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Integrated stratigraphy and paleoceanographic evolution of the pre-evaporitic phase of the
 Messinian salinity crisis in the Eastern Mediterranean as recorded in the Tokhni section
 (Cyprus island)

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- 23
- 24 Abstract

25 An integrated micropaleontologic, magnetostratigraphic and cyclostratigraphic 26 investigation of the Tokhni composite section (Southern Cyprus Island, Eastern 27 Mediterranean) refines the previously published age model and paleoenvironmental 28 interpretation particularly concerning its uppermost pre-Messinian Salinity Crisis interval (pre-MSC), between 6.46 and 5.97 Ma. This section is characterized by a precessionpaced alternation of red shales and limestones, which correlate with insolation maxima and minima on the basis of their  $\partial^{18}$ O signatures and calcareous nannofossil assemblages. The planktonic foraminifer and magnetostratigraphic events permit the tuning of the sedimentary cycles to the 65°N summer insolation curve and to the Mediterranean pre-evaporitic reference sections.

The upper bathyal sedimentary succession of the Tokhni composite section records paleoceanographic changes at 6.4 and 6.1 Ma, indicating increasingly stressed conditions both at the sea floor and in the water column. Compared to the Western Mediterranean pre-MSC successions, we observe less severe sea floor anoxic conditions at times of insolation maxima and higher salinity surface and bottom waters at times of insolation minima. Moreover, from 6.1 Ma to the MSC onset we observe a progressively increase of continental-derived waters, which was likely caused by a tectonic pulse.

The MSC onset at 5.97 Ma is marked by the deposition of clastic carbonates rather than primary evaporitic facies and is approximated by the last recovery of foraminifera, the abundance peaks of *Helicosphaera carteri* and *Umbilicosphaera rotula* and the decrease of the <sup>87/86</sup>Sr. The MSC onset is recorded two cycles below the Messinian Erosional Surface (MES, 5.60 Ma) and the overlying clastic evaporites, suggesting a hiatus of approximately 350 kyr.

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Keywords: Eastern Mediterranean, Messinian, calcareous nannofossils, foraminifera,
 integrated stratigraphy, paleoenvironment.

51

52 1. Introduction

53 The pre-evaporitic phase of the Messinian salinity crisis (MSC) was characterized by 54 marked paleoceanographic changes culminating in the deposition of huge volume of

55 evaporites in the shallow and deep/intermediate settings of the Mediterranean basin 56 during stage 1 (from 5.971 Ma; Manzi et al. 2013) and stage 2 (from 5.60 Ma; Roveri et al. 2009), respectively. The major steps of this paleoceanographic evolution occurred at 7.16, 57 58 6.7, 6.4 - 6.29 and 6.1 - 6.0 Ma (see Kouwenhoven et al. 2006, with references therein). Each of these steps was conductive to increasingly stressing conditions both at the sea 59 floor and in the water column, as documented by the progressive increase of oligotypic 60 calcareous plankton and benthic foraminifer assemblages. The first two steps were related 61 62 to the tectonic narrowing and/or closure of the Atlantic connections in the Rifian corridors (Krijgsman et al. 1999a and b, Krijgsman and Langereis 2000), which caused the slowing 63 64 or stopping of the Mediterranean deep circulation. However, Blanc-Valleron et al. (2002) noted that these paleoceanographic steps roughly follow the 400 kyr eccentricity cycle, 65 and Kouwenhoven et al. (2006) also suggested a relation between the increasing 66 67 restriction of the deep circulation and this orbital parameter. In addition, the restricted 68 connections with the Atlantic from 7.16 Ma increased the sensitivity of the Mediterranean 69 basin to record the precession-controlled climatic changes resulting in a more regular 70 formation of sapropels at times of precession minima (Sierro et al. 2003).

71 In the deep- and intermediate-water successions, such as at Monte del Casino (northern 72 Apennines, Italy) and Pissouri (southern Cyprus) sections, respectively (Kouwenhoven et 73 al. 2003; Kouwenhoven et al. 2006), the step at 7.16 Ma was characterized by the 74 disappearance of oxyphilic benthic foraminifera and the increase of taxa tolerating low oxygen levels and raised salinity (Van der Zwaan 1982, Kouwenhoven et al. 1999). As 75 76 seen in the Gibliscemi-Falconara sections (Sicily), the central Mediterranean was more 77 severely affected by the reduced deep-water circulation, marked by a temporarily 78 disappearance of benthic foraminifera (Kouwenhoven et al. 2003). Moreover, diatomite 79 beds started to deposit in the Mediterranean between 7.16 and 6.7 Ma (Pestrea et al. 80 2002), indicating a significant availability of silica at times of enhanced eutrophication,

81 likely connected to upwelling episodes during insolation minima (Sierro et al. 2003, Flores
82 et al. 2005).

Beginning at 6.7 Ma (second step), not only the benthic foraminifera show an abundance 83 84 increase of stress tolerant taxa (indicating low oxygen and/or raised salinity bottom conditions) and are absent in sapropel (Sierro et al. 2003, Kouwenhoven et al. 2006), but 85 86 also the calcareous plankton assemblages are characterized by reduced diversity and abundance (Sierro et al. 2003). Moreover, an increased response to the insolation 87 88 fluctuations is highlighted by the dominance of cold/eutrophic calcareous plankton taxa at 89 times of insolation minima and warm/oligotrophic taxa at times of insolation maxima 90 (Blanc-Valleron et al. 2002, Sierro et al. 2003, Flores et al. 2005). It is worth noting that 91 changes in calcareous plankton assemblages from 6.7 Ma equally affected both deep and 92 shallow successions.

From 6.4 Ma the Plankton/Benthos ratio shows abrupt fluctuations, with values ranging from 100% to near 0%, indicating the occurrence of a-benthic foraminiferal layers due to anoxic conditions during insolation maxima and of strongly reduced/a-planktonic layers (insolation minima) (Sierro et al. 2003). In the superficial waters, the presence of oligotypic assemblages dominated by *Turborotalia quinqueloba* and *T. multiloba* (up to 80%) during insolation minima are usually related to increased salinity corresponding to very arid climatic phases (Sierro et al. 2003, Kouwenhoven et al. 2006).

The final step at 6.1 - 6.0 Ma certifies the gradual final decrease of calcareous microfossils,
whose disappearance approximates the MSC onset at 5.971 Ma (Manzi et al. 2007, Lozar
et al. 2010, Manzi et al. 2011, Manzi et al. 2013).

The eastern Mediterranean pre-evaporitic (pre-MSC) micropaleontologic record has been mainly studied onshore of Cyprus and Crete (Van der Zwaan, 1982, Triantaphyllou et al. 105 1999, Kouwenhoven et al. 2006, Drinia et al. 2007, Orszag-Sperber et al. 2009). On the southern part of the Cyprus Island, an integrated stratigraphy was obtained for the Tokhni

107 and Pissouri sections (Krijgsman et al. 2002, Orszag-Sperber et al. 2009), located in the Psematismenos and the Pissouri basins (Fig. 1), respectively. These two sections were 108 109 correlated with the Perales reference section (Sorbas basin) for the western 110 Mediterranean (Sierro et al. 2001) by means of biomagnetostratigraphic and 111 cyclostratigraphic constraints. Overall, the paleoceanographic evolution observed in the 112 Cyprus basin is similar to that envisaged in the western Mediterranean, although the pre-MSC sedimentary successions differ for the scarce presence or absence of sapropels and 113 114 for the occurrence of chaotic/carbonate deposits ("barre jaune") just below the lowermost gypsum bed (Kouwenhoven et al. 2006, Orszag-Sperber et al. 2009). Moreover, the 115 116 identification of MSC onset in these sections has been debated. According to Krijgsman et al. (2002) and Kouwenhoven et al. (2006) it is represented by the lowermost gypsum bed 117 in the Pissouri section. Differently, according to Orszag-Sperber et al. (2009), it coincides 118 119 in the Tokhni section with the basal stromatolitic bed of the "barre jaune" and, 120 consequently, the deposition of the lowermost gypsum bed is delayed by 60 kyrs. Both 121 these interpretations have been questioned by Manzi et al. (2016) based on 122 sedimentologic and stratigraphic evidences. These authors stated that on Cyprus the 123 Primary Lower Gypsum (PLG, Roveri et al. 2009) of the 1<sup>st</sup> stage of the MSC are lacking, 124 whereas the gypsum deposits are clastic facies floored by an angular unconformity likely 125 corresponding to the Messinian erosional surface (MES). Thus, the gypsum unit of the 126 Tokhni section has been ascribed to the stage 2 of the MSC (Roveri et al. 2009). Consequently, Manzi et al. (2016) suggested that MSC onset is best approximated by the 127 128 last occurrence of foraminifera, identified within the interval corresponding to the "barre 129 jaune" of Orszag-Sperber et al. (2009).

