affecting bone collagen preservation means that? other materials or
111 carbon fraction need to be targeted (Bocherens et al., 2000). In such cases bone bioapatite fraction
112 can be used as an alternative to date archaeological bones without collagen, returning a minimum
113 age determination (Zazzo and Saliège 2011). Other compounds such as charcoal or charred remains
114 are also used as alternatives. Recently development for the direct radiocarbon dating of lipids
115 preserved in pottery vessels offers a new alternative to date archaeological sites lacking conventional
116 materials for ^{14}C dating, to date directly the period of use of pottery vessels and to date specific food
117 commodities (Casanova et al., 2020c). This method targets the most abundant and most widely
118 recovered compounds preserved in the lipid extracts, the palmitic and stearic acids. Pottery vessels
119 from arid areas, such as Libya and Turkey were radiocarbon dated by this method (Casanova et al.,
120 2020c) demonstrating the possibility to ^{14}C date well preserved pottery lipids from arid areas. The
121 potential of lipids from bone as a viable source of carbon for chronological studies using the
122 radiocarbon method has nonetheless not yet been addressed. As mentioned previously the aridity
123 affects bone collagen preservation in Iran. Exploring other options to obtain radiocarbon
124 measurements from pottery and bone lipid fraction can provide suitable new alternatives for
125 chronological studies in the Iranian plateau.

126 This paper aims at a first view of lipid preservation in ceramics and bones in various environments
127 from Iranian context and assesses their potential for use in palaeodietary and chronological
128 investigations. For bone lipid preservation we particularly focus on the chronological aspect. An
129 additional aim of the study was to start establishing a baseline with local modern references.

130

131 **Materials and methods**

132 **Sites and materials**

133 Archaeological materials including 41 potsherds and 28 bones from eight sites were selected to
134 evaluate the lipid preservation in various burial environments of the Iranian Plateau (Table 1, Figure
135 1a, Supplementary Information). Three sites, Tappeh Kholah Kouh (transition from Neolithic to
136 Chalcolithic), Ālou (Late Chalcolithic) and Tappeh Zāgheh Ābyek (transition from Neolithic to and
137 Early Chalcolithic), are located in the semi-arid/steppe environment of the Qazvin Plain (Davoudi et
138 al., 2021, 2019a, 2019b; Moghimi and Davoudi, 2020). The site of Qazānchi (Pre-Pottery, Late
139 Chalcolithic and Middle Bronze Age) and Wezmeh (Late Neolithic/Chalcolithic) are located in a
140 Mediterranean/sub-tropical forest environment of Kermanshah (Mashkour et al., 2020; 2021), the
141 site of Shahrān (Late Neolithic/Early Chalcolithic and Iron Age) in the humid forestry environment of
142 Gilan and the site of Nishāpur (Islamic period) is from the semi-arid/steppe environment of the
143 Khorasan Razavi region (Laleh and Lorzadeh, 2019; Figure 1a). The site of Tepe Sohz (Chalcolithic) is
144 located in the semi-arid environment of the Behbahan plain. All the sites phases were assigned
145 relatively by pottery typology.

146 The bones from these sites correspond to human ($n=4$) and caprine ($n=12$), cattle ($n=3$), deer ($n=1$),
147 small ruminant ($n=6$) and large mammals ($n=3$; Figure 1b). The bones from Tepe Sohz and Wezmeh
148 showed poor collagen preservation for ^{14}C dating in previous programs (thus remained unpublished).
149 All potsherds derived from common plain coarse or painted vessels (Figure 1c), except for three
150 vessels from Nishāpur that correspond to specialised vessels named “*Fuqā‘ā*” (grenade shape).

151 Modern goat dairy products of *Kashk* ($n=5$), a dried casein paste, and *Qara qurut* ($n=1$; a black
152 residue from *Kashk* production used as condiment; Figure 1d) produced by contemporaneous mobile
153 pastoralists of the Zagros Mountains were collected in the Zagros mountains region by MM during
154 several visits to Bakhtiari nomads and analysed for their lipid molecular and isotopic signatures. The
155 aim of these analyses is to provide some local references for Iran and gain some information on
156 these typical products of the Near East (easily transportable as they are dried) which could have a
157 long tradition (Digard, 1981). Some studies previously focused on the characterization of the volatile,
158 chemical and protein component (Iranmanesh et al., 2018; Mashak et al., 2014; Pourjoula et al.,
159 2020) but not the stable carbon isotope signatures.

160 **FTIR analyses**

161 FTIR analyses on modern dairy products were performed to identify the presence of lipids in these
162 dehydrated casein pastes. FTIR spectra were recorded on a Perkin Elmer Spectrum 100
163 spectrometer. Less than a 1 mg of sample was pressed using an ATR sampling accessory and the data
164 were processed using Spectrum IR software.

165 Collagen preservation was evaluated using ATR-FTIR by quantifying collagen using a peak area ratio
166 approach on the amide I and phosphate bands following the method presented in Lebon et al.,
167 (2016). Spectra were recorded on a Vertex 70 (Bruker) spectrometer. Less than 1 mg of bone
168 powder was pressed against diamond crystal using a single reflection diamond ATR accessory
169 (Extended Range Diamond Quest accessory, Specac). ATR-spectra were acquired with a spectral
170 resolution of 4 cm^{-1} and the accumulation of 32 scans in the range of $4000\text{--}400\text{ cm}^{-1}$. Upon
171 comparison with modern bone, collagen is considered datable when the percentage of collagen
172 preservation is superior to 4% (Lebon et al., 2016)

173

174 **Lipid residue analyses**

175 A piece of about 10 mg from modern dairy products was carefully removed with a scalpel for
176 analysis. The surface of ceramics and bones were cleaned using a modelling drill (~1 g for ceramics
177 and ~0.5 g for bones) then ground to fine powder in a mortar for their analyses. Ceramic powders
178 were prepared at the Bioarchaeological laboratory at the University of Teheran before their export
179 for extraction and analysis at the University of Bristol. The whole procedure for bones and modern
180 dairy products was performed at the University of Bristol. The powdered samples were transferred to
181 a culture tube to which a known amount of an internal standard (*n*-tetratriacontane) at a
182 concentration of $1\mu\text{g}\cdot\mu\text{L}^{-1}$ was added for lipid quantification.

183 Lipids were extracted from modern dairy products (both on the surface and the middle), ceramic
184 powder and bone powder using an acidified solution of methanol ($\text{H}_2\text{SO}_4/\text{MeOH}$ 4% v/v, 5 mL, 70°C, 1
185 h) followed by a liquid/liquid extraction (*n*-hexane, 4x3 mL) and concentrated under a nitrogen
186 stream, based on published procedures (Correa-Ascencio and Evershed, 2014). The same protocol
187 developed for extraction of lipids in pottery vessels (Correa-Ascencio and Evershed, 2014) was used
188 for archaeological bones, as it was shown that the use of acid solution is also relevant for bone lipids
189 extraction (Colonese et al., 2015). During the extraction fatty acids (FAs) are simultaneously
190 derivatised into fatty acids methyl esters (FAMES). An aliquot was treated with BSTFA (*N,O*-
191 Bis(trimethylsilyl)trifluoroacetamide, 20 μL) for derivatisation, then dissolved in an appropriate
192 amount of solvent for their analyses by gas chromatography (GC), GC mass spectrometer (MS) and
193 GC-combustion-isotope ratio MS (GC-C-IRMS).

194 Lipids (1 μL) were injected in an Agilent Technology 7890A GC-FID (flame ionisation detector)
195 instrument for lipid quantification. Analyses were performed on a non-polar column (15 m x 0.32 mm
196 i.d., DB1 stationary phase, 0.17 μm film thickness). The GC oven temperature was held for 2 min at
197 50 °C (40°C for the GC-C-IRMS) and increased to 300 °C at 10 °C.min⁻¹ and held for 10 min.
198 Quantification of archaeological lipids was performed using the peak areas of the internal standard of
199 known amount and concentration, while removing the contribution of any modern contamination
200 (e.g. plasticizer) detected in the TLEs.

201 Another aliquot of the TLEs was injected into a Trace GC coupled to a Finnigan Trace mass
202 spectrometer (MS) instrument and into an Agilent Technologies 7890A, coupled to an Isoprime GC5
203 combustion interface and an Isoprime 100 MS (TLEs from ceramics only) for lipid structure and
204 source identification. Columns and temperature program are the same as mentioned above with a
205 starting temperature program at 40°C for the GC-C-IRMS analyses. GC-C-IRMS measurements
206 accuracy were controlled using an in-house standard solution of FAMES in between two samples, and
207 calibrated against the Pee Dee Vee international standard. The $\delta^{13}\text{C}$ measurements of FAMES were
208 then corrected for the derivative carbon using a mass balance to obtain the $\delta^{13}\text{C}$ values of the FAs
209 (Rieley 1994). The mean of $\delta^{13}\text{C}$ FAs values agreeing within 0.5‰ (two for pottery lipids, three for
210 dairy products) corresponds to the final values reported. The modern dairy products were
211 additionally extracted using a chloroform/methanol solution (2x10 mL, sonication 20min), following a
212 published procedure (Evershed et al., 1990), to identify the full TAGs, DAGs and MAGs distributions
213 in high temperature (HT) GC and HTGC-MS analyses. The column and temperature program for HTGC
214 and HTGC-MS are the same as described above excepted that final temperature was set at 350°C and
215 held for 15 min.

