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1 **Calcium isotopes offer clues on resource partitioning among Cretaceous**
2 **predatory dinosaurs**

3

4 A. Hassler¹, J.E. Martin^{1*}, R. Amiot¹, T. Tacail¹, F. Arnaud Godet¹, R. Allain², V. Balter¹

5 ¹Univ. Lyon, ENS de Lyon, Université Claude Bernard Lyon 1, CNRS, UMR 5276 Laboratoire
6 de Géologie de Lyon: Terre, Planètes, Environnement, F-69342 46 Allée d'Italie, Lyon, France.

7 ²Sorbonne Universités – CR2P – MNHN, CNRS, UPMC-Paris 6, Muséum national d'Histoire
8 naturelle, 57 rue Cuvier, CP 38, 75231 Paris cedex 05, France.

9 *Correspondence to: jeremy.martin@ens-lyon.fr

10

11 **Large predators are overabundant in mid-Cretaceous continental dinosaur assemblages of**
12 **North Africa. Such unbalanced ecosystem structure involves, among predatory dinosaurs,**
13 **typical abelisaurid or carcharodontosaurid theropods co-occurring with long-snouted**
14 **spinosaurids of debated ecology. Here, we report calcium isotope values from tooth enamel**
15 **(expressed as $\delta^{44/42}\text{Ca}$) to investigate resource partitioning in mid-Cretaceous assemblages**
16 **from Niger (Gadoufaoua) and Morocco (Kem-Kem Beds). In both assemblages,**
17 **spinosaurids display a distinct isotopic signature, the most negative in our dataset. This**
18 **distinct taxonomic clustering in calcium isotope values observed between spinosaurids and**
19 **other predators provides unambiguous evidence for niche partitioning at the top of the**
20 **trophic chains: spinosaurids foraged on aquatic environments while abelisaurid and**
21 **carcharodontosaurid theropods relied almost exclusively on terrestrial resources.**

22

23 **Keywords: Ecology; Cretaceous terrestrial ecosystems; dinosaurs; calcium isotopes;**
24 **spinosaurs; palaeodiet**

25

26 **1.INTRODUCTION**

27 Dietary inference in extinct vertebrates allows addressing the structure of extinct food webs and
28 remains critical for understanding how ecosystems were structured, and how these structures
29 evolved with time and environmental changes. In modern ecosystems, the trophic cascades
30 concept [1] illustrates the complex role of top carnivores in regulating, directly and indirectly,
31 populations from subsequent trophic levels. By modulating the intensity of resource exploitation
32 (top-down), along with productivity feedback loops (bottom-up), top predators can shape the

33 structure of ecosystems. However, reconstructing long extinct ecosystems, such as those of the
34 Mesozoic, remains challenging because taxa with unknown ecology and physiology dominated
35 the scene.

36 In contrast to modern mammalian continental ecosystems where herbivores represent
37 most of the biomass of vertebrates, large predatory theropod dinosaurs were overabundant in
38 mid-Cretaceous continental biotas of North Africa. The origin and the significance of this
39 overabundance is debated [2,3] but recent studies have highlighted the peculiar nature of such
40 predator-dominated ecosystems [4,5]. These faunas include very large theropods such as
41 abelisaurids, carcharodontosaurids, and spinosaurids, which are all considered as having high
42 food requirements due to their elevated metabolic rates [6,7]. Their high abundance relative to
43 herbivorous dinosaurs thus raises questions about how resources could be partitioned between
44 them. Due to their remarkable anatomy, spinosaurids have attracted attention with previous work
45 inferring a semi-aquatic lifestyle [7,8] associated to a diverse diet [4,5,9–18], although the
46 respective proportions of feeding sources, whether terrestrial or aquatic, remains unknown.
47 Indeed, if for some spinosaurids, trophic links can be assessed using direct evidence like stomach
48 content and feeding marks [10,16], such fossils remain rare and account for a short time window.
49 Morphology can also provide a wealth of data concerning potential trophic links, but
50 interpretations remain hypothetical due to the lack of modern analogues for spinosaurids and
51 their ecosystem. The jaw morphology of spinosaurids has been repeatedly, albeit inexactly,
52 considered analogous to that of crocodiles [14,15] and has recently been compared to that of pike
53 conger eels [18], reflecting some uncertainty in palaeoecological inference when considering
54 morphological evidence alone [19]. In this context, we investigate diet and resource partitioning
55 among mid-Cretaceous Saharan vertebrates using Ca isotopes ($\delta^{44/42}\text{Ca}$) of tooth enamel and

56 scale ganoine, a method that has previously proved informative both in modern [20–22] and
57 fossil vertebrate assemblages [22–25]. Because Ca is almost exclusively derived from food
58 among terrestrial vertebrates [20], the systematic change between the isotopic signature of their
59 food relative to their mineralized tissues allows inferring trophic levels and diet. Moreover, Ca is
60 the major constituent of the mineral phase of bone and tooth enamel, i.e. hydroxylapatite
61 ($\text{Ca}_{10}(\text{PO}_4)_6\text{OH}_2$), which gives the method two main advantages. First, the diagenetic
62 overprinting of biogenic Ca is unlikely to occur providing that secondary carbonates are absent
63 or have been leached accordingly [23]. Second, minute amounts of bone or tooth enamel sample
64 (~100 μg) is sufficient for a routine measurement of the Ca isotopic ratios. The technique is thus
65 applicable to precious fossils with minimal damages.

66 Our study is focused on fossils from the Aptian-Albian fluvial deposits of Gadoufaoua
67 (Niger, ~113 Ma ago) [26] and of the Cenomanian Kem Kem beds (Morocco, ~100 Ma ago)
68 [27]. In these vertebrate assemblages, crocodylomorphs, spinosaurids and non-spinosaurid
69 theropods are overabundant relative to herbivorous dinosaurs [11,26,27], making them typical
70 examples of North African mid-Cretaceous ecosystems. Despite age differences, the two
71 deposits share similarities in their environmental context and host a comparable diversity of
72 vertebrates. The analyzed specimens include fish, crocodylomorphs, pterosaurs, herbivorous
73 (sauropods, ornithomorphs) and predatory (theropod) dinosaurs, including spinosaurids. Fossils of
74 the spinosaurids *Suchomimus tenerensis* and *Cristatusaurus lapparenti* [11,12] are found in
75 Gadoufaoua, where predators such as the non-spinosaurid theropods *Kryptops palaios* and
76 *Eocarcharia dinops* [28] occur. In the Kem Kem beds, the spinosaurids *Spinosaurus aegyptiacus*
77 and *Sigilmassasaurus brevicollis* [7,13,29] are associated with fossils of non-spinosaurid
78 theropods such as *Carcharodontosaurus saharicus* [27].

79

80 2. METHODS

81 (a) *Sample collection*

82 Samples comprising 73 tooth enamel, enameloid and fish scale ganoine from the early to
83 middle Cretaceous of Morocco and Niger have been analyzed for elemental concentration and
84 calcium isotope composition. Niger samples, totaling 7 taxa, come from the locality of
85 Gadoufaoua belonging to the Aptian/Albian Elrhaz Formation [26]. They consist of 8
86 spinosaurid theropod teeth (*Suchomimus tenerensis* or *Cristatusaurus lapparenti*), 9 non-
87 spinosaurid theropod teeth (cf. *Kryptops palaios* and cf. *Eocarcharia dinops*), 2 teeth of the
88 sauropod *Nigersaurus taqueti*, 9 teeth of the ornithopod *Ouranosaurus nigeriensis*, 8 teeth of the
89 pholidosaurid crocodylomorph *Sarcosuchus imperator*, 3 teeth tentatively referred to the
90 notosuchian crocodylomorph cf. *Araripesuchus* sp., 3 teeth tentatively referred to an
91 indeterminate large pterosaur, 5 teeth referred to an indeterminate pycnodont fish, 2 scales of an
92 indeterminate lepisosteiform fish, and 1 scale attributed to the lepisosteiform fish *Pliodetes* sp..