Here, we present a reinvestigation of the upper part of the Tokhni section producing an updated calcareous plankton biostratigraphy and cyclostratigraphy, validated by new magnetostratigraphic data. The micropaleontologic and isotopic data, supported by the

new age model and integrated with the stratigraphic and sedimentologic evidences as presented in Manzi et al. (2016), allowed the reconstruction of the paleoenvironmental and paleoceanographic evolutions in the Psematismenos basin during the pre-MSC and their comparison with those of the central and western Mediterranean successions.

137

138 2. Geological setting and section

The Tokhni composite section is exposed in the badlands 1 km southwest from the village 139 140 of Tokhni (southern Cyprus Island), located at the southeast termination of the Troodos 141 Massif, in the Psematismenos basin (Fig.1). The composite section combines 4 sub-142 sections (To-0, To-1, To-2 and To-3; Fig. 2; Manzi et al. 2016), and comprises the upper part of the Pakhna Formation (Bagnall 1960, Gass 1960, Pantazis 1967), and the 143 evaporites of the Kalavasos Formation. Here we follow the subdivision of the Pakhna 144 145 Formation into four sub-units (from the bottom, PK-A<sub>1</sub>, PK-A<sub>2</sub>, PK-A<sub>3</sub> and PK-B) as 146 proposed by Manzi et al. (2016) (Fig. 2).

147 1) Sub-unit PK-A<sub>1</sub> – It consists of m-thick alternation of darker and lighter homogeneous
148 blue marls. Orszag-Sperber et al. (2009) recognized the Tortonian-Messinian boundary in
149 the upper part of this sub-unit on the basis of the first occurrence of the planktonic
150 foraminifer *Globorotalia miotumida*.

2) Sub-unit PK-A<sub>2</sub> – It is characterized by a lithologic cyclic stacking pattern formed by an
alternation of prominent marls and reddish shales.

3) Sub-unit PK-A<sub>3</sub> – It is mainly characterized by a cyclic alternation of reddish shales and whitish micritic limestones, and its base is marked by a 80 cm-thick whitish limestone bed. However, from ca. 15 m above the base of this sub-unit, marl layers and/or slightly prominent pink diatomitic layers are associated with or replace the whitish limestone within the basic cycle. Diatomites are particularly common in the upper part of this sub-unit and within the upper PK-B sub-unit (see below). The micritic limestone layers, in particular the

lowermost one, are laterally continuous and easily detectable in the field, and were used 159 160 as key-beds in the reconstruction of the composite section (Fig. 2). These layers are made up of a mixture of clay and coccoliths cemented by micron-sized calcite crystals (Manzi et 161 162 al. 2016). Another useful key-bed observed in the upper part of this sub-unit is a thin conglomerate layer (thickness 25-40 cm), showing an erosive base. This layer is entirely 163 164 made up of limestone-derived pebbles floating in a bioclastic packstone matrix; the latter 165 contains planktonic and benthic foraminifera, siliceous sponge spicules, echinoid spines 166 and fragments of coralline algae. Sparse silt-size terrigenous grains are also present.

4) Sub-unit PK-B - This uppermost sub-unit has been recognized only in the southern 167 168 subsections To-2 and To-3. It is characterized by the presence of 3 limestone beds (indicated as A, B and C) showing different characteristics with respect to the limestones 169 170 of sub-unit PK-A<sub>3</sub> and intercalated by reddish shales and pink diatomites. These limestone 171 beds belong to the "barre jaune" described in the Tokhni section by Orszag-Sperber et al. 172 (2009), who interpreted them as precession-controlled stromatolites suggesting periodical 173 shallowing and drying up of the basin. Differently, Manzi et al. (2016) did not recognize 174 evidences of shallowing or drying up. According to these authors the lower part of the 175 limestone A is a finely laminated microbialite and contains cavities possibly indicating the 176 displacive growth of sub-millimetric gypsum crystals, successively replaced by micrite. The 177 upper part of limestone A shows penecontemporaneous reworking of the microbialite by 178 subaqueous gravity flows. The two uppermost limestone layers (B and C) show completely 179 different characteristics and internal organization. They are made up of coarse-grained 180 clastic sediments, grainstone and rudstone likely deriving from the dismantlement of a 181 penecontemporaneous carbonate platform forming in the Tokhni area (Robertson et al. 182 1995).

The boundary between the Pakhna and the Kalavasos formations is sharp and discordant.
In fact, as described in Manzi et al. (2016), the uppermost part of the Pakhna Formation

185 shows from south to north: i) the disappearance of the PKB sub-unit; ii) a progressive truncation of the PK-A<sub>3</sub> sub-unit (Fig. 2); iii) a steeper inclination of the base of the 186 evaporites of Kalavasos Formation with respect to the Pakhna Formation. Accordingly, the 187 base of the Kalavasos Formation is an angular unconformity and coincides with the 188 189 Messinian erosional surface (MES); indeed, these evaporites are clastic and belong to the 190 Resedimented Lower Gypsum (RLG, Roveri et al. 2009) unit deposited during stage 2, 191 rather than representing the Primary Lower Gypsum (PLG of Roveri et al. 2009) of stage 1, 192 as previously argued by Orszag-Sperber et al. (2009). In the present study we focus on 193 the upper part of the Tokhni composite section, corresponding to sub-units PK-A<sub>3</sub> and PK-194 Β.

195

196 3. Material and Methods

197 **3.1.** Magnetostratigraphy

198 A total of 16 samples were collected in the subsection To-1 (10 - 31.5 m interval of the 199 composite section; Fig. 2), mainly from the limestone beds, due to the pervasive fracture of 200 the shales and lamination of the diatomites. Samples were thermally demagnetized in an 201 ASC electric hoven at the Alpine Laboratory of Paleomagnetism (Alp, Peveragno, Italy) 202 and were initially heated at 100°C and then up to 340-450°C, by successive increasing 203 steps of 30°C. At each step we measured the remanent magnetization in a 2G-Enterprises 204 DC SQUIDS cryogenic magnetometer, and the magnetic susceptibility to check for thermal alteration of the magnetic mineralogy. The NRM data were processed with the Remasoft 205 206 software (Chadima and Hrouda 2006), which computed the principal component analysis 207 of the linear vector (Kirschvink 1980) picked from orthogonal projection demagnetization 208 diagrams (Zijderveld 1967) to obtain the characteristic remanent magnetization (ChRM).

209

210 **3.2.** Foraminifera

211 The 87 samples collected from subsections To-1, To-2 and To-3 (Fig. 2) were dried and then soaked in diluted H<sub>2</sub>O<sub>2</sub> for a few days; successively, they were washed using a 63 µm 212 213 mesh, dried again and sieved to obtain the study fraction (> 125 µm). The planktonic 214 foraminifer record of the Tokhni section is biased by several problems: a) reworking of 215 Eocene to middle Miocene specimens (see also Orszag-Sperber et al. 2009) is present in 216 the PK-A<sub>2</sub> and lower part of PK-A<sub>3</sub> sub-units, up to 7 m of the composite section and it 217 gradually decreases upward; b) in the 0-28 m interval the foraminifer abundance is highly 218 variable and roughly decreases from the bottom to the top; in particular, the most 219 prominent limestones starting from 11.5 m are barren of both benthic and planktonic 220 foraminifera; c) inorganic particles (terrigenous grains, un-disaggregated sediment, etc.) represent a large portion of the washed residues; d) preservation is generally poor. For 221 these reasons qualitative observations were performed on the samples collected from PK-222 223 A<sub>2</sub> and lower part of PK-A<sub>3</sub> sub-units. A semi-quantitative analysis of selected taxa was 224 carried out on 48 out of 60 samples collected from PK-A3 and PK-B sub-units (from 7 m 225 upward of the Tokhni composite section, see Fig. 2), avoiding samples barren in 226 foraminifera (limestones) and samples characterized by very diluted foraminiferal content 227 and/or very poor preservation. Among planktonic foraminifera, all the Globigerina, 228 Globigerinoides and Globigerinita species were respectively clustered as genera; however, 229 Globigerina bulloides is largely predominant within the Globigerina gr., and Globigerinita 230 glutinata within the Globigerinita gr. Sinistrally and dextrally coiled Neogloboquadrina acostaensis were counted separately. Bolivinids, buliminds and elphidiids were considered 231 232 as generic groups among benthic foraminifera. A group of taxa showing low abundances 233 and including Hanzawaia boueana, Cancris oblungus, Valvulineria complanata, Rosalina 234 globularis and Ammonia sp. were lumped together as "inner shelf taxa". Each selected 235 taxon was picked and counted at maximum in 9 fields (out of 45) of a standard picking tray 236 if not exceeding 30 specimens. The total abundance of planktonic and benthic foraminifera

and the abundance of the selected taxa were then normalized to one field and plotted against the stratigraphic height. We also calculated the P/B ratio as P\*(P+B)/100, where P and B represent the total number of planktonic foraminifera and benthic foraminifera per field, respectively.