216

217 **Results**

218 **Modern references of dairy products produced in the Zagros Mountains**

219 FTIR analyses were performed on *Kashk* and *Qara qurut* as a first investigation of whether a lipid
220 fraction remains through the *chaîne opératoire* of producing these dehydrated and proteinaceous
221 dairy products.

222 FTIR analyses show all *Kashk* samples (Figure 2a) display a large band of medium intensity centered
223 at 3274 cm⁻¹ (N-H and/or O-H stretching), two medium bands at 2922 cm⁻¹ and 2853 cm⁻¹ (C-H
224 asymmetric and symmetric stretching), intense peaks at 1621 cm⁻¹ (N-H stretching of amides) and
225 1527 cm⁻¹ (N-H bending and C-N stretching), a medium intensity band at 1450 cm⁻¹ (N-H bending) and
226 medium intensity bands at 1228 cm⁻¹ and 1078 cm⁻¹ (C-N stretching) and a band of low intensity at
227 924 cm⁻¹ (C-H₂ rocking). These bands are all consistent with amides, carboxylic and aliphatic carbon
228 functions found in proteinaceous compounds such as casein. A lipidic component is also visible to a
229 lesser extent by the presence of a sharp band of medium intensity at 1740 cm⁻¹ (C=O of stretching
230 carboxylic acids) and medium intensity bands at 1170 cm⁻¹ and 1110 cm⁻¹ (C-O stretching).

231 The *Qara qurut* FTIR spectrum (Figure 2b) differs significantly from the *kashk* spectrum. This was
232 expected as this product is a residue from the *kashk* production. The intense bands at 1621 cm⁻¹,
233 1527 cm⁻¹ and 1450 cm⁻¹ characteristics of amides functions are missing, but a large band centred on
234 1574 cm⁻¹ is visible and suggest C-O stretching bonds of carbohydrates rather than the C-N stretching
235 of amides. The FTIR spectrum also displays an intense band centred at 3200 cm⁻¹ (O-H stretching), at
236 1400 cm⁻¹ and 1274 cm⁻¹ (C-O stretching of ether), at 1047 cm⁻¹ (C-O stretching of alcohols) are
237 coherent with the presence of ether functions found in lactose. The bands at 2978 and 2926 cm⁻¹ (C-
238 H stretching), at 1720 cm⁻¹ (C=O of stretching carboxylic acids) and medium intensity bands at 1117
239 cm⁻¹ (C-O stretching) suggest a lipid component remains in the *Qara qurut* composition.

240 *Kashk* and *Qara qurut* were further analysed for their lipid contents by GC, GC-MS and GC-C-IRMS
241 using two extraction methods, a solvent method that preserves all lipid classes and an acidic method
242 that hydrolyzes the esters and makes them amenable directly for GC-MS and GC-C-IRMS analyses
243 (Table 2). The TLEs, after acid treatment, display a lipid distribution dominated by the C_{16:0}, C_{18:1} and
244 C_{18:0} FAs for all of the products (Figure 2c1). The total lipid extracts (TLEs) obtained through the
245 solvent extraction showed intact triglycerides with T28 to T54 carbon chain length (Figure 2c2). These
246 components were of relatively low intensity in the *Qara qurut* compared to the *Kashk* samples.

247 The lipids correspond on average to 9.3 ± 7 % of the mass, with extreme values of 1 to 19%, (solvent
248 method) or 4.4 ± 4 % of the mass (acidic method; two samples taken from the outer and the middle
249 part), with extreme values of 0.9 to 8 %. The *Qara qurut* contained, however, 1% of lipids (values
250 obtained using both methods). The results support a relatively low lipid content, as was expected
251 from these dehydrated dairy products. It also indicates that only a small portion of lipids goes into
252 the *Qara qurut* formation during *Kashk* production.

253 The δ¹³C_{16:0} and δ¹³C_{18:0} values were recorded on the TLEs from the acid treatment. The δ¹³C_{16:0} and
254 δ¹³C_{18:0} values (mean of values obtained from 3 replicates of the same sample) were corrected for the
255 post-industrial revolution effects of fossil fuel burning (Copley et al. 2003). They are enriched
256 compared to modern reference ellipses of ruminant dairy products from UK animal raised on a pure
257 C₃ diet (Table2; Figure 2d1). They remain coherent with the universal dairy products range using Δ¹³C
258 (=δ¹³C_{18:0} - δ¹³C_{16:0}) values (Figure 2d2). This dataset gives a first range of the regional δ¹³C values of
259 dairy products and the shift from the δ¹³C values from the UK C₃ raised animals reference database,
260 is coherent with δ¹³C reference values from other arid countries, such as Libya or Kazakhstan (Dunne
261 et al., 2012; Outram et al., 2009).

263 **Lipid preservation in archaeological ceramics**

264 A total of 14 TLEs were shown to be contaminated with modern fats, likely originating from post-
 265 excavation cleaning treatments. This was evident from the C_{18:1} fatty acid being present in
 266 abundances comparable to the C_{16:0} and C_{18:0} fatty acids. This unsaturated fatty acid usually degrades
 267 in an archaeological context, so its recovery in high abundances is unlikely. In addition, these TLEs
 268 present a cluster of peaks eluting before the internal standard (IS) that corresponds to modern
 269 contamination. These TLEs were likely contaminated by an industrial detergent used that contained
 270 triglycerides despite them not being listed in the chemical list; it was removed from the cleaning
 271 procedure during preparation of the other potsherds. These TLEs were disregarded in the remainder
 272 of the analyses as the archaeological origin of C_{16:0} and C_{18:0} fatty acids are dubious in this case.
 273 Therefore, the results discussed hereafter are on the 27 remaining potsherds. Most TLEs also showed
 274 some modern plasticizer contamination, likely arising from their storage in plastic bags.

275 The remaining potsherds ($n=26$ out of 27) presented lipid concentration in appreciable abundances
 276 ($>5 \mu\text{g}\cdot\text{g}^{-1}$ of sherds) with an average lipid concentration of $108 \mu\text{g}\cdot\text{g}^{-1}$ of clay with the maximum
 277 concentration reaching $514 \mu\text{g}\cdot\text{g}^{-1}$ (Table 3, Figure 3). A total of 15 TLEs (or 55% of the
 278 uncontaminated sherds) were dominated by the C_{16:0} and C_{18:0} fatty acids characteristic of degraded
 279 animal fats (Figure 3a,b). These included $n=4$ of the TLEs from Kholāh Kouh, $n=2$ of the TLEs from
 280 Ālou, $n=4$ from Zāgheh Ābyek, $n=3$ from Qazānchi, $n=1$ from Nishāpur and $n=1$ from Shahrān with a
 281 maximum lipid concentration of 273, 246, 474, 104, 38 and $514 \mu\text{g}\cdot\text{g}^{-1}$ of clay, respectively. At the site
 282 of Nishāpur one of the two undecorated vessels showed appreciable lipids with an even numbered
 283 distribution of fatty acids (C_{14:0}-C_{18:0}) dominated by the C_{16:0} FAs, unsaturated C_{16:1} and C_{18:1} FAs, a
 284 suite of even numbered n -alkanols (C₁₄-C₂₀), and a suite of n -alkanes (C₂₀-C₂₈). The presence of
 285 unsaturated FAs is not dubious in this case as this ceramic is from a relatively recent period (Islamic),
 286 where survival of such molecules in elevated concentration remains possible. This lipid distribution
 287 suggests plant oil processing in this particular vessel (Gunstone, 2004; Dunne et al., 2016). One of the
 288 *Fuqā'a* showed a weak preservation of animal fats, the other two, however, showed low lipid
 289 recoveries that could not be traced to specific products and would need further investigation with
 290 high resolution GC-MS.