93 Moroccan samples previously analyzed for their apatite oxygen and carbon isotope
94 compositions [7] come from three localities (Bou Laâlou, Jebel Al Qabla, and Takemout) from
95 the early Cenomanian Ifezouane Formation of the Kem Kem Beds [27]. They consist of 7 teeth
96 attributed to non-spinosaurid theropods (4 teeth of *Carcharodontosaurus saharicus*, and 3 teeth
97 of an indeterminate large theropod), 8 teeth referred to the spinosaurid theropods *Spinosaurus*
98 *aegyptiacus* or *Sigilmassasaurus brevicollis*, 3 teeth of an indeterminate crocodylomorph, 3 teeth
99 of an indeterminate large pterosaur, 1 scale of the amiiform fish *Stromerichthys* sp. and 1 tooth
100 plate of the lungfish *Neoceratodus africanus*.

101 Here we investigate dietary and ecological aspects using the calcium isotope composition of
102 tooth enamel, enameloid and scale ganoine ($\delta^{44/42}\text{Ca}$) rather than bone or dentine, which are
103 porous and incorporate secondary diagenetic minerals. These biological materials are compact
104 and resistant to diagenetic alteration [24]. Sampling was performed using a handled micro-drill
105 (8200 Dremel with tungsten steel solid carbide bit and diamond burr), a micro-drill station
106 (MicroMill with tungsten steel solid carbide bit and diamond burr), and a scalpel made in
107 stainless steel. Tooth enamel, enameloid and scale ganoine have been carefully separated from
108 dentine and sediment using these tools.

109

110 ***(b) Analytical techniques***

111 Fossil samples were placed in Teflon beakers and dissolved in 500 μL of ultrapure nitric
112 acid (15 N) at 130°C during 1 hour. Following this attack, samples were dried out, then dissolved
113 in a dilute solution of ultrapure nitric acid (0.5 N).

114 A fraction of the solutions was measured for elemental concentrations of trace elements
115 (Rare Earth Elements (REE), strontium (Sr) and barium (Ba)) and major elements (such as
116 calcium (Ca), magnesium (Mg) and phosphorus (P)). These measurements were performed on an
117 inductively coupled plasma mass spectrometer (7500 Series ICP-MS, Agilent Technologies) for
118 trace elements, and on an inductively coupled plasma atomic emission spectrometer (ICAP 6000
119 Series ICP spectrometer, Thermo electron corporation) for major elements. The reliability of
120 measurements has been controlled through a set of blanks and standards (see measures NIST-
121 SRM1400, USGS-MAPS-4 and USGS-MAPS-5 in supplementary table).

122 Calcium from samples was chemically purified following previously described protocols
123 [30]. Briefly, samples were dissolved in double distilled HCl and loaded onto columns filled with

124 AG 50WX-12, separated from matrix including phosphates. Calcium, collected together with
125 strontium, is then further processed in HNO₃ medium using Eichrom Sr-specific resin, allowing
126 removal of Sr. The reliability of elutions have been controlled by processing and analyzing
127 standard materials (see measures of NIST-SRM1486 in the supplementary table).

128 Calcium isotope abundance ratios (⁴⁴Ca/⁴²Ca) were measured using a Neptune Plus MC-
129 ICP-MS (Multi-Collector Inductively Coupled Plasma Mass Spectrometer) following previously
130 described methods [30,31]. After purification, Ca samples were dissolved in ultrapure 0.05N
131 HNO₃ and Ca concentration was set at 2 mg.l⁻¹ for all samples and standards. All Ca isotope
132 compositions are expressed using the “delta” notation defined as follows:

133

$$134 \quad \delta^{44/42}\text{Ca} = ((^{44}\text{Ca}/^{42}\text{Ca})_{\text{sample}} / (^{44}\text{Ca}/^{42}\text{Ca})_{\text{ICP Ca Lyon}}) - 1) \times 1000$$

135

136 where (⁴⁴Ca/⁴²Ca)_{sample} and (⁴⁴Ca/⁴²Ca)_{ICP Ca Lyon} are the Ca isotope abundance ratios measured in
137 sample and ICP Ca Lyon reference standard respectively. The ICP Ca Lyon standard, used as a
138 bracketing standard, is a Specpure calcium plasma standard solution (Alfa Aesar) previously
139 described here: [22,23,30–32]. The SRM1486 cow bone meal standard (NIST) was used as
140 secondary standard, in order to assess accuracy of analytical procedure, including chemical
141 purification.

142

143 ***(c) Monitoring of the isotopic measurement accuracy***

144 Blanks realized during purification protocols have been analyzed with the MC-ICP-MS. All
145 blanks contained less than 85 ng of Ca, which is 470 times less than the Ca contained in samples.

146 Thus this nano-pollution does not alter the isotopic measurements of our samples, considering
147 our measurement precision.

148 The mass dependency curve of the isotopic measures follows the expected relation for Ca
149 (figure 1), with a slope value of 0.509 ± 0.016 (2SE, n=74) [22,23,25,30–32], supporting the
150 accuracy of the measures.

151 Measurements collected for the standard SRM1486 exhibit a $\delta^{44/42}\text{Ca}$ of $-1.00 \pm 0.10\%$ (2SD,
152 n=32), which is in the range of previously published data for this standard: $-0.96 \pm 0.14\%$ (2SD,
153 n=17) [30], $-1.04 \pm 0.11\%$ (2SD, n=25) [22], $-1.03 \pm 0.13\%$ (2SD, n=120) [31], $-1.03 \pm 0.12\%$
154 (2SD, n=147) [32].

155

156 ***(d) Diet modelling***

157 In order to perform diet inferences with Ca isotope data, we make use of our data to feed a
158 simple diet mixing model. This model provides estimates of the relative contribution of Ca
159 sources in the diet of predators thanks to $\delta^{44/42}\text{Ca}$. Two main parameter need to be constrained
160 prior to using it.

161 First, the isotopic signature of Ca sources needs to be constrained for studied animals.
162 Calcium is more concentrated in mineralized tissues (bone, enamel, scale ganoine) than in soft
163 tissues or in water. Thus, it has been shown that even a few percent of mineralized tissues in diet
164 was sufficient to constitute the major source of Ca for organisms [24]. There is fossil evidence of
165 bone and fish scale consumption among numerous taxa of crocodylomorphs, spinosaurid and
166 non-spinosaurid theropods [10,33]. Thus, even if this consumption can be restricted to occasional
167 events such as feeding on juveniles for bones, the high concentration of Ca in mineralized tissues
168 supports that these tissues were most likely the main source of Ca among these taxa. Moreover,

169 considering the heterogeneity of modern animal data on soft tissues [20,30,34], and that blood
170 can exhibit $\delta^{44/42}\text{Ca}$ close to bones [30], this supports the use of $\delta^{44/42}\text{Ca}$ values of mineralized
171 tissues as representative of the main source of Ca for predators in our model. We have collected
172 Ca isotope data for herbivorous dinosaurs and fishes from Gadoufaoua, which represent the most
173 likely prey of the analyzed apex predators (crocodylomorphs, spinosaurid and non-spinosaurid
174 theropods). Thus we have constrained the isotopic signature of the potential Ca sources to be
175 entered in the model.

176 Second, our model needs an estimate of the isotopic shift between Ca from food and teeth
177 of the predators. According to data collected on a variety of extant vertebrates (chicken, horse,
178 seal, deer, minipigs, sheep and mouse [20,30,34]), the mean isotopic shift between Ca from diet
179 to bones is $0.57 \pm 0.10\text{‰}$ (2SE, n = 21) and is relatively homogenous among these vertebrates.
180 Thus, considering that the studied predators are phylogenetically close (all are archosaurs) and
181 that all of them share comparable Ca-cycles [20], we can reasonably assume that they exhibited a
182 similar diet-bone isotopic offset. Only limited data have been collected on diet and enamel
183 couples in modern faunas. Some studies show differences between these two shifts ($\pm 0.2\text{‰}$; [22–
184 24,30,35]) but these studies are often conducted on mammals, whose teeth formed in early life,
185 and thus were not representative of the adult diet for which the comparison is made. However, in
186 human wisdom teeth, which developed along with an adult diet, the isotopic shift between
187 enamel and food is similar to the diet-bone shift of $-0.57 \pm 0.10\text{‰}$ (2SE, n = 21) [32]. Based on
188 these results, we assume a similar diet-enamel shift among studied taxa, and use this shift to
189 estimate the $\delta^{44/42}\text{Ca}$ value of the food of predators. We thus applied this model to *Sarcosuchus*
190 *imperator*, spinosaurid and non-spinosaurid theropods, in order to investigate the proportion of
191 fish and herbivorous dinosaurs in the diet of these predators.