241

# 242 3.3. Calcareous nannofossils

Calcareous nannofossil assemblages from 60 out of 87 samples collected in the Tokhni 243 244 composite section within PK-A<sub>3</sub> and PK-B sub-units (8 - 36.5 m; Figs. 2, and 5) were 245 studied in smear slides with a microscope at 1250X under cross polarized light microscope. 246 Slide preparation was kept simple using standard techniques in order to retain the original composition of the sediment (Bown and Young 1998). Only the uppermost samples in the 247 248 studied interval were barren of calcareous nannofossils. Quantitative analyses were 249 carried out by counting at least 500 specimens per sample and relative abundances are 250 expressed in % of the total assemblage. Abundance percentages of significant taxa were 251 plotted against their stratigraphic level. Frequencies of very rare and biostratigraphically 252 significant genera (Amaurolithus, Discoaster) were estimated by counting the number of 253 specimens in 500 fields of view, corresponding to an area of 11.250 mm<sup>2</sup>. Abundances of 254 these taxa are expressed in n/mm<sup>2</sup>. *Discoaster* spp. were identified at the species level, nevertheless, due to the very low abundance of *D. surculus*, we lumped *D. surculus* and *D.* 255 256 variabilis into the Discoaster variabilis group.

257

258 3.4. Isotopic geochemistry

259 3.4.1. Oxygen and Carbon stable isotopes

Stable oxygen and carbon isotope composition of 79 bulk samples were measured in the Geochemistry Laboratory of the Physical and Earth Science Department of the University of Parma. The isotopic composition of calcite samples (originating from micritic crystals,

foraminifera and calcareous nannofossils) was obtained from the reaction of the powdered 263 carbonate with 100% H<sub>3</sub>PO<sub>4</sub> "in vacuo" and under controlled temperature (25°C). The 264 isotopic composition of CO<sub>2</sub> samples was measured on a Finnigan Delta S mass 265 266 spectrometer against a CO<sub>2</sub> standard gas obtained by the reaction at 25°C of very pure Carrara marble powder with 100% phosphoric acid. The standard deviation of these 267 measurements was systematically equal to or lower than  $\pm 0.15 \ \text{\%} (1\sigma)$ . The CO<sub>2</sub> standard 268 269 from the Carrara marble used in our laboratory is periodically calibrated against NBS-19. Its isotopic composition is -2.43 % ( $\delta^{18}$ O vs. VPDB) and +2.45 % ( $\delta^{13}$ C vs. VPDB). 270

271

### 272 3.4.2. Strontium isotopes

273 Samples were collected from every prominent limestone bed from 10 m upwards of the composite section and <sup>87</sup>Sr/<sup>86</sup>Sr isotope analyses were carried out at SUERC (Scottish 274 275 Universities Environmental Research Centre, East Kilbride, Scotland). Samples were 276 leached in 1M ammonium acetate prior to acid digestion with HNO<sub>3</sub>. Sr was separated using Eichrom Sr Spec resin. Matrix elements were eluted in 8M HNO<sub>3</sub> and 3M HNO<sub>3</sub> 277 278 before elution of Sr in 0.01M HNO<sub>3</sub>. Total procedure blank for Sr samples prepared using 279 this method was <200 pg. In preparation for mass spectrometry, Sr samples were loaded 280 onto single Re filaments with a Ta-activator. Sr samples were analyzed with a VG Sector 281 54-30 multiple collector mass spectrometer. A <sup>88</sup>Sr intensity of 1V (1 x 10 - 11 A)  $\pm$  10% was maintained. <sup>87</sup>Sr/<sup>86</sup>Sr ratio was corrected for mass fractionation using <sup>86</sup>Sr/<sup>88</sup>Sr = 282 0.1194 and an exponential law. The mass spectrometer was operated in the peak-jumping 283 mode with data collected as 15 blocks of 10 ratios, which gives an internal uncertainty of 284 <0.000020 (2 S.E.). For this instrument NIST SRM 987 gave 0.710249 ± 0.000008 (1 S.D., 285 286 n = 17) during the course of this study. The 2 standard error internal precision on individual 287 analyses was between 0.000014 and 0.000020 for  $2\sigma$ .

288

289 4. Results

## 4.1. Magnetostratigraphy

The thermal demagnetization paths show that between the NRM and 180°C a normally 291 292 oriented, low temperature component is generally demagnetized. Between 180° and 340° - 560°C a second component can be isolated, displaying both normal and reversed 293 294 polarities and thus interpreted as the characteristic component (ChRM). However, at 340° C the specimens collected between 10 and 19 m still retain 10-20% of the initial NRM, and 295 296 a further increase of temperature usually results in randomly oriented direction and/or a 297 remanence increase. This fact indicates that a potential third component with higher 298 coercitivity also could be present, but not detected in our samples, due to the formation of authigenic magnetic minerals with increasing temperature above 340°C. In the lower half 299 of the sampled subsection, To13b is the only sample showing a certain reversed ChRM 300 301 orientation (Fig. 3a, c). Samples To11 and To13a display reversed inclination, but the 302 declination is respectively east and west oriented (Fig. 3 b). Samples To12 and To10 were 303 discarded because the signal was too weak. In the upper part of the sampled subsection, 304 the demagnetization paths of samples To01, To05 - To08 show the presence of a more 305 stable normal ChRM isolated in a larger temperature range, between 180° and 460° -306 560°C (Fig. 3a). Summarizing, a reversed magnetozone is pinpointed between 11 and 12 307 m; however, its top is not well defined as sample To11, which tentatively represents the 308 uppermost reversed polarity level, is included in an interval of poorly defined polarities, 309 between 12 and 21 m. A normal magnetozone is identified from 21 and 31.5 m.

310

# 311 4.2. Planktonic foraminifera

In sub-unit PK-A<sub>2</sub> and in the lower part of sub-unit PK-A<sub>3</sub>, up to 7 m from the base of the composite section (Fig. 2), preservation varies from poor to moderate and deformation and/or incrustation commonly prevents the classification of foraminifera at the species 315 level. Inorganic grains (mainly terrigenous particles and un-disaggregated marly sediment) 316 are often abundant and regardless of the lithology can be prevalent on the fossil remains. 317 Foraminifera are usually more abundant in the marls than in the reddish shales, and 318 planktonic foraminifera are generally more common than the benthic. A common feature is 319 the presence of reworked Eocene to middle Miocene taxa, probably derived from the 320 erosion of the uplifted carbonate formations (Lefkara and Pakhna formations) widespread on the Island of Cyprus (Robertson et al. 1995). In this interval, Globigerinoides spp. and 321 322 Orbulina universa are generally the most common taxa, among the planktonic foraminifera, followed by neogloboquadrinids (mainly sinistrally coiled), Globigerinita glutinata and 323 Globigerina bulloides. 324

Semi-quantitative analyses, performed from 7 m of the composite section upward (Fig. 4), 325 indicate that the abundance pattern of foraminifera is characterized by the occurrence of 326 327 successive peaks, mainly observed in the reddish shales, and that from 11.5 m upward 328 almost all the prominent limestones are devoid of foraminifera (except at 14.6 m, where O. 329 universa occurs). Abundance of planktonic foraminifera first drops above 22 m, where 330 peaks are less prominent and then, just above the conglomerate bed at 31 m, where 331 foraminifera become scattered (Fig. 4). As shown by the P/B ratio, planktonic are generally 332 much more abundant than benthic foraminifera in the interval from 7 to 15.5 m; upward, 333 the ratio fluctuates with no evident relationship with lithology.

From 7 m upward, planktonic foraminifer assemblages are more oligotypic with respect to the lower part of sub-unit PK-A<sub>3</sub>. The genus *Turborotalita* is rather common, even though discontinuously distributed (Fig. 4). *T. quinqueloba* prevalently occurs in the shales, but it is also present in the thin limestone bed at 10 m and in the marl at 23 m. It also shows few prominent abundance peaks, reaching up to 100 specimens/field in a monospecific assemblage at 10 m and at 26.5 m. *T. multiloba* first occurs (FO) at 10.2 m and is characterized by a very scattered distribution and generally low abundance in the shales,

while it is quite common (up to 20-30 specimens/field) only in the limestone layer at 10.2 m
and in a marly level at 23.25 m (abundance influx, Ai). *T. multiloba* last occurs (LO) at 31
m, just above the conglomerate (Fig. 4).

The *Globigerinita* gr. is relatively common between 8 and 21 m (Fig. 4), attaining abundance maxima at 16 and 21 m; upward, it is very scattered and it disappears above the conglomerate layer at 31m.