291 The $\delta^{13}\text{C}$ values of individual fatty acids were recorded for all the sites (except Nishāpur where the
 292 concentration was too weak to be determined) and displayed values characteristic of non-ruminant
 293 ($n=1$) ruminant adipose ($n=7$) and ruminant dairy fats ($n=7$; Figure 4c, d). None of the data plots in
 294 the modern reference range of ruminant animal products raised in a pure C₃ diet and display
 295 elevated $\delta^{13}\text{C}_{16:0}$ values typical of arid environments (Copley et al., 2003a; Dunne et al., 2012; Outram
 296 et al., 2009). Half of $\delta^{13}\text{C}_{16:0}$ and $\delta^{13}\text{C}_{18:0}$ values assigned to ruminant dairy fats fits within the local
 297 range of $\delta^{13}\text{C}_{16:0}$ and $\delta^{13}\text{C}_{18:0}$ values from modern *Kashk* and *Qara qurut*. The remainder display
 298 enrichment in ^{13}C compared to the UK reference values. TLEs from the site of Zāgheh Ābyek
 299 originates from both ruminant adipose ($n=2$) and ruminant dairy ($n=2$), and TLEs from the site of Ālou
 300 are of ruminant adipose origin ($n=2$). Both sites showed higher $\delta^{13}\text{C}$ values than for the site of Kholāh
 301 Kouh where ruminant adipose ($n=3$) and ruminant dairy ($n=1$) fats were identified. These three sites
 302 are in the same local area suggesting access of their livestock animals to pastures containing either C₃
 303 or C₄ plants. The $\delta^{13}\text{C}$ values on individual FAs from the site of Qazānchi are characteristic of ruminant
 304 dairy fat ($n=2$) and non-ruminant adipose fats ($n=1$). The latter show lower $\delta^{13}\text{C}$ values than reference
 305 range of porcine adipose fats (Figure 3c,4c) which suggests, if not an outlier, an aquatic contribution.
 306 Finally, the $\delta^{13}\text{C}$ values of individual FAs at Shahrān are characteristic of ruminant dairy fats ($n=1$).

307 Overall, the lipid distribution and concentration are similar for all the environments (Table 3, Figure
308 3) of ceramics in the region. The site of Nishāpur is the only one with a specific pottery type, the
309 *Fuqā'a*, which showed a low lipid content likely related to the vessel function although one of them
310 showed low content of animal fats. Some dairy products were processed in pottery vessels at four of
311 the study sites, suggesting these would have been available as early as the Late Neolithic (Shahrān,
312 Qazanchi) and pursued in the Chalcolithic period. It should, nonetheless, be noted that this pilot
313 study is based on a low number of sherds only, so a proper picture of the use of pottery for specific
314 food products processing in prehistoric Iran is not possible from this dataset.

315

316 **Lipid and collagen preservation in archaeological bones**

317 The lipid recoveries from the 28 bones analysed averaged $34.6 \mu\text{g.g}^{-1}$ of bone and ranged from 0.5 to
318 $171.4 \mu\text{g.g}^{-1}$ (Table 4, Figure 3). A total of thirteen bones (46 %) were dominated by diagnostic lipids
319 (i.e. cholesterol and/or fatty acids; Figure 5 a,b ; Evershed et al., 1995). Other lipid compounds in
320 some bones could likely come from the soil, thus of exogenous origin. Diagnostic lipids were
321 identified, $n=3$ from Shahrān, $n=3$ from Ālou, in $n=4$ from Kholāh Kouh, in $n=2$ from Qazānchi, and in
322 $n=1$ from Wezmeh. The sites of Tappeh Zāgheh Ābyek and Tepe Sohz showed the poorest
323 preservation with no diagnostic lipids identified. The lipid concentration of bones with diagnostic
324 lipids is quite variable. It is rather low with a maximum of $21 \mu\text{g.g}^{-1}$ at Ālou but reaches over $100 \mu\text{g.g}^{-1}$
325 at Kholāh Kouh, Qazānchi and Wezmeh. The sites of Qazānchi and Wezmeh showed the highest
326 lipid concentration of bones with diagnostic lipids compared to the other sites (Figure 3). This
327 difference could be related to their depositional environment (here Mediterranean
328 (subtropic)/forest), the sample size is nonetheless too small to draw a pattern based on the burial
329 environment or ages of the sites.

330 For the purpose of investigating the potential of bone lipid as an alternative fraction in case of poor
331 collagen preservation for radiocarbon dating, we assessed as well collagen preservation in the
332 studied bones. FTIR analyses on bones for collagen preservation prediction (Lebon et al., 2016)
333 suggest that collagen is well preserved (i.e. > 4%) for all the bones from the sites of Kholāh Kouh,
334 Shahrān, Ālou and Zāgheh Ābyek and $n=3$ of bones from Qazānchi (Table 4, Figure 5c,d). Four bones
335 from Qazānchi had a low collagen content, as did the two bones from Tepe Sohz and one from
336 Wezmeh (< 4%), from which no collagen could be extracted.

337 Figure 6 represents the lipid concentration in bones with diagnostic lipids identified plotted against
338 the percentage of collagen preserved in bones. The linear trendline set to intercept with the origin
339 gives a correlation coefficient of 0.7415 which supports no direct correlation of lipid and collagen
340 preservation for the studied bones. The sites of Kholāh Kouh, Qazānchi and Wezmeh showed the
341 highest preservation of collagen with bones presenting over 10% of collagen. These bones also
342 showed diagnostic lipids in appreciable concentrations. Interestingly two bones from Qazānchi show
343 good collagen and lipid preservation, one good collagen preservation without the survival of lipids
344 and three both poor lipid and collagen preservation. Preservation of the organic matter in bones at
345 this site is highly variable and raises questions about these two contrasting patterns of preservation.
346 The bones from the sites Shahrān and Ālou showed that sufficient collagen was preserved (>4%) with
347 some relatively low preservation of diagnostic lipids. The preservation of both lipids and collagen in
348 appreciable concentrations suggests palaeodietary studies on bone lipid and collagen at these sites
349 are possible. Palaeodietary reconstruction at Tappeh Zāgheh Ābyek is likely limited to bone collagen,
350 which appears well preserved compare to bone lipids, and seem not possible at Tepe Sohz which
351 showed no collagen or diagnostic lipid preservation in the bones. Again, it should be noted this

352 dataset is too small to draw definitive conclusions. One of the bones from Wezmeh showed
353 diagnostic lipids (fatty acids and cholesterol) in relatively elevated concentrations (171 $\mu\text{g}\cdot\text{g}^{-1}$ of
354 bone). Collagen preservation prediction from this bone suggested it to be datable, however, in
355 practice no collagen could be extracted using conventional extraction methods, denoting poor
356 collagen preservation. This highlights the possibility of lipid survival in cases of poor collagen
357 preservation which could be use as alternative for dietary and chronological reconstructions.

358 This difference of bone lipid and collagen preservation between these sites could be related to the
359 depositional environment, as previous studies showed it affects collagen preservation in the region
360 of Iran (Bocherens et al., 2000). Study of a larger dataset of bones would be required to confirm this
361 hypothesis.

362

363 **Discussion**

364 **Implications for dietary studies**

365 The dataset presented on lipid preservation in ceramics from Iran suggests lipid residue analysis
366 programs have potential for the regional dietary reconstruction and that the climatic variation in
367 deposition conditions in the region is unlikely to cause lipid preservation problems for this country
368 (Figure 3). The range of $\delta^{13}\text{C}$ values obtained for individual fatty acids for ruminant products show
369 ranges more compatible with the values obtained from locally sourced *Kashk* compared to the
370 reference obtained from animals raised on a C_3 diet in the UK. The $\delta^{13}\text{C}$ values on modern dairy
371 products confirm that the use of the universal proxy ($\Delta^{13}\text{C}$ values; Evershed et al., 1999, 2008) is
372 better suited to qualitatively identify the source of animal fats for the region. To perform
373 quantitative analyses and evaluate mixtures of commodities based on $\delta^{13}\text{C}$ values and mixing plots
374 (Mukherjee et al., 2007; Casanova et al., 2020b), recording more local references on conventional
375 dairy products (milk, butter), ruminant adipose fats but also non-ruminant adipose fats would be
376 needed. In Iran, in addition to ruminant animals, numerous Neolithic sites showed also the
377 exploitation of wild local animals such as hemiones or turtles (e.g. Mashkour et al. 2006) which were
378 part of the diet and could complement the modern reference database.

379 About 46% of the bones investigated here contained diagnostic lipids (cholesterol and fatty acids;
380 Figure 3). This suggests the palaeodietary information available from human and animal remains
381 using $\delta^{13}\text{C}$ values on cholesterol may be scarcer than for other regions worldwide that showed better
382 preservation (Evershed et al., 1995; Stott et al. 1996, 1999; Colonese et al. 2015). The dietary record
383 from bone lipids does not seem to provide as much information as the one that can be obtained
384 from collagen for the Iran region, as two sites showed collagen preservation in absence of lipid
385 preservation. Nonetheless, where present, cholesterol offers a complement to bone collagen for
386 palaeodietary reconstruction, as it reflects different macronutrients in the diet (Jim et al., 2004). The
387 sample size is also not representative enough to draw definitive conclusions.