192 We estimate the Ca source fractions using the estimation of food $\delta^{44/42}\text{Ca}$ between the first
193 and the third quartile of each population of predators, considering that it is more representative of
194 their general diet. Moreover, to take into account the variability of $\delta^{44/42}\text{Ca}$ values among
195 sources, these fractions are calculated as if predators with the most depleted $\delta^{44/42}\text{Ca}$ values fed
196 on fish and herbivorous dinosaurs with the most enriched $\delta^{44/42}\text{Ca}$ values, and vice versa. In this
197 way, we maximized the source fraction scattering, which sets the limit of diet inferences
198 achievable with our model.

199

200 **3. RESULTS**

201 *(a) Elemental concentrations*

202 The full dataset compiling all the elemental concentrations measured in this study are
203 reported in the supplementary table of this article.

204 Among important results, the mean calcium versus phosphorus ratio is of 2.3 ± 0.1 (1SD).

205

206 *(b) Calcium isotope ratios*

207 The full dataset compiling all the calcium isotopic ratios measured in this study are reported in
208 the supplementary table of this article. The $\delta^{44/42}\text{Ca}$ values of vertebrates from Gadoufaoua range

209 between -0.35 and -1.51‰ with a mean 2SD of 0.10‰ . In this locality, the sauropod

210 *Nigersaurus taqueti* is the taxon which exhibits the highest $\delta^{44/42}\text{Ca}$ with a mean value of -0.43

211 $\pm 0.11\text{‰}$ (2SD, n=2), whereas the spinosaurids exhibit the lowest values with $-1.30 \pm 0.15\text{‰}$

212 (2SD, n=8). Among the other dinosaurs, the ornithomimid *Ouranosaurus nigeriensis* exhibits

213 values at $-0.56 \pm 0.05\text{‰}$ (2SD, n=9), whereas non-spinosaurid theropods exhibit values at -0.94

214 $\pm 0.16\text{‰}$ (2SD, n=9). The small, supposedly terrestrial crocodylomorph, cf. *Araripesuchus* sp.,

215 exhibits a mean value of $-1.12 \pm 0.05\text{‰}$ (2SD, n=3), whereas for the semi-aquatic
216 crocodylomorph *Sarcosuchus imperator* this value is of $-1.12 \pm 0.13\text{‰}$ (2SD, n=8). For fishes
217 mean values are of $-0.74 \pm 0.02\text{‰}$ (2SD, n=2) for indeterminate Lepisosteiformes, of -0.67‰
218 (n=1) for *Pliodetes* sp., and of $-1.08 \pm 0.04\text{‰}$ (2SD, n=5) for Pycnodontiformes. Finally,
219 pterosaurs exhibit a mean value of $-0.78 \pm 0.05\text{‰}$ (2SD, n=3). The $\delta^{44/42}\text{Ca}$ of spinosaurid
220 theropods is significantly lower than that of non-spinosaurid theropods (Wilcoxon rank sum test:
221 p value = 9.9×10^{-4}).

222 The $\delta^{44/42}\text{Ca}$ values of Kem Kem beds vertebrates range between -0.41 and -1.56‰ with a
223 mean 2SD of 0.09‰ . In Kem Kem beds, the lungfish *Neoceratodus africanus* is the taxon which
224 exhibits the highest $\delta^{44/42}\text{Ca}$ with a value of -0.41‰ (n=1), whereas the other fish *Stromerichthys*
225 sp. exhibits a low value of -1.26‰ (n=1). Spinosaurids exhibit the lowest mean value of this
226 fauna, which is $-1.39 \pm 0.27\text{‰}$ (2SD, n=8). For large non-spinosaurid theropods
227 (*Carcharodontosaurus saharicus* and indeterminate theropods) this value is of $-0.64 \pm 0.13\text{‰}$
228 (2SD, n=7). Semi-aquatic indeterminate crocodylomorphs exhibit a mean value of $-0.66 \pm 0.09\text{‰}$
229 (2SD, n=3), and indeterminate pterosaurs from the family of Anhangueridae exhibit a mean
230 value of $-0.67 \pm 0.17\text{‰}$ (2SD, n=3). The $\delta^{44/42}\text{Ca}$ of spinosaurids is significantly lower than for
231 non-spinosaurid theropods (Wilcoxon rank sum test: p value = 3.1×10^{-4}).

232

233 **(c) Fractions of Ca sources in diet**

234 With our diet mixing model we estimate that the maximum Ca fraction from fish in the diet
235 reaches 30%, 58% and 100% for non-spinosaurid theropods, *Sarcosuchus imperator* and
236 spinosaurids, respectively. On the other hand, the maximum Ca fraction from herbivorous
237 dinosaurs in the diet reaches 100% for all of these predators. The maximum Ca fraction derived

238 from fish reaches 100% for 6 of the 8 studied spinosaurid individuals according to this model,
239 which is possible for only two individuals of *Sarcosuchus imperator* and one non-spinosaurid
240 theropod. However, the maximum Ca fraction derived from herbivorous dinosaurs is also 100%
241 for 4 of the 8 spinosaurid individuals.

242

243 **4. DISCUSSION**

244 *(a) Diagenesis*

245 Enamel, ganoine and to a lesser extent enameloid tissues, are highly mineralized and exhibit
246 a lower porosity than dentine or bone. They have a limited exchange surface with surrounding
247 fluids and sediment, and for this reason they are considered resistant to diagenesis [23,24,36].
248 Moreover, with almost 40% of their mass constituted of Ca, it is very unlikely that the Ca
249 isotopic signature of these tissues can be entirely overprinted during early diagenetic processes.
250 The Ca/P ratios measured for samples (supplementary table) exhibit values of 2.3 ± 0.1 (1SD),
251 which are close to those measured in modern animals [37]. This supports that Ca isotope ratios from
252 these fossils have not been extensively modified during diagenesis.

253 Quantifying elemental concentration of non-essential trace elements such as rare earth
254 elements (REE) and of major elements such as Ca and phosphorus, can provide information on
255 both the autochthony and diagenetic alteration [38,39]. Among fossils from Gadoufaoua, REE
256 enrichment profiles (normalized to the Post Archean Australian Shale, PAAS) exhibit a similar
257 shape with different REE enrichment intensity (figure 2A and supplementary table). They can
258 thus reasonably be considered as autochthonous [39]. Three teeth exhibit distinct REE profile
259 shapes (figure 2A). They have thus been identified as allochthonous fossils and have not been
260 considered in the palaeoecological discussion. The general REE profile at Gadoufaoua is “hat

261 shaped” according to the definition given by Reynard & Balter 2014 [38]. This shape, associated
262 with measured La/Yb and La/Sm ratios (supplementary table), differs from the REE profiles and
263 ratios expected after extensive recrystallization processes during diagenesis [38,40].

264 Among fossils from the Kem Kem beds REE enrichment profiles (normalized to the Post
265 Archean Australian Shale, PAAS) exhibit a distinct profile between the three studied localities
266 (figure 2B and supplementary table). However, within each locality, fossils exhibit similar REE
267 profiles. They can thus reasonably be considered as autochthonous [39]. As for Gadoufaoua, the
268 general “hat shaped” REE profile of the Kem Kem beds [38] and the measured La/Yb and La/Sm
269 ratios (supplementary table) are different from those expected for diagenetic recrystallization
270 processes [38,40].