The distribution range of the *Globigerina* gr. is similar to that of the *Globigerinita* gr. (Fig. 4); however, it is only abundant in the shale at 9 m and, differently from the latter, it disappears at about 34.5 m, just above the lowermost limestone bed of sub-unit PK-B, i.e. at the stratigraphic level of the last recovery of planktonic foraminifera.

Neogloboquadrina acostaensis is usually rare (< 5 specimens/field) except at 25.5 m (35 specimens/field). The Last regular influx of sinistrally coiled individuals (Lri of Sierro et al. 2001) is recognized at 12.6 m, while at 13.5 m dextrally and sinistrally coiled specimens occur equally (Fig. 4). Dextrally coiled *N. acostaensis* prevails from 15.25 m (first abundant occurrence, FAO, of Sierro et al. 2001) and is quite regularly present up to 19.5 m. This taxon is more scattered upwards, vanishing at about 33.5 m, just below the base of subunit PK-B (Fig. 4).

*Globigerinoides* spp. is subordinated, showing few minor peaks (<20 specimens/field) between 11 and 20 m and disappear above 25.5 m (Fig. 4). *O. universa* is irregularly distributed along the composite section and is abundant only between 13 and 14.5 m (where it is also common in a prominent limestone layer). *Globorotalia scitula* is present with rare individuals at 29.7 m (Gs influx in Fig. 4). It is worth observing that the topmost planktonic foraminifer assemblage is represented by *O. universa*, *N. acostaensis* and *Globigerina* gr. and the last recovery of planktonic foraminifera (LRp) occurs at 34.5 m.

365

366 4.3 Benthic foraminifera

In sub-unit PK-A<sub>2</sub> and the lower part of sub-unit PK-A<sub>3</sub> up to 7 m of the composite section,
the presence of benthic foraminifera is generally subordinate to planktonic ones and
mainly represented by *Elphidium* spp, *Neoconorbina orbicularis*, *Rosalina globularis*, *Valvulineria complanata*, *Melonis* sp., *Gyroidinoides* sp., *Cibicides* spp., *Cibicidoides* spp., *Bolivina* gr. (mainly *B. spathulata* and *B. dilatata*), *Bulimina* gr. (mainly *B. aculeata*, *B.*echinata and *B. elongata*), *Hanzawaia boueana*, *Cancris oblungus* and *Uvigerina*bononiensis.

374 From 9 m of the Tokhni composite section upward, the benthic assemblages become prevalently dominated by the Bolivina gr. (mainly B. dilatata and B. spathulata), which is 375 particularly abundant in the 15.5 - 28 m interval, where it peaks in the shales in 376 correspondence to the P/B minima (Fig. 4). Benthic foraminifera are absent in the 377 378 limestone beds from 11 m upward, and scattered or absent in some shale levels. A 379 marked abundance decrease is observed from 28.5 m up to 35.2 m, where the last 380 recovery of benthic foraminifera (LRb), represented by the Bolivina gr., is recorded. In the 381 benthic abundance peaks, the Bolivina gr. is commonly associated with subordinated 382 buliminids, Elphidium spp. and with the inner shelf taxa group (H. boueana, C. oblungus, V. 383 complanata, R. globularis and, rarely, Ammonia sp.). These generally subordinated taxa 384 represent major components of the assemblages just in two samples, at 18 and 25 m (Fig. 385 4).

386

### 387 4.4. Calcareous nannofossils

Calcareous nannofossil assemblages in the upper part of sub-unit PK-A<sub>3</sub> and sub-unit PK-B, from 7 m up to the top of the composite section, show moderate to poor preservation. In general, preservation within the micritic limestone layers is poorer than in the shale intervals. Calcareous nannofossils are common to abundant along the section but decrease in abundance at 28.3 m and at 34 m (Fig. 5); however they are still present up to

the uppermost sample at 36.5 m. Reworking of calcareous nannofossils from sediments as old as Early Cretaceous occurs sporadically and mainly in the reddish shales of each cycle. However, abundance of reworked specimens is very low and does not hinder the quantitative analysis of the assemblages. Diatoms were also detected in the smear slides prepared for calcareous nannofossil analyses and occur, with discontinuous abundances, from 12.2 m upward; remarkably, limestones were devoid of diatoms (R. Jordan, pers. comm., 2014).

400 Major component of the fossil assemblages along the entire section are Reticulofenestrids 401 (Reticulofenestra minuta, R. haqii, R. pseudoumbilicus, which represent up to 80% of the total assemblage), together with Sphenolithus abies (up to 80%), Calcidiscus leptoporus 402 403 (up to 70%) and *U. jafari* (up to 45%) (Fig. 5). *R. minuta* is present in both the limestones 404 and in the middle/upper part of the shales; however, in the latter it retains the abundance 405 maxima. Differently, S. abies is virtually absent in the limestones and generally shows 406 greater abundance in the lower part of the shales. The greater abundance of Cd. 407 leptoporus occurs in the limestone layers up to 29.25 m; above this level its abundance 408 drops to less than 5% and it is replaced by Reticulofenestra antarctica within the 409 limestones up to 32.5 m. U. jafari is prevalently present with abundance peaks in the 410 limestones up to 31 m, where it disappears.

*U. rotula* is a minor component (with peak close to 18%) of the assemblage up to 34 m, but it reaches 90% in the abundance peak (Ap) between limestones A and B of PK-B subunit (35 m) (Fig. 5). *Helicosphaera carteri* shows low abundances up to 26 m, reaching 15% of the total assemblage at 34.5 m. *Discoaster* spp. occurs from the bottom of the section and the *Discoaster variabilis* gr. (< 5%) is quite regularly present in three intervals: 10 - 12 m, 18 - 21 m and 25.5 - 29.5 m. It is prevalent in the shales, but it also occurs in the lower part of the limestones at 11.5 m and at 26 m. *Braarudosphaera bigelowii* shows

an abundance peak (130 n/mm<sup>2</sup>) at 25.8 m (Fig. 5), being otherwise absent below and
above this layer.

420 The biostratigraphically useful taxa *Amaurolithus delicatus* and *A. primus* are generally 421 very rare and shows very scattered occurrences.

422

423 4.4. Isotopic geochemistry ( $\delta^{18}O$ ,  $\delta^{13}C$  and  ${}^{87}Sr/{}^{86}Sr$ )

424 Oxygen isotope values (Fig. 6) range from -4.57‰ to 2.49‰ (PDB), showing a cyclic 425 stacking pattern with heavier values in the limestones and lighter values in the reddish 426 shales and pink diatomites. The heaviest values are observed in the limestones below 9 427 m; a shift towards more depleted values is recorded between 9 and 18.5 m, both in the 428 limestones and in the shales. From 18.5 up to 34 m (base of sub-unit PK-B) positive and negative values of  $\delta^{18}$ O guite regularly match with the limestones and shales, respectively, 429 430 however, relative maxima close to zero are documented between 26.5 and 28.5 m. At 34 m a sudden drop towards the most negative values of the succession is observed; minima 431 occur in the grey and pink shales, while the uppermost three carbonates show values 432 433 close to zero. Carbon isotope values are prevalently negative, ranging from -3.19‰ to -434 0.5‰ (PDB), without any evident relationship with lithology (Fig. 6). Just one positive peak 435 at 0.51‰ was measured in the lowermost limestone of the sub-unit PK-B. However, a 436 covariance is observed in the long-term trends between the oxygen and the carbon 437 curves: an increase from the bottom to 4.5 m is followed by an overall decrease down to a 438 minimum at 16 m. Upward an increasing trend is recorded up to 22 m, where a relative maximum of -0.74‰ occurs. Further up, values range between -2 to -1‰ and are 439 punctuated by a positive peak corresponding to the laminated portion of the lowermost 440 441 carbonate of sub-unit PK-B. As the lowermost carbonate of sub-unit PK-B is laminated, we measured the isotope ratios for two white and brown consecutive laminas. Remarkably, 442 the isotopic signatures of these two laminae are different, as we obtained  $\partial^{18}O$  and  $\partial^{13}C$ 443

values of -1.31‰ and -0.58‰ for the white and of 0.17 ‰ and 0.51 ‰ for the brown
lamina, respectively.

The <sup>87</sup>Sr/<sup>86</sup>Sr isotopic ratio is generally slightly greater than that of the oceanic mean values (Mc Arthur et al. 2001) in sub-unit PK-A<sub>3</sub> (Fig. 6), except for samples at 20.25, 24.5 and 32.5 m. Conversely, in sub-unit PK-B the isotopic ratio gradually decreases below the oceanic values, showing two minima in the clastic limestones B and C.