388

389 **Implications for chronological studies**

390 In terms of chronology established using radiocarbon dating, studies involving the direct dating of
391 lipid residues preserved in pottery using the compound-specific radiocarbon analysis (CSRA)
392 approach usually require a lipid concentration of 500 μg per g of clay or more (Casanova et al.,
393 2020c). The maximum lipid concentration recovered in this study reached 510 $\mu\text{g}\cdot\text{g}^{-1}$ which is at the
394 lower limit of what is required to yield 200 μg of C for each FAs. At a concentration of 250 $\mu\text{g}\cdot\text{g}^{-1}$

395 about 10 g of pottery would be required for a ^{14}C date. This is usually the maximum amount of clay
396 we can sample from the same potsherd to minimise its destruction. This means smaller samples sizes
397 (< 200 μg) may need to be considered to radiocarbon date ceramics in the region, which would
398 involve some methodological adjustments to handle lower amount of carbon.

399 The possibility to directly date bone lipids was explored following the same approach as for pottery
400 lipid dating, which is targeting ~ 200 μg of C per single lipid (e.g. cholesterol, fatty acid) for ^{14}C dating.
401 The results obtained here are highly variable for the sites. At Qazānchi and Wezmeh 6 to 7 g of bones
402 would be required for a ^{14}C date, whereas at Kholah Kouh 8 to 18 g of bones are required and about
403 20 to 50 g of bones are required at Ālou and Shahrān. Lipid preservation in bones suggests that lipid
404 concentration above 100 μg per g of bone is required to minimise the amount of bone used for a
405 radiocarbon date (< 10 g). Only one bone (at Wezmeh) showed diagnostic lipids in appreciable
406 concentration to attempt dating bones in instances of low collagen preservation. This dataset
407 highlights the potential for lipids in bones as being an alternative to collagen for radiocarbon dating
408 in the absence of collagen preservation. Nonetheless, the number of specimens needing pre-
409 screening to find a bone with the potential to be dated seems rather high and may not be considered
410 a suitable alternative based on the extra amount of analysis required. More bone lipid analyses from
411 Iran and other surrounding regions need to be studied to determine the full potential and limits of
412 this alternative.

413

414 **Conclusions**

415 This study shows lipids are recovered in appreciable concentrations in ceramics despite the extreme
416 environmental conditions found in Iran. The $\delta^{13}\text{C}$ values from locally sourced modern dried dairy
417 products showed the Iran region to be influenced by aridity effects, and this should be taken in
418 consideration when interpreting an archaeological dataset. The lipids preserved in ceramics indicate
419 dairy products were already exploited during the late Neolithic period in Iran. Dietary reconstruction
420 and chronological studies involving the analysis of lipids preserved in ceramics have therefore good
421 potential for this area.

422 Lipid preservation in bones is very variable and the number of bones studied here does not allow us
423 to draw definitive conclusions on the conditions of lipid survival. The dataset suggests lipid
424 preservation could offer some alternatives for dietary and chronological studies in cases of poor
425 collagen preservation but would not be possible at all the sites from the region.

426 Overall, lipids from ceramics have a better potential for dietary and chronological study due to their
427 wider recovery in higher concentrations than bone lipids. Pottery vessels used to process food
428 accumulate lipids from their usage and can reach extremely rich lipid concentrations in the clay
429 matrix (e.g., Dunne et al., 2012). Lipid concentrations in archaeological bones are limited to their
430 natural abundances in bones, which do not exceed 50 $\mu\text{g}\cdot\text{g}^{-1}$ in the mineral phase and about 900 $\mu\text{g}\cdot\text{g}^{-1}$
431 1 in the bone marrow cavity (During et al., 2015).

432

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439

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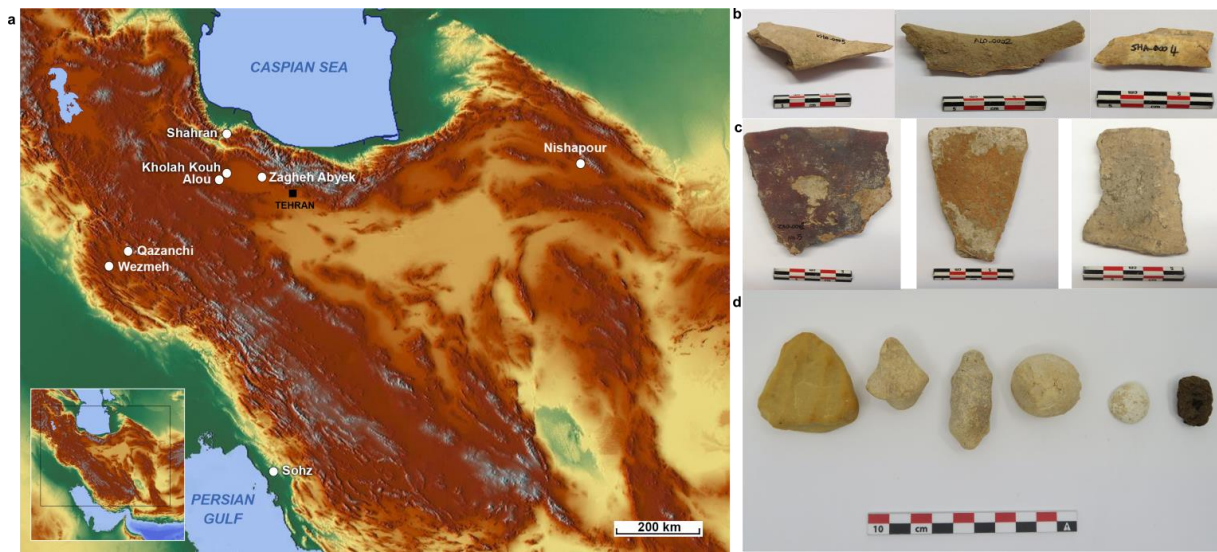
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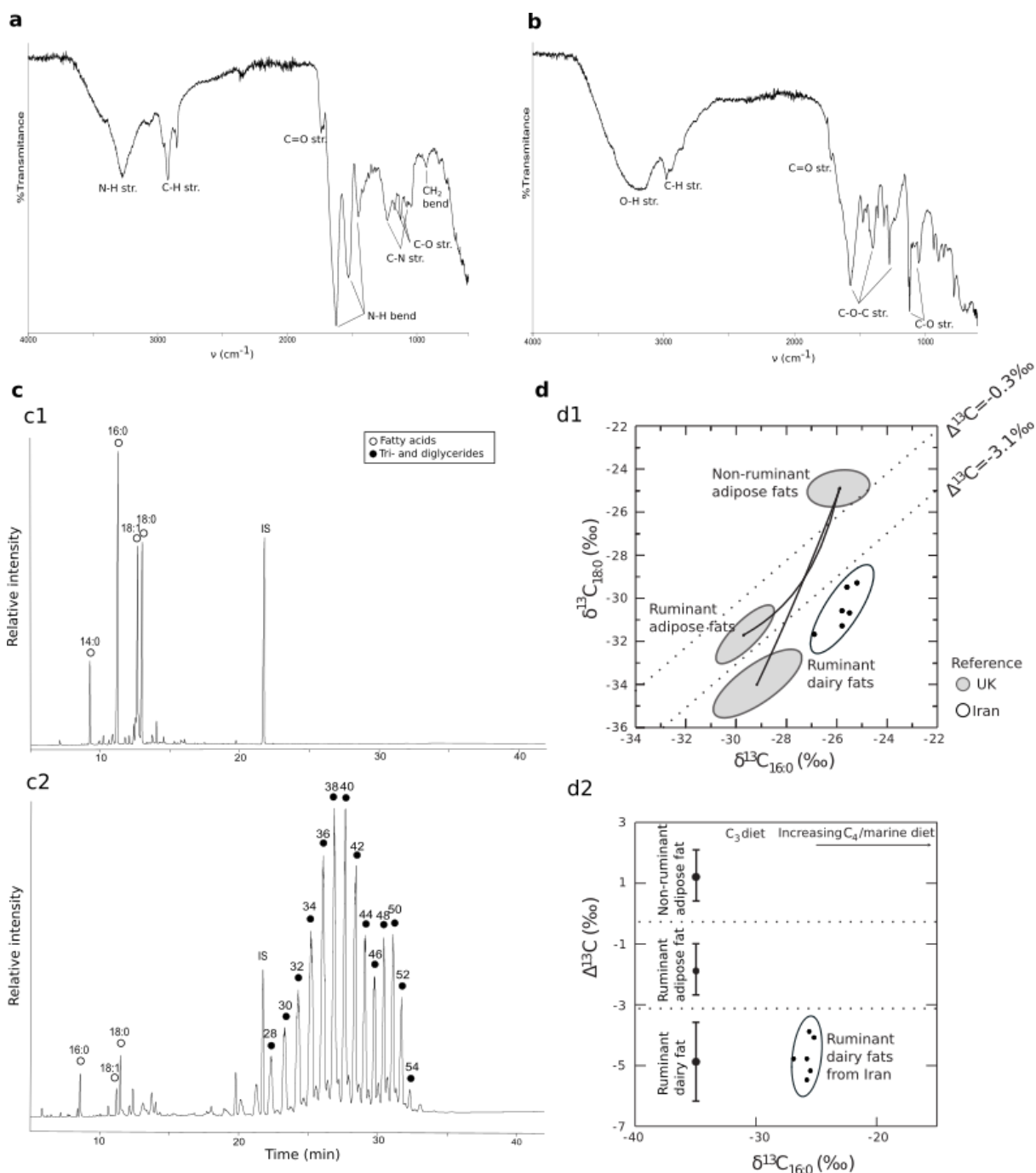
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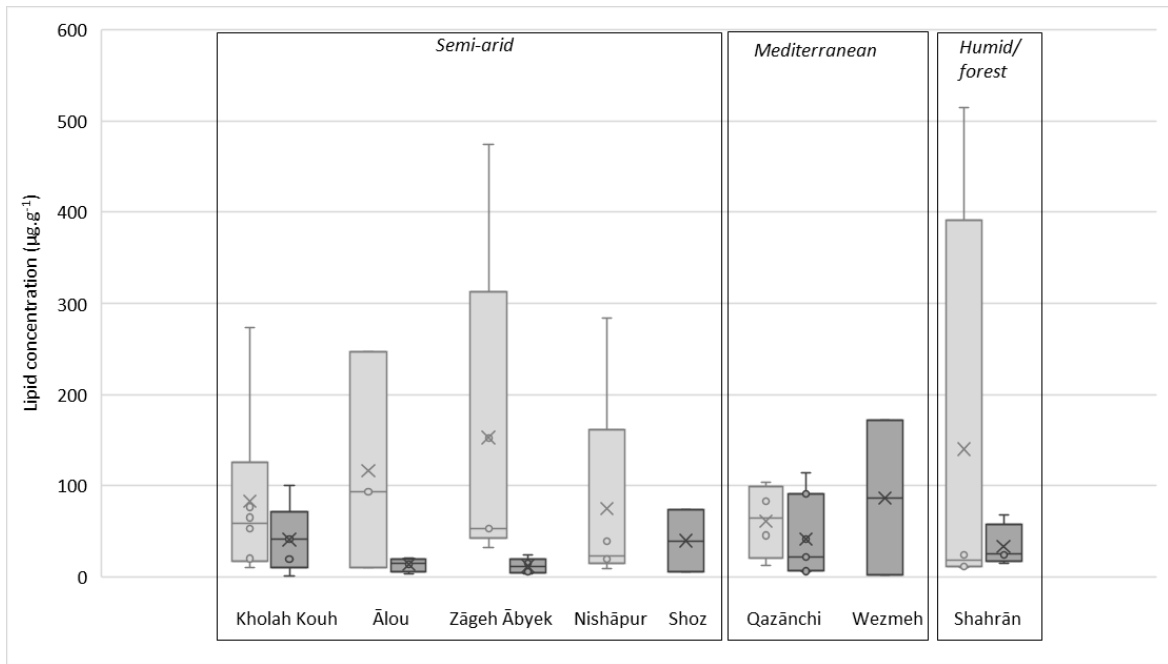
617