271 Thus, the Ca/P ratios and the REE concentrations jointly support the hypothesis that the Ca
272 isotopic composition of the fossils have not been extensively affected by diagenesis. Moreover,
273 taxa from Gadoufaoua exhibit $\delta^{44/42}\text{Ca}$ values consistent with their putative ecology (figure 3A).
274 Indeed, herbivorous dinosaurs represented by *Nigersaurus taqueti* and *Ouranosaurus nigeriensis*
275 exhibit the highest $\delta^{44/42}\text{Ca}$ values of this dataset, with values ranging between $-0.35\text{‰} \pm 0.14$
276 (2SD, n=3) and $-0.66\text{‰} \pm 0.05$ (2SD, n=4), respectively. Their potential predators (the
277 pholidosaurid crocodylomorph *Sarcosuchus imperator*, spinosaurid and non-spinosaurid
278 theropods) exhibit lower $\delta^{44/42}\text{Ca}$ values, below -0.70‰ . This isotopic distribution between
279 herbivores and predators follows what has been observed in modern continental faunas [20], and
280 supports, along with the observations presented above, the biogenic nature of $\delta^{44/42}\text{Ca}$ values.

281

282 ***(b) Resource partitioning among predators***

283 Compared to non-spinosaurid theropods, spinosaurids exhibit distinctly lower $\delta^{44/42}\text{Ca}$ values
284 (Wilcoxon rank sum test: p-value = 9.9×10^{-4}), the lowest ones of the dataset (figure 3A). This
285 distinct pattern is also observed in the Kem Kem beds of Morocco (figure 3B) with spinosaurids
286 possessing significantly lower $\delta^{44/42}\text{Ca}$ values compared to non-spinosaurid theropods (Wilcoxon
287 rank sum test: p-value = 3.1×10^{-4}). Physiological homogeneity in Ca isotope fractionation from
288 food to tooth enamel in these theropods is supported both by their phylogenetic proximity, all are
289 archosaurs, and the observed homogeneity in modern vertebrates [20,30,34]. This pattern thus
290 highlights the distinctive ecological niche of spinosaurids relative to the other theropods.

291 The trophic pyramid in these fluvial palaeo-ecosystems could have been supported both
292 by herbivorous dinosaurs and fish [4,5,26,27], which would represent two potential primary
293 sources of Ca for predators. In Gadoufaoua, the Ca source constituted by herbivorous dinosaurs
294 exhibits a higher $\delta^{44/42}\text{Ca}$ value than fish, which allows discriminating potential dietary sources
295 among predators. Using the diet model described above (Method (d)) for the vertebrate
296 assemblage of Gadoufaoua, we observed that the estimated fractions of Ca originating from
297 herbivorous dinosaurs and fish in the diet of predators do overlap between predators. The
298 important range of fraction between the maximum and minimum fraction estimated for each
299 predator was expected considering the scattering of the $\delta^{44/42}\text{Ca}$ data from fish, herbivorous
300 dinosaurs and predators. However, even with this important scattering in isotopic values, the
301 difference between spinosaurid and non-spinosaurid theropods remains statistically significant,
302 implying distinct resource partitioning between these large predators. Here, $\delta^{44/42}\text{Ca}$ data support
303 the idea that a majority of non-spinosaurid theropods mainly fed on herbivorous dinosaurs, with
304 less than 30% of their Ca potentially stemming from fish sources (figure 4A). The giant
305 crocodylomorph *Sarcosuchus imperator* exhibits an intermediate diet, possibly composed of

306 both fish and herbivorous dinosaurs (figure 4B). Moreover, if the diet of spinosaurid theropods
307 potentially included a mix of fish and herbivorous dinosaurs according to the model, these data
308 also support that a majority of spinosaurids could be exclusively piscivorous (along with one
309 non-spinosaurid theropod and two *Sarcosuchus imperator* individuals), and that half of them
310 were at least partially piscivorous (figure 4C). Nevertheless, if this clearly illustrates that
311 spinosaurids were the predators of Gadoufaoua which had the most piscivorous-oriented diet,
312 this also leads to the conclusion that their diet was potentially a mix between fish and
313 herbivorous dinosaurs. Similarly, Kem Kem spinosaurids are also strongly depleted in heavy Ca,
314 confirming a piscivorous diet in another fossil assemblage. In previous studies, several diets have
315 been proposed for these groups of theropods. For spinosaurids, fish consumption inferred on the
316 basis of the ecosystem structure [4,5], morphology [9,11,13–15,17,18], or direct fossil evidences
317 [10] is thus supported by our dataset. The consumption of terrestrial preys, which is also
318 proposed in these papers, as well as the direct evidences related to this behavior [10,16], also
319 remain possible according to our data. In addition, the consumption of herbivorous dinosaurs that
320 is commonly assumed for non-spinosaurid theropods [33], such as abelisaurids and
321 carcharodontosaurids, is also supported by our dataset. The fraction of terrestrial preys in the diet
322 of these theropods appears to be significantly higher than that of spinosaurids. However, whether
323 these preys have been acquired through hunting [10,14,16,17], scavenging [10,14,16] or active
324 fishing [9,10,13–15,17,18] cannot be assessed with our dataset.

325 In the model, for some non-spinosaurid theropod individuals, the inferred $\delta^{44/42}\text{Ca}$ signature
326 of the diet is more positive than the source with the most positive $\delta^{44/42}\text{Ca}$ values. Four non-
327 mutually exclusive hypotheses could be proposed to explain this inconsistency. First of all, it can
328 be due to the absence of a heavier source of Ca in our dataset. One of the possibilities is that this

329 heavy source indeed corresponds to an unidentified terrestrial taxon at the bottom of the food
330 chain. Alternatively, it can correspond to *Nigersaurus taqueti* mineralized tissues for which only
331 two teeth have been measured, and which exhibit the highest $\delta^{44/42}\text{Ca}$ values of the dataset.
332 Another possibility is that this source of heavy isotopes of Ca comes from the digestion of
333 carbonated gastroliths. The ingestion of gastroliths among dinosaurs has been documented
334 thanks to comparisons with modern archosaurs and fossil evidence, notably for spinosaurids
335 [10]. Because of the heavy Ca isotope composition of carbonates [34,41,42], this consumption
336 could buffer the $\delta^{44/42}\text{Ca}$ towards ^{44}Ca enriched values among studied archosaurs. This could
337 lead to an overestimation of the fraction of herbivorous dinosaurs in diets inferred from our
338 mixing model, which could explain the inconsistency with non-spinosaurid theropods in our
339 model. The third hypothesis is that this inconsistency is the result of an overestimation of the
340 diet-enamel shift, due to the lack of data collected on modern archosaurs, which can also lead to
341 an overestimation of the fraction of herbivorous dinosaurs in diets (values potentially shifted
342 toward heavy calcium). However, none of these hypotheses is susceptible to change the
343 conclusions made on the resource partitioning among these predators or to the more piscivorous-
344 oriented diet of spinosaurids. Indeed, a validation of these hypotheses will imply a more
345 piscivorous diet for all these predators, even providing more support to the piscivorous-oriented
346 diet of spinosaurids.

347

348 **5. CONCLUSIONS**

349 Our data imply that the abundance of apex predators in these peculiar African mid-Cretaceous
350 ecosystems can be sustained simultaneously by at least two main sources: fish and herbivorous
351 dinosaurs, resulting in a general abundance of prey. The present Ca isotope dataset also supports

352 the ecological partitioning of potential predatory competitors [7,8]. Indeed, the different
353 ecological specializations of these predators resulted in different mixtures between carnivorous
354 and piscivorous habits and are likely to have reduced the ecological competition between them.
355 Calcium isotopes support the hypothesis of spinosaurids regarded as semi-aquatic predators [7,8]
356 and adopting a piscivorous diet, with non-spinosaurid theropods having a more terrestrial
357 ecology and diet, feeding mainly on herbivorous dinosaurs. This ecological partitioning,
358 combined with the abundance of prey, is consistent with the observed high abundance of
359 predators in these faunas [4,5].