450

451 5. Discussion

452 5.1. Biomagnetostratigraphy

453 In terms of planktonic foraminiferal biostratigraphy, the FO of *T. multiloba* (dated at 6.415) 454 Ma in the Perales section by Sierro et al. 2001) at 10.2 m, the Lri of *N. acostaensis* with predominant sinistral coiling (6.378 Ma; Sierro et al. 2001) at 12.6 m, the FAO of dextrally 455 456 coiled *N. acostaensis* (6.339 Ma; Sierro et al. 2001) at 15.25 m allow us to assign the middle and upper part of PK-A<sub>3</sub> sub-unit to the Messinian biozones MMi13b and MMi13c 457 458 of laccarino et al. (2007). The MMi13b/MMi13c subzonal boundary is defined by the coiling 459 change of *N. acostaensis* between 12.6 and 15.25 m. The succession of these events also indicates that discontinuous reversed polarity samples recorded between 11 and 12 m and 460 at 17 m are referable to the C3An.1r sub-chron (Sierro et al. 2001). In the upper part of the 461 462 composite section, the influx of G. scitula (29.7 m) and the LO of T. multiloba (31 m) occur within a normal polarity magnetozone. The same succession of events is observed in the 463 Perales section, where they occur within sub-chron C3An.1n and are respectively dated at 464 465 6.102 and 6.04 Ma (Sierro et al. 2001). Thus, the influx of G. scitula at 29.7 m can be correlated to its 2<sup>nd</sup> influx recognized in the Perales section (Sierro et al. 2001) and the 466 467 normal magnetozone between 22 and 31.5 m is correlated to sub-chron C3An.1n. The LR of planktonic and benthic foraminifera between limestones A and B in the sub-unit PK-B 468 marks the base of the Non-Distinctive Zone (NDZ) of laccarino et al. (2007) and are 469

470 considered as a good approximation of the MSC onset. In fact, the LR of planktonic (LRp) 471 and benthic (LRb) foraminifera (> 125  $\mu$ m) have been recognized in the penultimate cycle 472 (Perales, Falconara, Legnagnone) and in the last cycle of the pre-MSC unit (Perales, 473 Legnagnone), respectively (Blanc-Valleron et al. 2002, Gennari et al. 2013, Manzi et al. 474 2011, 2013, Sierro et al. 2003). In the Pollenzo section the LRp and the LRb (>125 $\mu$ m) 475 have been recognized in the 1<sup>st</sup> and 2<sup>nd</sup> PLG cycles, respectively (Violanti et al. 2013), but 476 a strong decrease in the foraminiferal abundance marks the onset of the MSC.

477 Regarding calcareous nannofossils, the occurrence of *A. delicatus* and *A. primus* from the 478 bottom of the composite section, albeit scattered, indicates the presence of the MNN11b/c biozones (Raffi et al. 2003). In addition, recently, calcareous nannofossil bioevents have 479 480 been identified associated with the onset of the MSC in the Northern Apennine (Manzi et 481 al. 2007, Dela Pierre et al. 2011, Violanti et al. 2013). For instance, , a sharp abundance 482 peak of S. abies, associated with abundant H. carteri and shortly followed by an abundance peak of *U. rotula* close to 60% of the assemblage, is reported in the Pollenzo 483 484 section (NW Italy) in the sediments deposited during the first Primary Lower Gypsum 485 (PLG) cycle (Lozar et al. 2010 and this volume, Violanti et al. 2013). Remarkably, both at 486 Pollenzo and in the Fanantello borehole (Northern Apennine, Italy; Manzi et al. 2007), S. abies shows low abundances all over the section, with a noticeable 60% abundance peak 487 488 at the base of the first PLG equivalent cycle. In the Tokhni section, on the contrary, this S. 489 abies peak is not unambiguously detectable since this taxon is abundant and regularly 490 occurs in all the shales. However, the occurrence of the sharp increase in abundance of 491 both *H. carteri* and *U. rotula* at 34.5 and 35 m respectively, close to the LR of planktonic 492 and benthic foraminifera, shows their reliability to approximate the onset of the MSC.

493

494 5.2. Cyclostratigraphy

495 The Tokhni section presents a well-defined cyclic lithologic stacking pattern. The cycles of 496 sub-unit PK-A<sub>3</sub> are generally made up of reddish shales and prominent whitish limestones. 497 However, while the shaley hemicycle is a constant feature of the sedimentary cycles along 498 all the composite section, limestones can be associated with or replaced by marls and/or 499 pink diatomites. In the sub-unit PK-B the recognition of sedimentary cycles is complicated 500 by the clastic nature of the limestones A (upper part), B and C. This suggests that they should not be considered equivalent to the limestones of the sub-unit PK-A<sub>3</sub> (see Manzi et 501 502 al. 2016). On the whole, in the Tokhni composite section from 7 m up to the base of the 503 gypsum deposits, we identified 25 cycles (Fig. 7); the thickness of these cycles is rather variable and ranges from 0.5 m (cycle 21) up to 2 m (e.g., cycles 2 and 8). The occurrence 504 of 16 cycles in a time interval of 340 kyr (between the FO of *T. multiloba* and the 2<sup>nd</sup> influx 505 of G. scitula) implies an average duration of 21 kyr for each cycle, suggesting a 506 507 relationship with precession, as previously observed by Orszag-Sperber et al. (2009) 508 Because of the absence of sapropels and the scarcity of diatomites, the sedimentary 509 cycles at Tokhni are more similar to those of the Pissouri section described as an 510 alternation of carbonates (insolation minima) and marls (insolation maxima) (Krijgsman et 511 al. 2002) than to other pre-MSC cycles. In particular, they differ from a) the tripartite cycles 512 of the Falconara and Gavdos sections (Blanc-Valleron et al. 2002, Perez-Folgado et al. 513 2003), where sapropels and marls, intercalated by diatomites are tied to insolation maxima 514 and minima, respectively; b) the quadripartite cycles of Sorbas basin, where the sapropel is correlated to insolation maxima and followed by lower marl, diatomite and upper marl 515 516 (insolation minima) (Sierro et al. 2001, 2003).

517 The cyclicity at Tokhni is also emphasized by fluctuations of the oxygen isotope values 518 and of the abundance of several calcareous nannofossil taxa. In fact, heavier  $\delta^{18}$ O values 519 are associated with limestones and marls, indicating more arid and/or colder conditions, 520 while lighter values are recorded in shales and diatomites, pointing to more humid and/or

521 warmer conditions (Fig. 6). These oscillations were also observed in the bulk sample 522 stable isotope record of Falconara section (Blanc-Valleron et al. 2002), where the heavier and lighter values respectively correspond to marls/diatomites (insolation minima) and to 523 524 sapropels (insolation maxima) (see also Hilgen and Krijgsman 1999). Regarding calcareous nannofossils, shales are characterized by the occurrence of S. abies, and R. 525 *minuta*, (dominant in the lower/middle and upper part of the layer, respectively), both 526 527 sporadically associated with less abundant *D. variabilis* gr (<5%). This assemblage is strikingly similar to that observed in the sapropels of the Sorbas basin (Flores et al. 2005). 528 Thus, the calcareous nannofossil assemblages and the lighter  $\delta^{18}$ O values suggest the 529 530 correlation between the reddish shales of the Tokhni composite section and insolation 531 maxima (Fig. 7).

532 *Cd. leptoporus* and *U. jafari* are the main components of the calcareous nannofossil 533 assemblage in the limestones and marls up to 29.5 m and their co-occurrence was also 534 observed in the upper marls of the Sorbas basin, correlated with insolation minima (Sierro 535 et al. 2003, Flores et al. 2005). Thus, the occurrence of these two taxa and the heavier 536  $\delta^{18}$ O suggest the correlation between the limestones of the Tokhni composite section and 537 insolation minima.