618 Figure 1: (a) Map of Iran with studied sites (b) selection of bones showing lipids in appreciable
619 concentration, (c) selection of analysed potsherds showing animal fat residues in appreciable
620 concentration, (d) *Kashk* (five cream/white dried paste on the left side) and *Qara qurut*, (one black
621 paste on the right side).

622

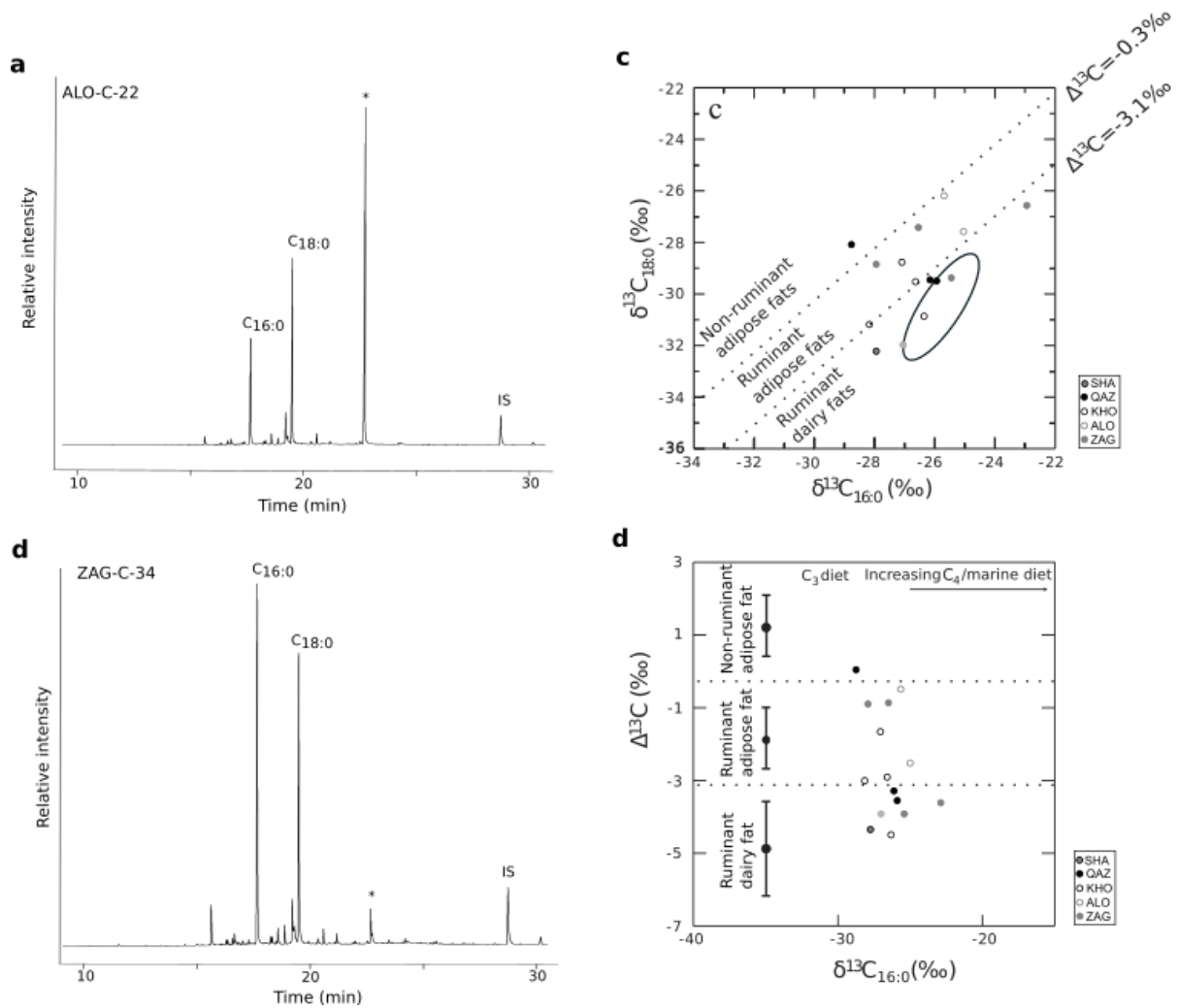


623
 624 Figure 2: **a, b.** FTIR spectra of kashk and qara qurut, respectively. **(c) c1.** Partial gas chromatogram of
 625 kashk (sample K-3) lipid extract using the methanolic/sulphuric acid extraction, **c2.** Partial gas
 626 chromatogram of the same kashk using the solvent extraction. **(d) d1.** $\delta^{13}\text{C}_{18:0}$ values plotted against
 627 the $\delta^{13}\text{C}_{16:0}$ values of fatty acids for the modern Iranian dairy products. Numbers represent the carbon
 628 chain length, IS the internal standard. Ellipses denote the 68% confidence intervals recorded on
 629 reference animal fed on a pure C_3 diet from the UK and the ones obtained on Iranian dairy products.
 630 **d2** $\Delta^{13}\text{C}$ ($= \delta^{13}\text{C}_{18:0} - \delta^{13}\text{C}_{16:0}$) values plotted against the $\delta^{13}\text{C}_{16:0}$ for the same modern dairy products.
 631