360 The present study underlines a complex trophic structure relying on the interaction
361 between aquatic and terrestrial environments. One of the key points for the sustainability of these
362 palaeoecosystems was the piscivorous-oriented diet of spinosaurids. Whether this ecological
363 structure is a global feature among other mid-Cretaceous faunas is an open question [7, 10 and
364 reference therein], but beyond this, Ca isotopes offer promising perspectives to study trophic
365 structures in long extinct vertebrate assemblages.

366

367 **Data accessibility.**

368 The dataset table compiling all elemental concentrations and isotopic ratio measurements is
369 available as electronic supplementary material.

370

371 **Authors' contributions.**

372 The project was conceived by J.E.M., A.H., R.Am., T.T., R.Al. and V.B. . R.Am. and R.Al.
373 accessed samples for analysis. A.H. and J.E.M. sampled the specimens and conducted

374 purification in the clean lab. A.H. and F.A.G. performed elemental concentration measurements.
375 A.H., J.E.M. and T.T. performed MC-ICP-MS measurements. All authors discussed the results.
376 The first draft version of the text was written by A.H. then subsequently, J.E.M., R.Am., T.T.,
377 R.Al., F.A.G. and V.B. contributed to it.

378

379 **Competing interests.**

380 We declare we have no competing interests.

381

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405

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519

520 **Captions**

521 **Figure 1.** Three isotopes plot: $\delta^{43/42}\text{Ca}$ as a function of $\delta^{44/42}\text{Ca}$ (‰, reference standard ICP Ca)
522 for all samples and standards analyzed for Ca isotope compositions in this study. Ca isotope
523 compositions fall on a line with a y axis intercept of 0.009 ± 0.015 (‰, 2SE), indistinguishable
524 from theoretical 0‰ intercept. The slope value of this line is 0.509 ± 0.016 (2SE),
525 indistinguishable from the 0.507 slope predicted by the exponential mass-dependent
526 fractionation law. Error bars at the bottom right are average 2SD for $\delta^{43/42}\text{Ca}$ and $\delta^{44/42}\text{Ca}$. The
527 two most external lines delimit the prediction interval, and the two lines accompanying the
528 middle line correspond to the 95% confidence interval of the regression line.

529

530 **Figure 2. (A)** PAAS Shale-normalized REE profiles of fossil bioapatite from the Aptian-Albian
531 of Gadoufaoua, Niger. Concentrations are plotted on a logarithmic scale. Plain black line: typical
532 REE profile at Gadoufaoua; yellow line: exotic profile of sample GCA5; purple line: exotic
533 profile of sample GSA7; red line: exotic profile of sample GSP3. **(B)** PAAS Shale-normalized
534 REE profiles of fossil bioapatite from the Cenomanian of Kem Kem beds, Morocco.
535 Concentrations are plotted on a logarithmic scale. For comparison, the plain black line represents
536 the typical REE profile at Gadoufaoua; yellow line: typical profile of samples from the locality
537 of Jebel Al Qabla; blue line: typical profile of samples from the locality of Bou Laâlou; red line:
538 typical profile of samples from the locality of Takemout.

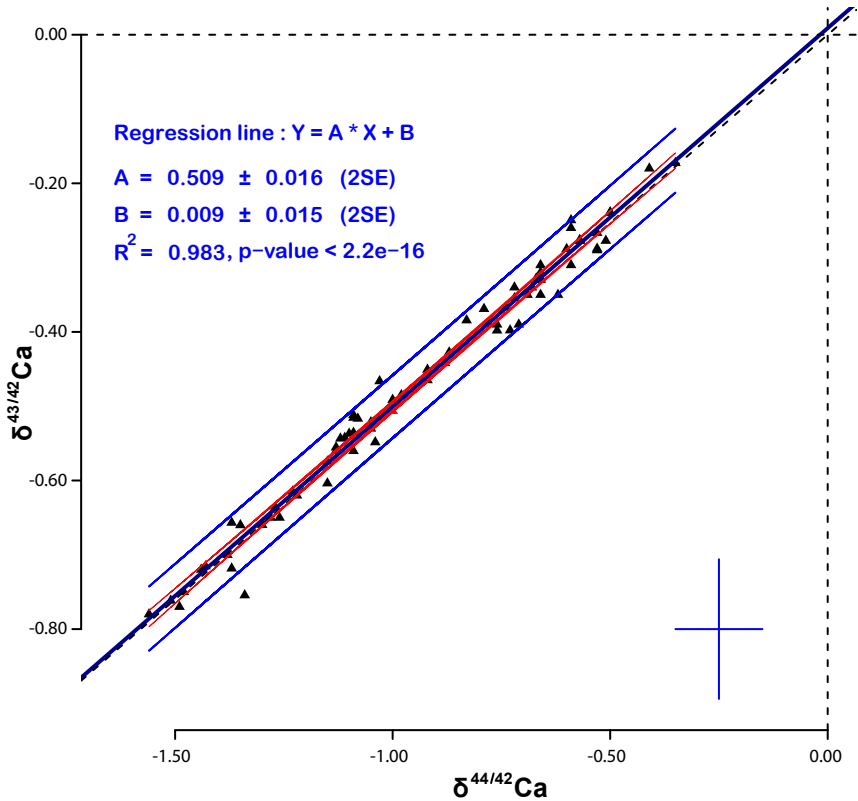
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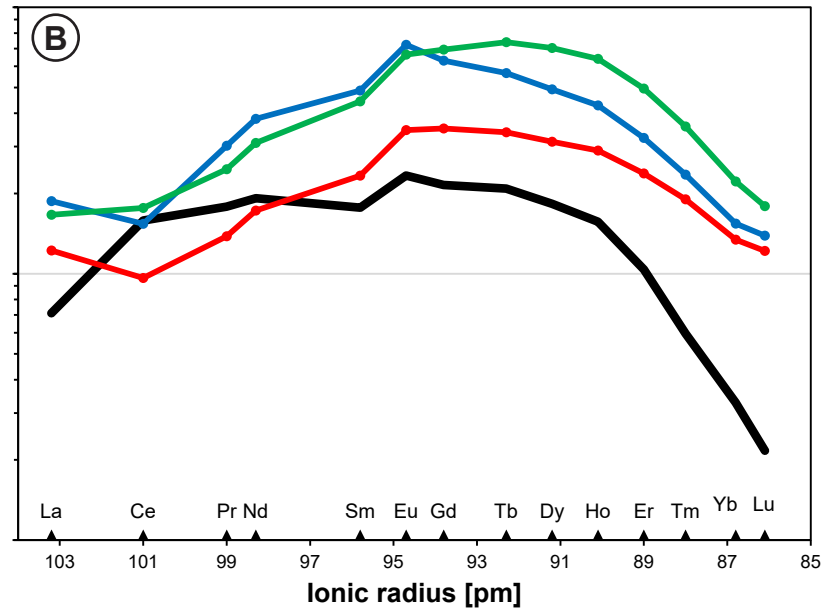
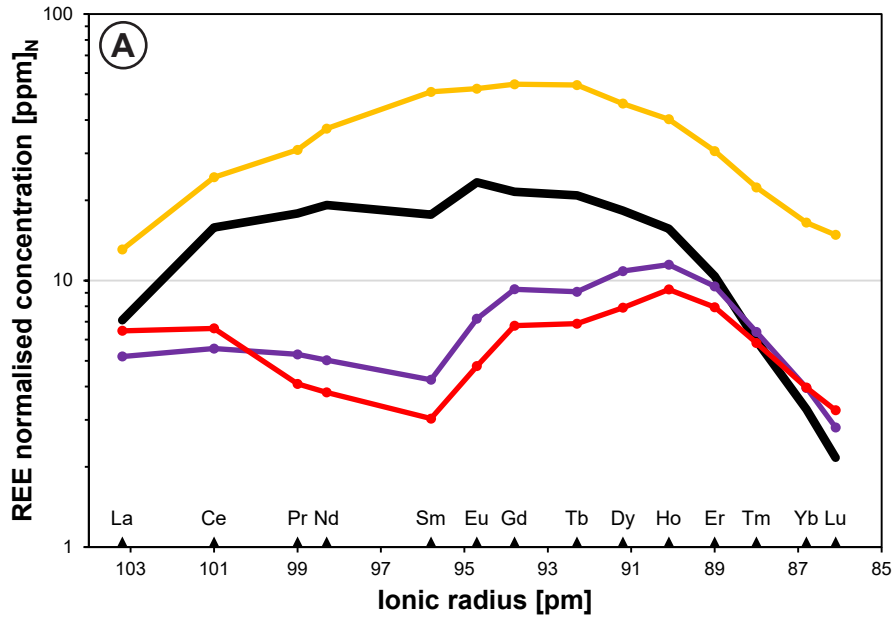
540 **Figure 3.** $\delta^{44/42}\text{Ca}$ data reported from Gadoufaoua **(A)** and Kem Kem beds faunas **(B)**. Groups
541 representing at least 5 specimens are represented through boxplots, for which the middle line
542 represents the median, the box limits correspond to the 1st and 3rd quartiles, and the whiskers are
543 extended between the maximum and the minimum $\delta^{44/42}\text{Ca}$. The mean 2SD of each measure is
544 represented at the bottom right of each graph. The degree of significance of the difference
545 between the theropod groups are represented with stars (*), with the three stars indicating a high
546 significance (Wilcoxon rank sum test: p-value < 0.001). Taxonomic groups are identified by
547 their number and represent: 1: *Nigersaurus taqueti* (n=2), 2: *Ouranosaurus nigeriensis* (n=9), 3:
548 non-spinosaurid theropods from Gadoufaoua (n=9), 4: *Sarcosuchus imperator* (n=8), 5:
549 spinosaurids from Gadoufaoua (n=8), 6: Lepisosteiformes (n=3), 7: Pycnodontiformes indet.
550 (n=5), 8: cf. *Araripesuchus* (n=3), 9: Pterosauria indet. (n=3), 10: non-spinosaurid theropods
551 from Kem Kem beds (n=7), 11: Crocodylomorpha indet. (n=3), 12: spinosaurids from Kem