538

### 539 **5.3.** Age model

540 То reconstruct the age model for the Tokhni composite section the 541 biomagnetostratigraphic framework has been used for the correlation to the astronomically 542 calibrated Messinian reference sections of Pollenzo, Perales and Falconara (Fig. 7). The calcareous plankton events have been used as tie points and the sedimentary cycles have 543 544 been correlated to the 65°N summer insolation curve (Laskar et al. 2004), on the basis of the phase relation above exposed (Fig. 7). The resulting age model fits well with the 100 545 546 kyr eccentricity cycle, reflected in the intervals of low and high amplitude variations of the

summer insolation curve, corresponding to eccentricity minima and eccentricity maxima, 547 548 respectively. In particular, the thick limestone of cycle 3 corresponds to the high amplitude insolation cycle at ca. 6.4 Ma; the thick cycles 8, 9 and 10 well correlate with the cluster of 549 550 high amplitude insolation cycles centred at 6.3 Ma; the prominent limestones of cycle 13 551 and 14 well fit with the high amplitude insolation cycle at 6.2 Ma. Indeed, eccentricity 552 minima (6.36, 6.14, 5.97 Ma) can be identified in the sedimentary record for the absence of prominent limestones, replaced by thin marls or pink diatomites (cycles 5, 15-16 and 23; 553 554 Fig. 7). Moreover, the oxygen isotope curve also displays overall lighter values and smoother oscillations in both the 6.36 and 6.14 Ma eccentricity minima. The sedimentary 555 556 response to the 100 kyr eccentricity cycle is a good constraint for the proposed age model; 557 however, an additional tie point could be represented by the influx of *T. multiloba* in the 15 558 - 29.5 m interval, where the C3An.1r/C3An.1n reversal is poorly constrained and standard 559 bioevents are lacking (Fig. 7). This bioevent occurs at the base of the sub-chron C3An.1n, 560 five precessional cycles above the FAO of dextrally coiled *N. acostaensis* and six cycles below the 2<sup>nd</sup> influx of *G. scitula*. In the same stratigraphic position, in the Perales and 561 562 Falconara successions (Sierro et al. 2001, Blanc-Valleron et al. 2002) T. multiloba show a 563 very prominent influx (up to 100% of the assemblage), which marks a paracme end dated at 6.21 Ma, based on its occurrence in the upper part of cycle UA23 of Sierro et al. (2001). 564 565 In the upper part of the composite section, the relative position of the LO of *T. multiloba* with respect to the 2<sup>nd</sup> influx of *G. scitula* (within sub-chron C3An.1n) indicates that an 566 erosion of ca. 2 sedimentary cycles is associated with the base of the conglomerate at 567 568 30.5 m (Fig. 7). In fact, at Tokhni, these events are recorded in two consecutive 569 sedimentary cycles, i.e. below and above the conglomerate, while in the Perales section 570 they are separated by three insolation minima (Sierro et al. 2001).

571 The proposed age model indicates that the onset of the MSC at 5.971 Ma (Manzi et al. 572 2013) occurs between the two lowermost limestones of the sub-unit PK-B (Manzi et al.

573 2016), ca. 4 precessional cycles above the LO of *T. multiloba* and well approximated by 574 the LR of benthic and planktonic foraminifera and by the abundance peaks of *U. rotula* and 575 *H. carteri*. In this interval the sedimentary cyclicity is poorly defined by the thin alternation 576 of shales and diatomites due to the concomitant eccentricity minima.

577 Moreover, the identification of the MSC onset at ca. one precessional cycle below the MES 578 (5.60 Ma, Krijgsman et al. 1999a) implies a hiatus of ca. 350 kyr, due to the erosion of 579 most of the sediments associated with Stage 1 of the MSC.

580

581 5.4. Paleoenvironmental reconstruction

582 5.4.1. Interval 1: pre 6.4 Ma

In the lower part of the section, prior to 6.4 Ma, benthic foraminifer assemblages are 583 584 similar to those recorded in the 7.16 - 6.4 Ma interval of the Pissouri section 585 (Kouwenhoven et al. 2006). They include genera typically representative of an inner shelf 586 to upper bathyal habitats (Murray 2006). The presence of inner shelf genera, such as 587 Elphidium, Neoconorbina, Rosalina and Ammonia, as well as of older foraminifera, is 588 interpreted as due to downslope transport, a process commonly observed in the pre-MSC 589 successions of Crete and Cyprus (Van der Zwaan 1982, Kouwenhoven et al. 2006).. Thus, 590 the diversified benthic foraminifer assemblages and the common occurrence of ostracods 591 indicate an upper bathyal paleoenvironment and the prevalence of an oxygenated sea 592 floor (Boomer and Eisenhauer 2002). Still, the relative high abundances of the Bolivina 593 and Bulimina groups at some levels point to episodes of enhanced nutrient availability and 594 lower oxygen levels (Jorissen 1987, Kahio 1994). The prevalence of *Globigerinoides* spp. 595 and O. universa in the planktonic assemblages of the basal portion of the Tokhni 596 composite section suggests overall warm and oligotrophic conditions in the upper water 597 column (Fig. 8). The occurrence of *T. guingueloba*, *T. multiloba* and of *G. bulloides* from

598 cycle 2 (6.44 Ma) upwards documents the establishment of more eutrophic conditions599 (Sierro et al. 2003).

600

601 5.4.2. Interval 2: 6.4 - 6.1 Ma

602 From 6.4 Ma, the shales are characterized by the dominance of the opportunistic Bolivina 603 (mainly *B. spathulata* and *B. dilatata*) and, subordinately, *Bulimina* groups (mainly *B.* echinata and B. aculeata), which co-occur with inner shelf benthic foraminifera and with 604 605 abundant sponge spicules (Fig. 8), both considered the result of downslope transport. The 606 autochthonous fauna indicates bottom waters characterized by high organic matter supply, 607 low oxygen levels and, according to Kouwenhoven et al. (2006), also by increased salinity. The disappearance of ostracods also points to substantial oxygen decrease at the sea 608 609 bottom, whereas the abundance of sponge spicules either reveals increased availability or 610 optimal preservation of silica. These changes occurred synchronously in the adjacent 611 Pissouri basin and we infer that they are related to the establishment of stressed condition 612 at the sea floor (Kouwenhoven et al., 2006), rather than a shallowing upward trend (see 613 also Orszag-Sperber et al. 2009).

This interval in the Mediterranean is usually characterized by increased oscillations of the 614 P/B ratio (0 - 100%) indicating the establishment of precession-paced anoxic bottom water 615 616 conditions (insolation maxima) and stressed surface waters (hypertrophic/high salinity) 617 during insolation minima (Blanc-Valleron et al. 2002, Sierro et al. 2003; Kouwenhoven et al. 2006). At Tokhni, as well, we observe an increased response to the insolation index, which 618 619 involves marked micropaleontologic differences between the shales (insolation maxima) 620 and the limestones (insolation minima). However, a clear relation between assemblage 621 composition and lithology is only shown by calcareous nannofossils, as foraminifera are 622 generally only present in the shales and do not show a regular distribution pattern.

623 The shaley hemicycles record the association of the aforementioned opportunistic benthic foraminifera with S. abies, D. variabilis gr., R. minuta and with lighter  $\partial^{18}$ O values. In the 624 625 Atlantic Ocean Pliocene record and in the Messinian sapropels of the Sorbas basin, Discoaster spp. and S. abies positively respond to low nutrient availability in the lower 626 627 photic zone (LPZ) and to warm sea surface temperatures (Gibbs et al. 2004a, Flores et al. 628 2005). R. minuta is an opportunistic taxon tolerating wide ecological changes and 629 blooming in nutrient-rich surface waters (Aubry 1992, Flores et al. 1995); in particular, the 630 increase of small sized Reticulofenestra has been correlated with increased riverine input in the Caribbean Pliocene record (Kameo 2002) and, associated with a decrease of 631 Discoaster, has been related to meso- to eutrophic condition in the Late Miocene Pacific 632 633 record (Imai et al. 2015). In the lower/middle part of the Tokhni shales the occurrence of R. 634 minuta associated with dominant S. abies and rare specimens of the D. variabilis gr. 635 suggests that at times of insolation maxima *R. minuta* could thrive in the upper photic zone. 636 where high nutrient levels were mainly provided through increased continental runoff, which also favoured the stratification of the water column. More oligotrophic condition 637 638 prevailed in the LPZ favouring the proliferation of S. abies and, to a minor extent, of D. 639 variabilis gr. In the upper part of the shales, R. minuta becomes dominant (40-60%), 640 generally following a drop in abundance of *S. abies*. Similar to the Sorbas basin (Flores et 641 al. 2005), this could indicate increasing nutrient availability in the surface layer immediately after the insolation maxima, when the transition to a more arid condition, typically 642 established during insolation minima, favoured the onset of deep/intermediate water 643 ventilation. 644

Therefore, by analogy with the formation of the Late Miocene and Plio-Pleistocene sapropels (Nijenhuis et al. 1996), we argue that continental runoff increased during insolation maxima, triggering the stratification of the water column and raising nutrient and silica levels. The oxygen consumption at the sea floor was favoured by the elevated

649 organic matter levels supplied directly from the continent and from the primary productivity in the photic zone (Fig. 8). The combination of these processes favoured the formation of 650 dysoxic or anoxic bottom waters, as indicated by abundance peaks of the Bolivina and 651 Bulimina groups and levels with scarce or absent benthic foraminifera. Differently, benthic 652 foraminifera are completely absent in sapropels deposited after 6.4 Ma in the Western 653 654 Mediterranean Perales section, indicating the establishment of more permanent sea floor anoxia in response to a stronger density stratification of the water column at times of 655 656 insolation maxima (Sierro et al. 2003).

In the marls and limestones of this interval *Cd. leptoporus* occurs with *U. jafari*, while, remarkably, foraminifera are generally absent. The possibility that dissolution had altered the micropaleontologic record of the limestones is regarded as unlikely, since the cooccurrence of the very resistant *Cd. leptoporus* (McIntyre and McIntyre 1971, Dittert et al. 1999, Ziveri et al. 2007) with the very delicate *U. jafari* (Gibbs et al. 2004b), recorded in abundances higher than 30%, is taken as indication of minor diagenetic effects on the assemblage.