632

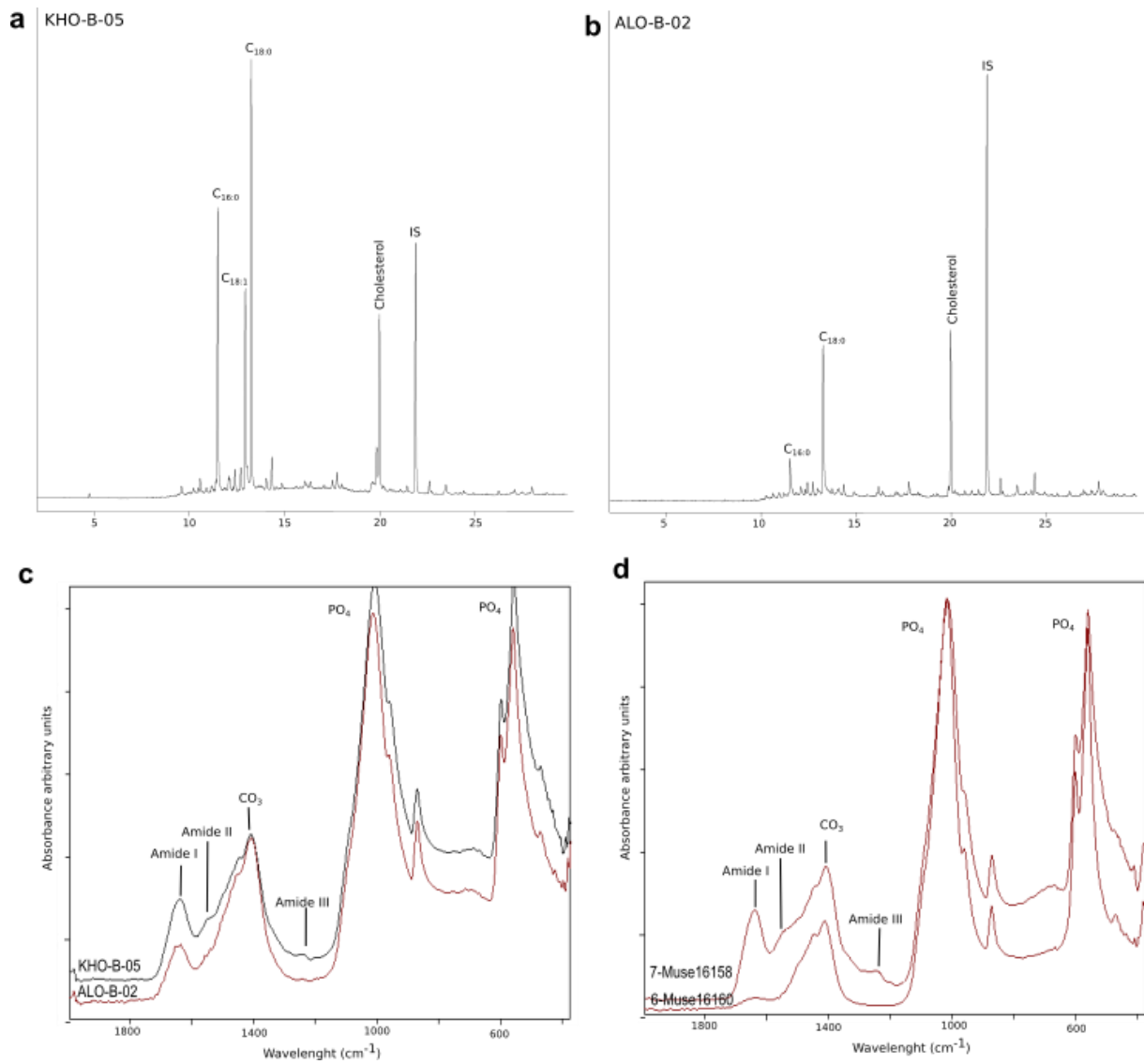
633 Figure 3: Box and Whisker plot of lipid concentrations for archaeological pottery (in light grey) and
 634 bones (in dark grey) for each site and environment.



635

636 Figure 4: Partial gas chromatograms of TLEs from pottery vessels from the site of (a) Ālou and (b)
 637 Zāgheh Ābyek. *denotes modern plasticizer contamination. (c) $\delta^{13}\text{C}_{18:0}$ values plotted against the
 638 $\delta^{13}\text{C}_{16:0}$ values of fatty acids for archaeological potsherds containing animal fats. The ellipse denotes
 639 the 68% confidence intervals recorded on reference dairy products from Iran. (d) $\Delta^{13}\text{C}$ (= $\delta^{13}\text{C}_{18:0}$ -
 640 $\delta^{13}\text{C}_{16:0}$) values plotted against the $\delta^{13}\text{C}_{16:0}$ for the same archaeological potsherds containing animal
 641 fats. Ranges denotes reference values for animal products across the world (Copley et al., 2003). KHO
 642 = Kholah Kouh, SHA = Shahrān, QAZ = Qazānchi, ALO = Ālou, ZAG = Zāgheh Ābyek, NIS = Nishāpur.

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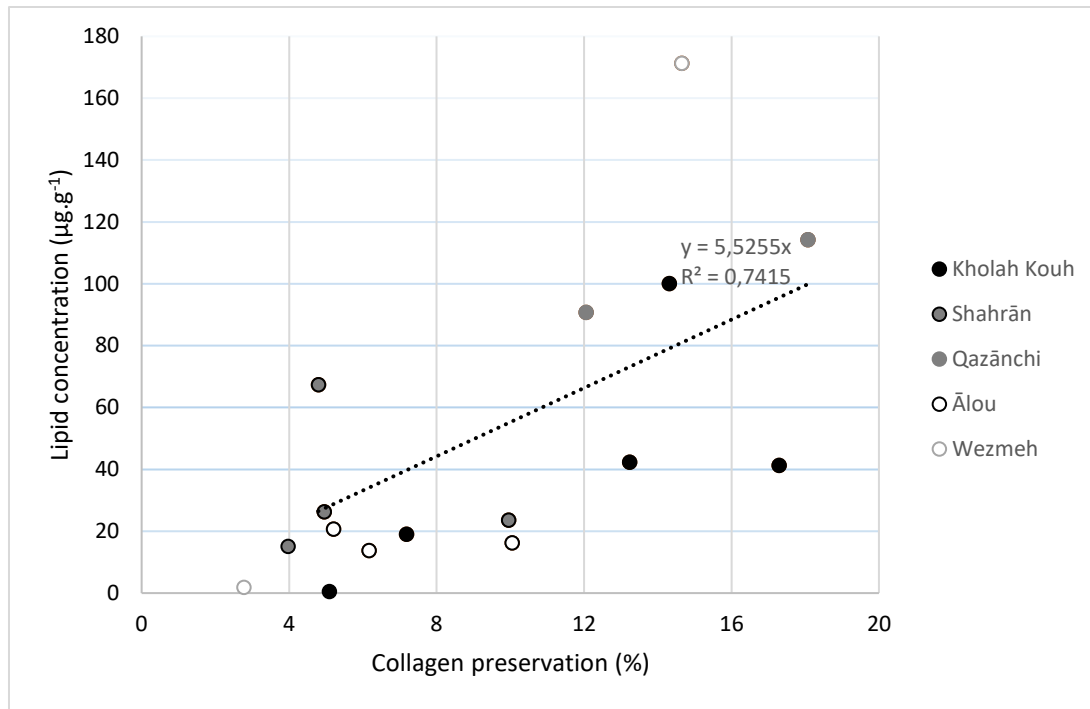
644

645 Figure 5: Partial gas chromatogram of lipid extracted from bones at (a) Kholah Kouh and (b) Alou. (c)
 646 FTIR spectra of the same bones, showing appreciable collagen preservation. (d) FTIR spectra of the
 647 two Wezmeh bones, showing poor collagen preservation for one (6-Muse16160) and appreciable
 648 collagen (7-Muse16158), although none could be extracted for ¹⁴C dating.

649

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651



652

653 Figure 6: Lipid concentration preserved in archaeological bones with diagnostic lipids identified
654 against the collagen preservation in the same bones. The dotted line is the linear regression line, y
655 the equation and R^2 the correlation coefficient.