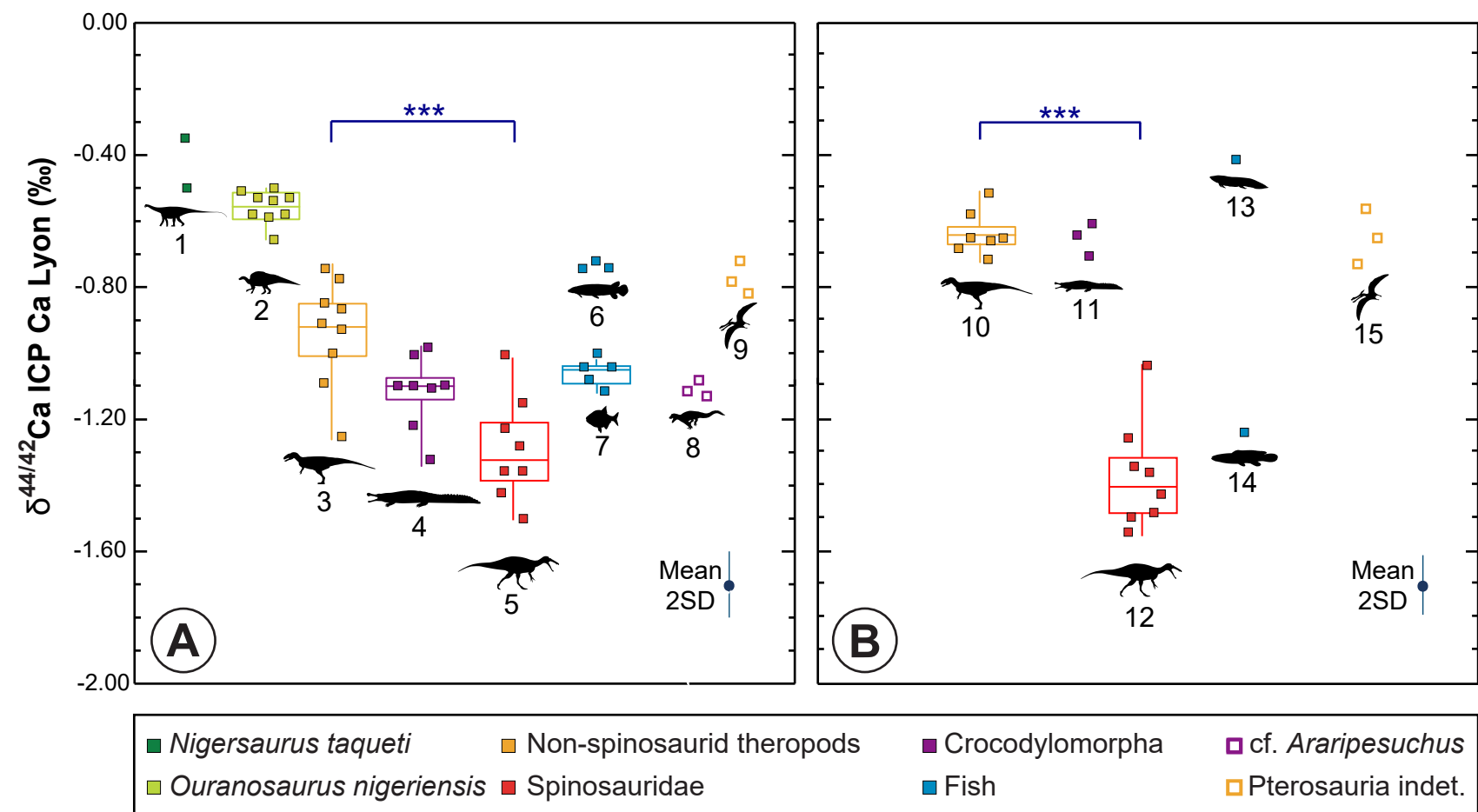
552 Kem (n=8), 13: *Neoceratodus africanus* (n=1), 14: *Stromerichthys* sp. (n=1), 15: Anhangueridae
553 indet. (n=3).