664 Extant Cd. leptoporus has broad ecological preferences and opportunistic behaviour (Renaud et al. 2002, Boeckel et al. 2006, Baumann et al. 2016). In fact, it is related to 665 warm/temperate waters with higher nutrient input to the upper photic zone and a shallower 666 667 nutricline, possibly driven by effective mixing due to upwelling (Ziveri et al. 2004, Ausin et al. 2015). Cd. leptoporus is abundant and associated with small placoliths (i.e. small 668 Reticulofenestrids) in the basal homogeneous marls of each cycle of the pre-MSC Serra 669 670 Pirciata and Torrente Vaccarizzo sections (Caltanissetta Basin, Sicily; Bellanca et al. 671 2002), where it co-occurs with T. quinqueloba and N. acostaensis, indicating cooler and 672 eutrophic waters. At Sorbas, U. jafari can be abundant in the upper marls (insolation 673 minima) and is inferred to tolerate high salinity (Flores et al. 2005).

The presence of these taxa together with the heavier  $\partial^{18}$ O values suggest that during 674 insolation minima the higher evaporation rate increased the sea surface salinity, favouring 675 the mixing of the water mass, the formation of deep/intermediate waters and triggering 676 677 eutrophic conditions in the upper part of the water column. This mechanism is similar to that allowing the formation of the Levantine Intermediate (and Deep) Water, which, at 678 679 present, originates in winter in the Cyprus-Rhodes Area and resides between 150 and 600 m in the Eastern Mediterranean (Rohling et al. 2015). Notably, the absence of *U. jafari* and 680 681 the lighter  $\partial^{18}$ O in the intervals correlated to eccentricity minima (cycles 6-7, 11 and 15-17) 682 could indicate that the lowered seasonality decreased the formation rate of highly saline superficial waters. Note, however, that the relatively lighter  $\partial^{18}$ O maxima measured in the 683 684 limestones of these intervals may be also related to the abundance of Cd. leptoporus in these layers, as this species is known to calcify at negative disequilibrium with the original 685 686 sea water (Dudley and Nelson, 1989, Ziveri et al. 2003, Hermoso et al. 2014).

687 The absence of benthic and planktonic foraminifera in almost all limestones of this interval 688 represents a peculiarity of the Tokhni section with respect to the Mediterranean pre-MSC 689 successions. In the adjacent Pissouri basin a-planktonic layers occur; however, their origin 690 and relation with the insolation index remains unclear (Kouwenhoven et al. 2006). In the 691 coeval portion of the Sorbas basin planktonic foraminifera solely can be absent or strongly 692 reduced during insolation minima, while benthic foraminifera show abundance maxima 693 (Sierro et al. 2003). The authors speculated that the absence of planktonic foraminifera could be explained by the highly eutrophic, relatively toxic waters supplied to the photic 694 695 zone during phase of intense mixing. This explanation could hold true for the Tokhni 696 limestones; however the higher abundances of *U. jafari* and the heavier  $\partial^{18}$ O values also 697 suggest that highly saline waters formed in the superficial layer could have prevented the 698 proliferation of planktonic foraminifera. The advection of the newly formed highly saline 699 waters to the sea floor, could have created unfit conditions for benthic foraminifera, too.

700

## 701 5.4.3. Interval 3: 6.1 - MSC onset

702 After 6.1 Ma planktonic and benthic foraminifera show a drop in abundance also in the 703 shales, indicating increasingly stressed environmental conditions. However, the benthic 704 assemblage composition remains the same and, together with the presence of shelf to 705 bathyal cuspidariids bivalves in the sub-unit PK-B (Manzi et al. 2016), indicates that no 706 remarkable paleodepth variation occurred in the proximity of the MSC onset, similarly to 707 the Pissouri basin (Kouwenhoven et al. 2006). Conversely, calcareous nannofossil 708 assemblages record important variations (Fig. 8). The D. variabilis group, P. japonica, U. jafari, and Cd. leptoporus vanish, while R. antarctica, H. carteri and U. rotula show a 709 710 remarkable abundance increase.

711 In this interval, the shales are still characterized by S. abies and R. minuta but not D. 712 variabilis gr. This assemblage could suggest a shallower nutricline (being *Discoaster* spp. 713 adapted to the lower photic zone, like the extant *Florisphaera profunda*; Flores et al. 2005, 714 Stoll et al. 2007) and a moderate nutrient availability. Strong changes in sea surface 715 salinity are also envisaged, since S. abies has been reported to be common to abundant 716 between the gypsum beds of Stage 3 of Roveri et al. (2009), suggesting that it could be an 717 euryhaline taxon (Polemi basin, Cyprus; Wade and Bown 2006). The continuous cyclic abundance of S. abies after 6.1 Ma confirms that the latter taxon shares only some 718 719 ecological preferences with *D. variabilis* gr.

*Cd. leptoporus* and *U. jafari* are replaced in the limestone beds by *R. antarctica*, a *Reticulofenestra* species characterized by closed central area. Such Reticulofenestrids are also documented by Krhovsky et al. (1992) from diatom-rich Oligocene sediments from the Czech Republic (Uhercice Fm. and Dynow Marlstone), where they co-occur with fresh to brackish water diatom species *Melosira* (*Aulacosira*) indicating that this taxon and its relatives could flourish in eutrophic environments with brackish to normal marine salinity.

726 In the topmost samples (across and above the MSC onset) planktonic foraminifera are nearly absent and calcareous nannofossils assemblages become increasingly dominated 727 728 by S. abies, R. minuta, U. rotula, and to a lesser extent H. carteri. H. carteri is a meso- to eutrophic taxon (Ziveri et al. 2004), reported from eutrophic, hyposaline waters (Giraudeau 729 730 1992, Flores et al. 1997) and estuarine environments (Cachão et al. 2002). This taxon 731 commonly occurs in the clays of the Upper Gypsum unit of Stage 3 of the Polemi basin together with Umbilicosphaera specimens that were interpreted as high salinity taxa 732 733 (Wade and Bown 2006). In the Tokhni section high abundances of U. rotula occur both 734 together and just above the *H. carteri* abundance peak, suggesting on the contrary that Umbilicosphaera spp. could be euryhaline and thrive in abnormal salinity waters. As for R. 735 736 *minuta*, small Reticulofenestrids are also particularly abundant in the sediments deposited 737 at the beginning of the MSC in the Piedmont Basin, testifying their tolerance to stressed 738 (Lozar et al. 2010, Violanti et al. 2013) and nearshore environments (Perch-Nielsen 1985), characterized by eutrophic surface waters and variable salinity. The association of U. 739 rotula and *R. minuta* with *H. carteri*, the distinct negative  $\partial^{18}$ O values and the decline of the 740 741 <sup>87</sup>Sr/<sup>86</sup>Sr ratio (Fig. 8), which indicates a marked decreased influence of oceanic-derived 742 waters, typical of the Stage 1 deposits (Roveri et al. 2014), suggest a preference of these 743 taxa for lowered salinity. All this evidence indicates that the deposition of the conglomerate, 744 coeval to the 6.1 Ma Mediterranean paleoceanographic step, marked an increasing 745 riverine influence, both during insolation minima and maxima, reaching an acme at the MSC onset. The conglomerate is coeval to the slurry bed deposited in the Pissouri basin 746 747 (Krijgsman et al. 2002, Manzi et al. 2016) and to a similar layer observed in the Fanantello 748 borehole, which marks an increase of the terrigenous input into the Northern Apennine 749 foredeep. Slope instability is also recorded in the Piedmont basin just below the LO of T. 750 multiloba (Lozar et al. 2010, Violanti et al. 2013). These evidences suggest that a tectonic

impulse, likely affecting the Eastern and Northern Mediterranean, could have contributed
to trigger the paleoceanographic changes observed at ca. 6.1 Ma.

753

#### 5.5. Paleohydrologic implication for the *Braarudosphaera bigelowii* peak

Braarudosphaera bigelowii has a very scattered but continuous occurrence along the 755 756 geological record since the Early Cretaceous and shows sporadic blooms as that recorded at 26 m (up to 130 n/mm<sup>2</sup>) in the Tokhni composite section (Fig. 5). It has often been 757 758 related to neritic environments (Bown et al. 2005, Bartol et al. 2008), to nutrient enrichment and/or low salinity waters, and is generally absent in oceanic settings (Peleo-Alampay et al. 759 760 1999, Kelly et al. 2003, Bartol et al. 2008). The affinity of *B. bigelowii* for hypohaline waters is well illustrated by its presence in the Black Sea, where surface waters have an average 761 salinity of 17% to 18%, and by its absence in the high-salinity waters of the Red Sea 762 763 (Bukry 1974). This episodic occurrence suggests a sporadic and transient supply of 764 fresher waters, either from enhanced runoff or from the connection with a brackish/fresh 765 water body. The stratigraphic position of this abundance peak, three cycles below the 2<sup>nd</sup> 766 influx of G. scitula, dated at 6.102 Ma (correlated to cycle UA27 in the Abad composite, 767 Sierro et al. 2001), suggests that these low salinity waters could have been sourced from the paleo-Black sea, during the transient connection of the two basin approximating the 768 769 Meotian/Pontian boundary (Grothe et al. 2014, Vasiliev et al. 2015).