656

657 Table 1: Archaeological sites studied, relative age, environment and number of material studied per
 658 site

Site name	Province	Relative age	Climate/ environment	Pottery	Bones
Kholah Kouh	Qazvin	Transition from Neolithic to Chalcolithic (5200-4600 BC)	semi-arid /steppe	Plain buff ware <i>n</i> =3 Painted buff ware <i>n</i> =3 Crusted buff ware <i>n</i> =5	Human <i>n</i> =1 Caprine <i>n</i> =2 Small ruminant <i>n</i> =1 Large mammal <i>n</i> =1
Shahrān	Gilan	Late Neolithic/Early Chalcolithic (5000-4000BC) Iron Age (800-550 BC)	Humid/forest	Plain red ware <i>n</i> =6	Human <i>n</i> =1 Small ruminant <i>n</i> =3
Qazānchi	Kermanshah	Pre-Pottery Neolithic (8600-8200 BC) Late Chalcolithic (4600-4200 BC) Middle Bronze Age (2600-2200 BC)	Mediterranean (subtropics)/ forest	Plain plum ware <i>n</i> =6	Human <i>n</i> =1 Caprine <i>n</i> =2 Small ruminant <i>n</i> =1 Large mammal <i>n</i> =2
Ālou	Qazvin	Late Chalcolithic (3700-3400 BC)	semi-arid /steppe	Plain plum ware <i>n</i> =1 Plain buff ware <i>n</i> =5 Plain red ware <i>n</i> =1	Caprine <i>n</i> =2 Cattle <i>n</i> =2
Zāgheh Ābyek	Qazvin	Early Chalcolithic (4300-3900 BC) Transition from Neolithic to Chalcolithic (5000-4300 BC)	semi-arid /steppe	Plain plum ware <i>n</i> =3 Plain buff ware <i>n</i> =3 Crusted buff ware <i>n</i> =3	Caprine <i>n</i> =4 Small ruminant <i>n</i> =1
Tepe Sohz	Behbahan plain	Early Chalcolithic (5000-4000 BC)	Semi-arid	-	Caprine <i>n</i> =2
Wezmeh	Kermanshah	Late Neolithic/Chalcolithic	Mediterranean/ continental	-	Cattle <i>n</i> =1 Deer <i>n</i> =1
Nishāpur	Khorasan Razavi	Sassanian-Islamic period (since 4th century CE)	semi-arid /steppe	<i>Fuqā'a</i> <i>n</i> =3 Plain buff ware <i>n</i> =2	-

659

660 Table 2: Lipid concentration and stable carbon isotopes values on individual fatty acids (corrected for
 661 the Suess effect) from *Kashk* and *Qara qurut*

Sample	Solvent extraction		Acid extraction (mean of 3 replicates)				
	Lipid concentration ($\mu\text{g}\cdot\text{mg}^{-1}$)	% lipids	Lipid concentration ($\mu\text{g}\cdot\text{mg}^{-1}$)	% lipids	$\delta^{13}\text{C}_{16:0}$ (‰)	$\delta^{13}\text{C}_{18:0}$ (‰)	$\Delta^{13}\text{C}$ (‰)
Quara Qurut	5.3	0.5	9.0	0.9	-25.8	-31.3	-5.5
Kashk 1	189.8	19.0	76.9	7.7	-25.2	-29.3	-4.1
Kashk 2	148.4	14.8	69.3	6.9	-26.9	-31.7	-4.8
Kashk 3	101.3	10.1	59.3	5.9	-25.6	-29.5	-3.9
Kashk 4	13.1	1.3	9.0	0.9	-25.8	-30.6	-4.8
Kashk 5	37.6	3.8	42.0	4.2	-25.5	-30.7	-5.2

662

663

664

665 **Supplementary Information**

666 Lipid preservation in ceramics and bones from the Iranian Plateau: implications for dietary and
667 chronological reconstructions

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684

685 **Site descriptions**

686

687 **Shahrān**

688 The archaeological site of Shahrān is located in the Sefid-Rud River Valley, in the Rudbar
689 County, Gilan province. The salvage excavation of site was conducted by a team under direction
690 of Shahram Ramin in the winter 2019 on behalf of Archaeological Centre, Cultural Heritage,
691 Tourism and Handicrafts office of Gilan. The site lies on a foothill overlooking the Sefid-Rud
692 River. It was first occupied by Late Neolithic/Early Chalcolithic communities (ca. 5000-4000
693 BC) and then used as a cemetery during the Iron Age. Due to the lack of Early Village (Late
694 Neolithic/Early Chalcolithic) sites in northern part of Iran (only two excavated sites so far) it is
695 important to document human occupations from this period in the region. It is noteworthy that
696 no absolute dating is available from this period in the region.

697

698 **Kholah Kouh, Ālou and Zāgheh Ābyek**

699 The two ancient sites, Kholah Kouh and Ālou, are located near Tākestān, and Zāgheh Ābyek
700 near Ābyek in the Qazvin province (Davoudi et al. 2019a and b, Davoudi et al. 2021, Moghimi
701 and Davoudi 2020). Excavation of the three sites were conducted by Hossein Davoudi on behalf
702 of Archaeological Centre, Cultural Heritage, Tourism and Handicrafts office of Qazvin
703 (ICHTO).

704 Kholāh Kouh is located in the city of Tākestān. A salvage excavation on the site was conducted
705 in the summer 2014. The mound site is about 3.5 ha. in area. According to pottery typology and
706 other artefacts the site is dated to the transition period from the Neolithic to Chalcolithic period,

707 ca. 5200 to 4600 BC. The evidence of residential context, human burial, pottery, spindle whorls,
708 copper object and other atefacts were found in 4 meters of cultural deposits.

709 Ālou is located in the Rādekan village in Tākestān County and was excavated in the summer
710 2016. The site is an open-air site, of about 1 ha. In area, without visible mound contrary to
711 Kholāh Kouh. The site is dated relatively to the Late Chalcolithic period in 3700-3400 BC
712 according to pottery typology and administrative technological devices that were discovered at
713 the site, and are known for this period and for the understanding of the origins of socioeconomic
714 developments in the Central Plateau of Iran.

715 The site of Zāgheh Ābyek is located in the Zāgheh village in Ābyek County. The site was
716 sounded in the winter 2018. The site is a mound (Tappeh) of about 14 ha. area. The site was
717 occupied during the transitionnal period from Neolithic to Chalcolithic (5200-4600 BC) and
718 Early Chalcolithic (4300-4000 BC) periods. The overlap of Transitional and Early Chalcolithic
719 deposits was discovered only in two sites in the Central Plateau of Iran. Therefore, this site has
720 a potential for estimating the time span of transition between two mentioned periods.

721

722 **Qāzānchi**

723 Qāzānchi, is located to the northwest of Kermanshah city at 1323 masl. It lies close to the
724 Razavar river. The earliest aerial picture of the site was taken in 1965 when the mound (Tappeh)
725 was complete and of circular shape. The site, especially of the north and east zones, has then
726 been gradually damaged by human and mechanized agricultural activities. As a result,
727 approximately one hundred meters of the northern section has been exposed, and found to
728 contain cultural sequences from the Holocene. The site was surveyed during the winter 2019 to
729 identify the cultural sequences, site formation and settlement patterns present. The extent of the
730 core area was estimated to be ca. two hectares, following results from test trenches excavated
731 to define the core area and buffer zone. The first results from the stratigraphy and the relative
732 and absolute chronologies indicate that the upper levels of the site belong to the Middle Bronze
733 Age and the Late Chalcolithic periods, and the lower levels contain a Pre-Pottery Neolithic
734 sequence. The results of archaeobotanical, archaeozoological, soil micromorphological and
735 palaeoclimatological studies point to a localised wet environment during the Holocene with
736 periodically flooded zones around the site. The pottery found at Qāzānchi is comparable to the
737 Middle Bronze Age of Chogha Maran (upper phase) and Godin Tepe (Period III), as well as the
738 Late Chalcolithic of Siahbid (middle and late phases), Chogha Maran and Godin Tepe (Period
739 IX), that are located in the same cultural range in Central Zagros. There are also evidence of
740 the Late Neolithic period occupation in the one of test trenches in the western slope of the
741 mound. The Pre-Pottery Neolithic phases at Qazānchi are contemporaneous with Sheikh-e
742 Abad, East Chia Sabz, Kelek Asad Morad and Chogha Golan which belong to the first early
743 Holocene settlements in the east of the Fertile Crescent. The potsherds analysed in this study
744 are from the Late Neolithic and the Late Chalcolithic contexts.

745

746 **Tepe Sohz**

747 The archaeological site of Tepe Sohz is located in the Behbahan Plain in southwestern Iran. In
748 1970 Hans Nissen began an archaeological project in the region, consisting of surveys of the
749 Behbahan and Zohreh Plains conducted in the spring. In the fall of 1970 excavations were
750 conducted at Tepe Sohz, the largest prehistoric site in the region (approximately 13 ha).

751 Nissen's plan was to study the economic relationships and interactions among settlements in
752 the region; examine the mechanisms of ancient trade and investigate intra-site variation in
753 architecture, occupations and food consumption as indicators of social status (Nissen 1970;
754 Nissen 1976). Stylistic comparisons of the Tepe Sohz painted pottery with Bakun ceramics
755 known from the highlands of southwestern Iran and the Susiana pottery tradition characteristic
756 of the lowlands reveal similarities with both regions, but also motifs and structures that appear
757 to be of local inspiration.
758

759 **Wezmeh Cave**

760 Wezmeh Cave is located 46 km southwest of Kermanshah, southeast of Islamabad-e Gharb
761 city, in the Qaziwand Mountain. Several investigations from 2001 have contributed to the
762 discovery of a large number of Late Pleistocene faunal remains, a Neanderthal premolar tooth,
763 Middle Paleolithic lithic artefacts, as well as Early Holocene human remains and cultural
764 materials left by transhumant pastoralists of the Neolithic and Chalcolithic periods (Abdi et al.,
765 2002; Trinkaus et al., 2008, Mashkour et al 2009, Broushaki et al 2016, Zanolli et al. 2019,
766 Biglari et al 2021). Wezmeh cave yielded rich data that can shed new light on Pleistocene and
767 Holocene human occupations, and regional fauna in this part of the west-central Zagros. The
768 site is also remarkable for the abundant carnivore remains that accumulated from 70Ky to pre-
769 modern times while the cave was used as a den for cave lions, bears, spotted hyenas and foxes
770 among other carnivores.