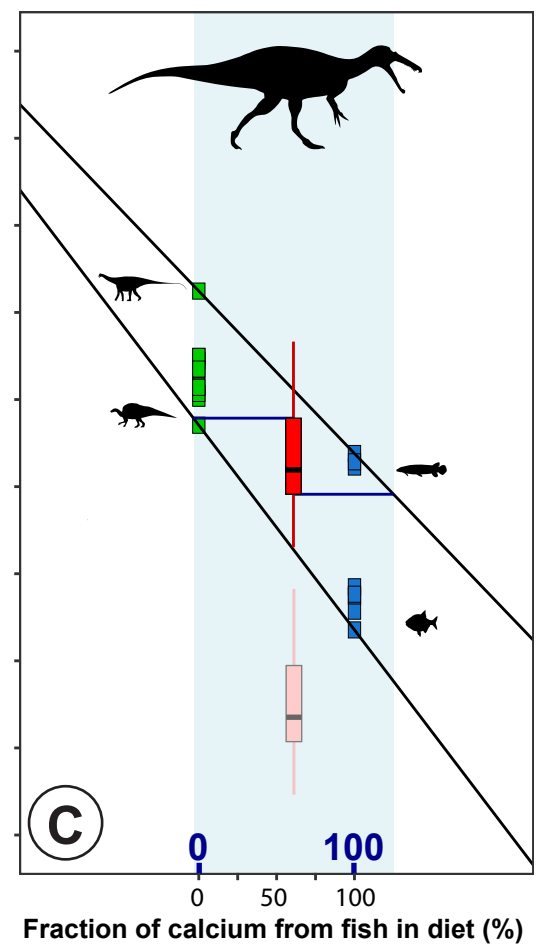
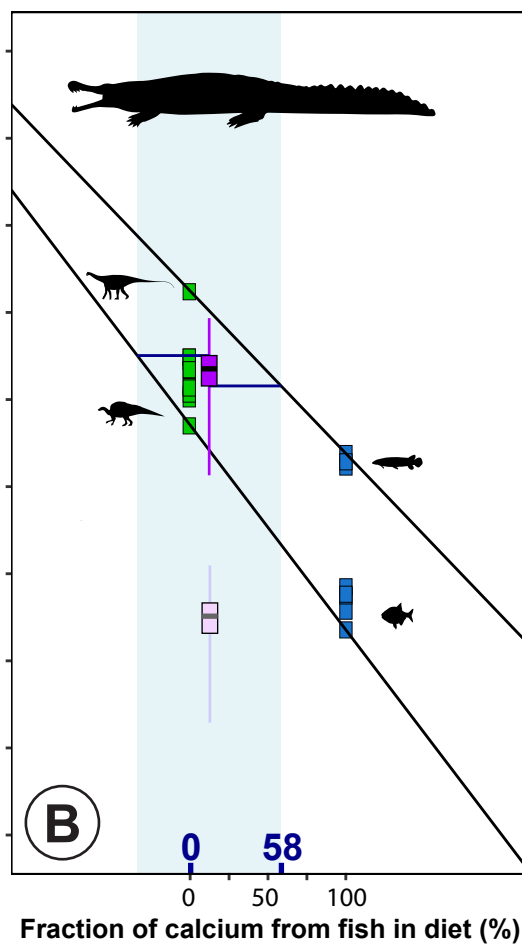
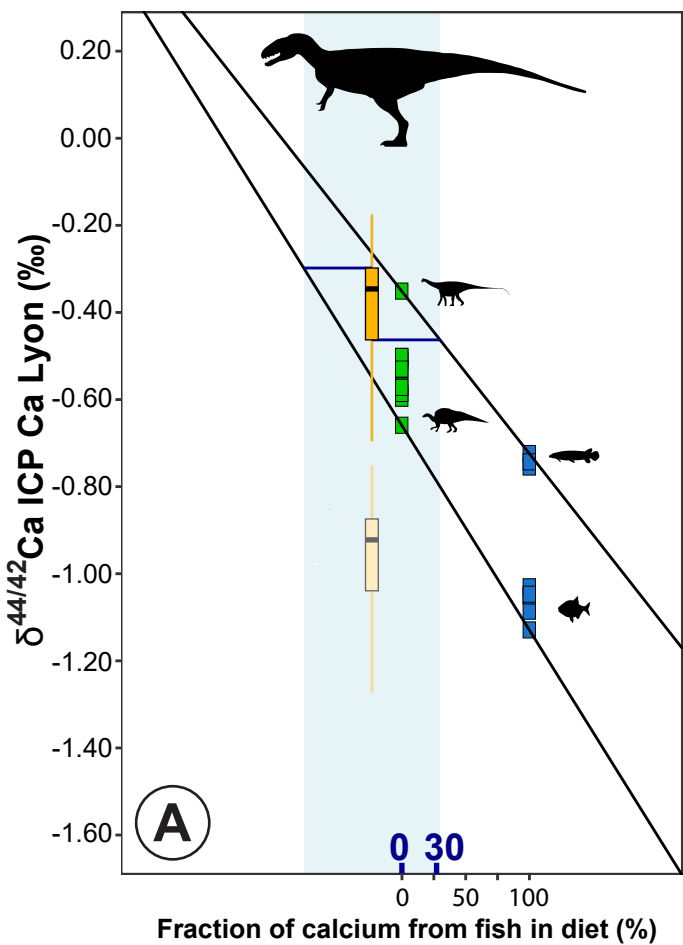
554

555 **Figure 4.** Mixing model of the calcium source for non-spinosaurid theropods **(A)** *Sarcosuchus*
556 *imperator* **(B)** and spinosaurids **(C)** from the Gadouafaoua fauna. $\delta^{44/42}\text{Ca}$ data collected on
557 herbivorous dinosaurs and fishes are represented respectively in green and blue squares. In each
558 part of the figure the faded lower boxplot represents the enamel $\delta^{44/42}\text{Ca}$ values measured for
559 each apex predators, whereas the dark upper boxplot represents the isotopic signature of their
560 calcium source estimated to be shifted by +0.57‰ from the signature of their enamel (Methods
561 *(d)*). Boxplots are yellow for non-spinosaurid theropods (A), violet for *Sarcosuchus imperator*
562 (B) and red for spinosaurids (C). The line in boxplot is equal to the median, boxes are limited by
563 their 1st and 3rd quartile, and whiskers represent the maximum and the minimum values. The
564 estimated diet fraction range of Ca derived from the fish source are indicated in bold above the
565 x-axis and expressed in percent.