770

771 6. Conclusions

The Tokhni composite section (Psematismenos basin, southern Cyprus Island) is characterized by the precession-paced alternation of red shales (precession minima/insolation maxima) and limestones (precession maxima/insolation minima). The phase relation between sedimentary cycles and precession cycles is based on cyclic variations of calcareous nannofossil taxa and  $\partial^{18}$ O values. The planktonic foraminifer and

777 magnetostratigraphic events set the tuning of the sedimentary cycles to the 65°N summer 778 insolation curve and to the Mediterranean pre-evaporitic reference sections (Sierro et al. 2001, Blanc-Valleron et al. 2002, Violanti et al. 2013) from 6.46 Ma up to the Messinian 779 780 Salinity Crisis onset at 5.97 Ma. As previously observed in other pre-MSC successions, 781 the onset of the crisis is approximated by: 1) the last recovery of planktonic and benthic 782 foraminifera (Manzi et al. 2007, Manzi et al. 2011, Violanti et al. 2013), 2) the peaks of U. rotula and H. carteri (Lozar et al. 2010, Violanti et al. 2013) and 3) an increase of the fresh 783 784 water input (based on <sup>87</sup>Sr/<sup>86</sup>Sr values; Flecker et al. 2002, Roveri et al. 2014). These events are recorded within an interval characterized by microbialitic and clastic carbonates 785 786 (included in the "barre jaune" of Orszag-Sperber et al. 2009), a few meters below the clastic evaporites of the Kalavasos fm. (Manzi et al. 2016). This supports the interpretation 787 788 that the evaporites of Tokhni do not represent the MSC beginning (Manzi et al. 2016), but 789 instead the Stage 2 of Roveri et al. (2009), deposited after 5.60 Ma above the Messinian 790 Erosional Surface.

791 The estimated paleodepth reconstructed for the Psematismenos basin is upper bathyal 792 and did not significantly change from ca. 6.5 Ma up to the MSC onset. Remarkable 793 paleoceanographic changes are documented at ca. 6.4 and 6.1 Ma, coeval to the 794 uppermost major steps of the pre-MSC phase, and interpreted as increasingly stressed 795 surface and bottom water conditions (Blanc-Valleron et al. 2002, Sierro et al. 2003, 796 Kouwenhoven et al. 2006). It is hypothesized that from 6.4 Ma the increased runoff favoured the stratification of the water column and enhanced nutrients levels in bottom and 797 798 surface waters at times of insolation maxima. Compared to the Western Mediterranean, 799 we observe a less severe oxygen reduction at the sea floor during insolation maxima, 800 probably caused by the episodic formation of intermediate/deep waters. During insolation 801 minima, excess evaporation in surface waters stimulated a vigorous mixing and we 802 speculate that the absence of benthic and planktonic foraminifera was due to the elevated

salinity. This indicates more extreme stressed conditions than in the Sorbas basin, where planktonic foraminifera solely are generally reduced in abundance (Sierro et al. 2003). After the deposition of the conglomerate at ca. 6.1 Ma, possibly related to a tectonic pulse, micropaleontologic assemblages were reduced in abundance and diversity in response to enhanced riverine influence. However, we note that a detectable decrease of the connection with the global ocean can be argued based on the reduction of the <sup>87</sup>Sr/<sup>86</sup>Sr values at the MSC onset.

810

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1148 Figure captions

Fig. 1 - A) Generalized geological map of the central and eastern Mediterranean and B) of the south-western portion of the Cyprus Island with the indication of the position of the Tokhni (Orszag-Sperber et al. 2009, Manzi et al. 2016, this study) and Pissouri (Krijgsman et al. 2002, Kouwenohven et al. 2006) sections.

1153

Fig. 2 - Lithological log of the four sub-sections outcropping near the Tokhni village. The dip and the inclination of the strata in the Pakhna and Kalavasos Formations are shown in order to highlight the presence of an unconformity related to the Messinian Erosional Surface (MES). The base of the Tokhni composite section (BTCS) is indicated with an arrow in sub-section To-1. The black bar next to sub-sections To-1, 2 and 3 shows the interval sampled for the quantitative calcareous nannofossil analysis and semi-quantitative foraminifer analysis.

1161

Fig. 3 - Magnetostratigraphy of sub-section To-1. A) Plots of the inclination and declination of the characteristic remanent magnetization (ChRM). B) Stereoplot of the tilt corrected ChRM directions; the relative statistical values are reported for the normal and reverse samples in the upper and lower box, respectively. C) Stereoplots, Zijderveld and demagnetization (M/M<sub>max</sub>) diagrams relative to the thermal demagnetization paths for three selected samples.

1168

Fig. 4 - Plots of the abundance (n° of specimens/field) of the planktonic and benthic foraminifer taxa considered in this study, total abundance of planktonic and benthic foraminifera (black and grey line respectively) and P/B ratio (%). The abundances of sinistrally and dextrally coiled *N. acostaensis* are plotted with increasing values from left to right (black line) at the bottom of the plot and from right to left (grey line) at the top of the

plot, respectively. The asterisks indicate samples not considered for semi-quantitativeanalyses.

1176

Fig. 5 - Plots of the total abundance of calcareous nannofossil (n/mm<sup>2</sup>) and relative abundances (%) of the taxa considered in this study. Note that *B. bigelowii* is only plotted as n/mm<sup>2</sup> and the *D. variabilis* gr. is plotted as n/mm<sup>2</sup> and % because of their scattered occurrences along the section.

1181

Fig. 6 - Plots of the bulk Oxygen and Carbon isotopic values (both measured in shales and limestones) and of the bulk Sr isotopic ratio (only measured in limestones) of the Tokhni composite section.

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1186 Fig. 7 - Biomagnetostratigraphic and cyclostratigraphic correlation of the Tokhni composite section to the Pollenzo (Lozar et al. 2010, Violanti et al. 2013), Perales (modified after 1187 1188 Sierro et al. 2001 and Manzi et al. 2013) and Falconara (modified after Hilgen and 1189 Krijgsman 1999, Blanc-Valleron et al. 2002 and Manzi et al. 2011) sections. The 1190 successions are tuned to the 65°N summer insolation curve (Laskar et al. 2004). 1191 Planktonic foraminifer events are indicated by numbers following the codification of Sierro 1192 et al. (2001) (a letter is added for new bioevents): 12) first occurrence (FO) of *T. multiloba*; 1193 13) Last regular influx (Lri) of *N. acostaensis* sinistral coiling; 14) first abundant occurrence 1194 (FAO) of *N. acostaensis* dextral coiling; 15) 1<sup>st</sup> influx of *G. scitula*; 15a) abundance influx of *T. multiloba*; 16) 1<sup>st</sup> influx of *N. acostaensis* sinistral coiling; 17) 2<sup>nd</sup> influx of *G. scitula*; 18) 1195 2<sup>nd</sup> influx of *N. acostaensis* sinistral coiling; 18a) last occurrence (LO) of *T. multiloba*; 19p) 1196 1197 last recovery (LR) of planktonic foraminifera ; 19b) last recovery (LR) of benthic 1198 foraminifera. Calcareous nannofossil events (Violanti et al., 2013): a) H. carteri peak; b) U. 1199 rotula peak. On the left side of the figure, the boundaries between the pre-MSC stage,

stage 1 and 2 of Roveri et al. (2009) and the MSC chronology are indicated. Note that a
hiatus of ca. 350 kyr is present between stage 1 and 2 due to the erosion associated with
the Messinian erosional surface (MES).

1203

1204 Fig. 8 - Summary of the paleoenvironmental changes recognized in the Tokhni composite 1205 section. The paleoenvironmental interpretation of the chronologically unconstrained 1206 lowermost 11 m of the section relies upon qualitative observations on foraminifera. The 1207 black star beside the insolation curve indicates the position of the influx of B. bigelowii, 1208 suggesting a transient connection with the paleo-Black sea. On the right side of the figure, the boundaries between the pre-MSC stage, stage 1 and 2 of Roveri et al. (2009) and the 1209 1210 MSC chronology are indicated. Note that a hiatus of ca. 350 kyr is present between stage 1211 1 and 2 due to the erosion associated with the Messinian erosional surface (MES).















Figure 6





Figure 8