771 **Nishāpur, Khorāsan, Iran**

772 Nishāpur as one of the main urban centres of Khorasan, Northeast Iran was founded in the
773 Sassanian period, and never abandoned until the present time. Today, the evidence of this
774 longevity remains over a vast area corresponding to the urban landscape of Nishāpur. At its
775 zenith (From the 10th to 12th century CE) a wide range of food and non-food agricultural
776 commodities were produced in Nishāpur, and processed foods were an integral part of the
777 Nishāpur economy (Lorzadeh and Laleh, 2018). Since 2009, a long-term archaeological project,
778 set up by the University of Tehran, aimed to establish the chronology of the city as well as the
779 genesis and development of the urban landscape through time (Laleh et al. 2012; Laleh et al.
780 2015). The first season of excavations in 2018 resulted in the discovery of a wide variety of
781 artefacts including more than 8500 pottery sherds from 15 trenches (Laleh and Lorzadeh 2019).
782 The pottery samples analyzed in the present study are discovered from the central core of the
783 city, i.e. two from the citadel, two from the residential area south of the citadel, and one from
784 the so-called mosque area. They are all related to preparing, processing and/or storing of food
785 materials and can be dated to the 11th and first half of the 12th centuries CE.

786

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842 reassessment of the Wezmeh 1 maxillary premolar, *Journal of Human Evolution*, 135 M. 102643, ISSN 0047-
843 2484,

844

845 Table 3: Summary of pottery vessels (uncontaminated) with appreciable lipid preservation, their lipid
 846 concentrations, stable carbon isotope values and source assignment. FA = Fatty acids, OH= *n*-
 847 alkanols, A = *n*-alkanes.

Site name	Sample number	Concentration ($\mu\text{g}\cdot\text{g}^{-1}$)	Lipid detected	$\delta^{13}\text{C}_{16:0}$ (‰)	$\delta^{13}\text{C}_{18:0}$ (‰)	$\Delta^{13}\text{C}$ (‰)	Assignment
Kholah Kouh	KHO-C-0001	10	C _{14:0} , C _{16:0} -C _{18:0} FA	-26.6	-29.5	-3.0	Ruminant adipose fats
	KHO-C-0003	20	-	-	-	-	<i>nd</i>
	KHO-C-0004	76	C _{14:0} , C _{16:0} -C _{18:0} FA	-26.3	-30.9	-4.6	Ruminant dairy fats
	KHO-C-0005	53	C _{14:0} , C _{16:0} -C _{18:0} FA	-27.0	-28.8	-1.7	Ruminant adipose fats
	KHO-C-0008	65	-	-	-	-	<i>nd</i>
	KHO-C-0009	273	C _{14:0} , C _{16:0} -C _{18:0} FA	-28.1	-31.2	-3.1	Ruminant adipose fats
Shahrān	SHA-C-0011	6	-	-	-	-	<i>nd</i>
	SHA-C-0012	11	-	-	-	-	<i>nd</i>
	SHA-C-0013	24	-	-	-	-	<i>nd</i>
	SHA-C-0015	514	C _{14:0} -C _{18:0} FA	-27.8	-32.2	-4.4	Ruminant dairy fats
Qazānchi	QAZ-C-0016	83	C _{14:0} -C _{18:0} FA	-26.1	-29.5	-3.4	Ruminant dairy fats
	QAZ-C-0017	104	C _{14:0} -C _{18:0} FA	-25.9	-29.5	-3.6	Ruminant dairy fats
	QAZ-C-0018	12	-	-	-	-	<i>nd</i>
	QAZ-C-0020	45	C _{14:0} -C _{18:0} FA	-28.7	-28.1	0.7	Non-ruminant/aquatic fats
Ālou	ALO-C-0022	246	C _{14:0} -C _{18:0} FA	-25.6	-26.2	-0.5	Ruminant adipose fats
	ALO-C-0026	93	C _{14:0} , C _{16:0} -C _{18:0} FA	-25.0	-27.6	-2.6	Ruminant adipose fats
	ALO-C-0027	10	-	-	-	-	<i>nd</i>
Zāgheh Abyek	ZAG-C-0029	152	C _{14:0} , C _{16:0} -C _{18:0} FA	-27.9	-28.9	-1.0	Ruminant adipose fats
	ZAG-C-0030	53	C _{14:0} , C _{16:0} -C _{18:0} FA	-25.4	-29.4	-4.0	Ruminant dairy fats
	ZAG-C-0034	474	C _{14:0} , C _{16:0} -C _{18:0} FA	-22.9	-26.6	-3.7	Ruminant dairy fats
	ZAG-C-0035	32	-	-	-	-	<i>nd</i>
	ZAG-C-0036	53	C _{14:0} -C _{18:0} FA	-26.5	-27.4	-0.9	Ruminant adipose fats
Nishāpur	NIS-C-0037	39	C _{14:0} -C _{18:0} FA	<i>nd</i>	<i>nd</i>	<i>nd</i>	Animal fats
	NIS-C-0038	23	-	-	-	-	<i>nd</i>
	NIS-C-0039	19	-	-	-	-	<i>nd</i>
	NIS-C-0040	9	-	-	-	-	<i>nd</i>
	NIS-C-0041	284	C _{14:0} -C _{18:0} FA, C ₁₄ -C ₂₀ OH, C ₂₃ -C ₂₈ A	-	-	-	Plant oil

848

849

850 Table 4: Summary collagen (prediction) and lipid preservation in archaeological bones. The mass
 851 required for ¹⁴C lipid dating was calculated based on the peak area of the main compound. D =
 852 datable, ND = non-datable, FA = fatty acids, C = cholesterol, *nd* = non-determined, * = non-diagnostic
 853 lipids likely soil contamination

Site name	Sample number	Collagen preservation (%)	Datable by ¹⁴ C on collagen	Lipid concentration (µg.g ⁻¹)	Lipids identification	Mass required for ¹⁴ C lipid dating (g)
Kholah Kouh	KHO-B-0001	7.18	D	19	C	35
	KHO-B-0002	17.29	D	41	C ₁₆ -C ₁₈ FA	14
	KHO-B-0003	13.23	D	42	C	19
	KHO-B-0004	5.09	D	0	-	-
	KHO-B-0005	14.31	D	100	C ₁₆ -C ₁₈ FA, C	9
Shahrān	SHA-B-0006	4.79	D	67	C	26
	SHA-B-0007	4.95	D	26	C ₁₆ -C ₁₈ FA, C	50
	SHA-B-0008	3.97	D	15	<i>nd</i>	-
	SHA-B-0009	9.95	D	24	C	32
Qazānchi	QAZ-B-0010	7.47	D	6	<i>nd</i>	-
	QAZ-B-0011	18.07	D	114	C ₁₆ -C ₁₈ FA, C	7
	QAZ-B-0012	12.05	D	91	C	6
	QAZ-B-0013	2.99	ND	5	-	-
	QAZ-B-0014	3.36	ND	7	<i>nd</i>	-
	QAZ-B-0015	3.49	ND	41	<i>nd</i>	-
	QAZ-B-0016	2.71	ND	22	<i>nd</i>	-
Ālou	ALO-B-0017	4.8	D	3	-	-
	ALO-B-0018	10.05	D	16	C ₁₆ -C ₁₈ FA, C	40
	ALO-B-0019	6.17	D	14	C	41
	ALO-B-0020	5.2	D	21	C ₁₆ -C ₁₈ FA	19
Zāgheh Abyek	ZAG-B-0021	9.13	D	11	<i>nd</i>	-
	ZAG-B-0022	7.22	D	15	<i>nd</i>	-
	ZAG-B-0023	5.96	D	6	<i>nd</i>	-
	ZAG-B-0024	8.74	D	24	<i>nd</i>	-
	ZAG-B-0025	5.87	D	3	-	-
Tepe Sohz	2-Muse234	3.31	ND	74	<i>nd</i>	-
	3-Muse228	3.78	ND	5	-	-
Wezmeh	6-Muse16158	2.77	ND	2	-	-
	7-Muse16160	14.65	D	171	C ₁₆ -C ₁₈ FA, C	6

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