Taxonomy	Locality	Lab name	Material	$\delta^{44}/^{42}\text{Ca}$ (‰)	2SD	$\delta^{43}/^{42}\text{Ca}$ (‰)	2SD	n
Non-spinosaurid theropod	Gadoufaoua, Niger	GCA1	tooth enamel	-0.92	0.34	-0.45	0.22	2
Non-spinosaurid theropod	Gadoufaoua, Niger	GCA2	tooth enamel	-0.74	0.00	-0.37	0.11	2
Non-spinosaurid theropod	Gadoufaoua, Niger	GCA3	tooth enamel	-1.03	0.03	-0.52	0.02	2
Non-spinosaurid theropod	Gadoufaoua, Niger	GCA4	tooth enamel	-0.87	0.24	-0.43	0.13	2
Non-spinosaurid theropod	Gadoufaoua, Niger	GCA6	tooth enamel	-0.78	0.12	-0.39	0.08	2
Non-spinosaurid theropod	Gadoufaoua, Niger	GCA7	tooth enamel	-0.88	0.17	-0.44	0.16	3
Non-spinosaurid theropod	Gadoufaoua, Niger	GCA8	tooth enamel	-1.09	0.18	-0.56	0.03	2
Non-spinosaurid theropod	Gadoufaoua, Niger	GCA9	tooth enamel	-0.92	0.05	-0.46	0.06	2
Non-spinosaurid theropod	Gadoufaoua, Niger	GCA10	tooth enamel	-1.27	0.12	-0.64	0.10	3
cf. <i>Araripesuchus</i> sp.	Gadoufaoua, Niger	GAR1	tooth enamel	-1.12	0.07	-0.54	0.02	2
cf. <i>Araripesuchus</i> sp.	Gadoufaoua, Niger	GAR3	tooth enamel	-1.15	0.15	-0.57	0.09	5
cf. <i>Araripesuchus</i> sp.	Gadoufaoua, Niger	GAR5	tooth enamel	-1.08	0.06	-0.52	0.01	2
Lepisosteiformes indet.	Gadoufaoua, Niger	GLE1	scale ganoine	-0.76	0.00	-0.40	0.00	2
Lepisosteiformes indet.	Gadoufaoua, Niger	GLE2	scale ganoine	-0.72	0.06	-0.35	0.06	2
<i>Nigersaurus taqueti</i>	Gadoufaoua, Niger	GAU1	tooth enamel	-0.35	0.14	-0.17	0.09	3
<i>Nigersaurus taqueti</i>	Gadoufaoua, Niger	GAU12	tooth enamel	-0.50	0.05	-0.24	0.00	2
<i>Ouranosaurus nigeriensis</i>	Gadoufaoua, Niger	GON1	tooth enamel	-0.60	0.07	-0.29	0.00	2
<i>Ouranosaurus nigeriensis</i>	Gadoufaoua, Niger	GON2	tooth enamel	-0.59	0.12	-0.25	0.08	2
<i>Ouranosaurus nigeriensis</i>	Gadoufaoua, Niger	GON3	tooth enamel	-0.53	0.01	-0.27	0.07	2
<i>Ouranosaurus nigeriensis</i>	Gadoufaoua, Niger	GON4	tooth enamel	-0.57	0.05	-0.28	0.02	2
<i>Ouranosaurus nigeriensis</i>	Gadoufaoua, Niger	GON5	tooth enamel	-0.51	0.03	-0.28	0.02	2
<i>Ouranosaurus nigeriensis</i>	Gadoufaoua, Niger	GON7	tooth enamel	-0.57	0.11	-0.28	0.13	3
<i>Ouranosaurus nigeriensis</i>	Gadoufaoua, Niger	GON8	tooth enamel	-0.50	0.09	-0.24	0.08	3
<i>Ouranosaurus nigeriensis</i>	Gadoufaoua, Niger	GON9	tooth enamel	-0.66	0.05	-0.33	0.07	4
<i>Ouranosaurus nigeriensis</i>	Gadoufaoua, Niger	GON10	tooth enamel	-0.53	0.14	-0.29	0.10	3
<i>Pliodetes</i> sp.	Gadoufaoua, Niger	GPL2	scale ganoine	-0.67	0.19	-0.33	0.17	2
Pterosauria indet.	Gadoufaoua, Niger	GAU3	tooth enamel	-0.83	0.06	-0.38	0.05	2
Pterosauria indet.	Gadoufaoua, Niger	GAU5	tooth enamel	-0.73	0.09	-0.40	0.07	2
Pterosauria indet.	Gadoufaoua, Niger	GAU14	tooth enamel	-0.79	0.10	-0.37	0.04	2
Pycnodontiformes indet.	Gadoufaoua, Niger	GPY1	tooth enameloid	-1.03	0.11	-0.47	0.04	2
Pycnodontiformes indet.	Gadoufaoua, Niger	GPY2	tooth enameloid	-1.13	0.10	-0.56	0.10	3
Pycnodontiformes indet.	Gadoufaoua, Niger	GPY3	tooth enameloid	-1.09	0.15	-0.54	0.19	3
Pycnodontiformes indet.	Gadoufaoua, Niger	GPY4	tooth enameloid	-1.05	0.11	-0.53	0.13	3
Pycnodontiformes indet.	Gadoufaoua, Niger	GPY5	tooth enameloid	-1.05	0.08	-0.52	0.04	3
<i>Sarcosuchus imperator</i>	Gadoufaoua, Niger	GSA1	tooth enamel	-0.98	0.08	-0.48	0.09	2
<i>Sarcosuchus imperator</i>	Gadoufaoua, Niger	GSA2	tooth enamel	-1.09	0.03	-0.51	0.00	2
<i>Sarcosuchus imperator</i>	Gadoufaoua, Niger	GSA3	tooth enamel	-1.22	0.26	-0.62	0.13	3
<i>Sarcosuchus imperator</i>	Gadoufaoua, Niger	GSA4	tooth enamel	-1.09	0.13	-0.52	0.04	3
<i>Sarcosuchus imperator</i>	Gadoufaoua, Niger	GSA5	tooth enamel	-1.00	0.16	-0.49	0.15	5
<i>Sarcosuchus imperator</i>	Gadoufaoua, Niger	GSA6	tooth enamel	-1.11	0.10	-0.54	0.10	4
<i>Sarcosuchus imperator</i>	Gadoufaoua, Niger	GSA9	tooth enamel	-1.10	0.04	-0.54	0.01	2
<i>Sarcosuchus imperator</i>	Gadoufaoua, Niger	GSA10	tooth enamel	-1.34	0.01	-0.75	0.08	2
<i>Suchomimus tenerensis</i>	Gadoufaoua, Niger	GSP1	tooth enamel	-1.43	0.01	-0.72	0.09	2
<i>Suchomimus tenerensis</i>	Gadoufaoua, Niger	GSP2	tooth enamel	-1.37	0.17	-0.66	0.02	2
<i>Suchomimus tenerensis</i>	Gadoufaoua, Niger	GSP4	tooth enamel	-1.23	0.03	-0.61	0.02	2
<i>Suchomimus tenerensis</i>	Gadoufaoua, Niger	GSP5	tooth enamel	-1.37	0.01	-0.72	0.03	2
<i>Suchomimus tenerensis</i>	Gadoufaoua, Niger	GSP6	tooth enamel	-1.30	0.15	-0.66	0.12	2
<i>Suchomimus tenerensis</i>	Gadoufaoua, Niger	GSP7	tooth enamel	-1.04	0.09	-0.55	0.04	3
<i>Suchomimus tenerensis</i>	Gadoufaoua, Niger	GSP8	tooth enamel	-1.51	0.09	-0.76	0.06	2
<i>Suchomimus tenerensis</i>	Gadoufaoua, Niger	GSP9	tooth enamel	-1.15	0.20	-0.60	0.10	2
Anhangueridae indet.	Bou Laâlou, Morocco	MBL-003	tooth enamel	-0.59	0.09	-0.31	0.09	2
<i>Carcharodontosaurus saharicus</i>	Bou Laâlou, Morocco	MBL-024	tooth enamel	-0.59	0.08	-0.26	0.08	3
<i>Carcharodontosaurus saharicus</i>	Bou Laâlou, Morocco	MBL-025	tooth enamel	-0.66	0.02	-0.31	0.02	2
<i>Spinosaurus aegypticus</i>	Bou Laâlou, Morocco	MBL-042	tooth enamel	-1.49	0.24	-0.77	0.24	2
<i>Spinosaurus aegypticus</i>	Bou Laâlou, Morocco	MBL-043	tooth enamel	-1.48	0.07	-0.75	0.07	2
<i>Spinosaurus aegypticus</i>	Bou Laâlou, Morocco	MBL-044	tooth enamel	-1.35	0.02	-0.66	0.02	2
<i>Spinosaurus aegypticus</i>	Bou Laâlou, Morocco	MBL-045	tooth enamel	-1.38	0.02	-0.70	0.02	2
Anhangueridae indet.	Jebel Al Qabla, Morocco	MJQ-001	tooth enamel	-0.76	0.06	-0.39	0.06	2
Theropoda indet.	Jebel Al Qabla, Morocco	MJQ-008	tooth enamel	-0.66	0.03	-0.35	0.03	2
Theropoda indet.	Jebel Al Qabla, Morocco	MJQ-009	tooth enamel	-0.66	0.05	-0.32	0.05	2
Theropoda indet.	Jebel Al Qabla, Morocco	MJQ-010	tooth enamel	-0.69	0.16	-0.35	0.16	2
<i>Neoceratodus africanus</i>	Jebel Al Qabla, Morocco	MJQ-020	tooth enameloid	-0.41	0.05	-0.18	0.05	2
<i>Stromerichthys</i> sp.	Jebel Al Qabla, Morocco	MJQ-036	scale ganoine	-1.26	0.07	-0.65	0.07	3
<i>Spinosaurus aegypticus</i>	Jebel Al Qabla, Morocco	MJQ-039	tooth enamel	-1.44	0.23	-0.72	0.23	2
Crocodylomorpha indet.	Jebel Al Qabla, Morocco	MJQ-040A	tooth enamel	-0.66	0.14	-0.33	0.14	2
<i>Carcharodontosaurus saharicus</i>	Takemout, Morocco	MTA-017	tooth enamel	-0.53	0.02	-0.29	0.02	2
<i>Carcharodontosaurus saharicus</i>	Takemout, Morocco	MTA-019	tooth enamel	-0.72	0.16	-0.34	0.16	2
Anhangueridae indet.	Takemout, Morocco	MTA-020	tooth enamel	-0.68	0.06	-0.34	0.06	2
<i>Spinosaurus aegypticus</i>	Takemout, Morocco	MTA-026	tooth enamel	-1.13	0.11	-0.56	0.11	2
<i>Spinosaurus aegypticus</i>	Takemout, Morocco	MTA-027	tooth enamel	-1.56	0.08	-0.78	0.08	2
<i>Spinosaurus aegypticus</i>	Takemout, Morocco	MTA-028	tooth enamel	-1.28	0.06	-0.65	0.06	2
Crocodylomorpha indet.	Takemout, Morocco	MTA-030	tooth enamel	-0.62	0.10	-0.35	0.10	2
Crocodylomorpha indet.	Takemout, Morocco	MTA-031	tooth enamel	-0.71	0.10	-0.39	0.10	2
SRM1486 (NIST)	-	standard	bone meal	-1.00	0.10	-0.51	0.09	32