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1 **Cleaning method impact on the Mg/Ca of three planktonic foraminifer species: a**
2 **downcore study along a depth transect**

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7 **Abstract**

8 We performed a downcore comparison of Mg/Ca ratios obtained using reductive and non-
9 reductive cleaning methods applied to *Globigerinoides ruber*, *Pulleniatina obliquiloculata* and
10 *Globorotalia menardii* picked along two nearby sediment cores retrieved at 2100m and 3875m in
11 the eastern equatorial Indian Ocean. At the shallower site, our results show statistically
12 significant discrepancies in averaged relative Mg/Ca loss induced by the reductive cleaning
13 compared to non-reductive cleaning: *G. menardii* (8.5%) > *G. ruber* (6.2%) > *P. obliquiloculata*
14 (4.4%), suggesting that reductive cleaning impact on relative Mg/Ca loss is species-specific. At
15 the deeper site, differences between the relative Mg/Ca losses for the three species are smaller:
16 *G. menardii* (3.7%), *G. ruber* (4.1%) and *P. obliquiloculata* (6.3%), suggesting the clear impact
17 of *in situ* preservation state on the cleaning related Mg/Ca loss. The Mg/Ca loss in both relative
18 (%) and absolute (mmol/mol) value of species *G. ruber* and *G. menardii* are significantly smaller
19 at the deeper site than the shallower site, implying that these two species which have experienced
20 intense dissolution on the sea-floor are less prone to lose further Mg during the reductive
21 cleaning. *P. obliquiloculata*, however, show non-significant difference in Mg/Ca loss between
22 the two sites, indicating that the cleaning related Mg/Ca loss in this species is somewhat
23 insensitive to the preservation state at our two sites. Our data reveal that Mg/Ca loss results from
24 partial dissolution induced by the reductive step. We conclude that the non-reductive cleaning
25 approach should be preferred for building inter-laboratory homogeneous datasets of Mg/Ca-
26 derived temperatures, whereas the reductive step should be only included when severe oxide
27 coating is observed.

28 **Key words:** Foraminifera; Mg/Ca thermometry; Cleaning protocol; Paleoceanography

29 **1 Introduction**

30 Foraminiferal Mg/Ca ratios are used for reconstructing past seawater temperatures
31 (Anand et al., 2003; Dai et al., 2019; Elderfield et al., 2012; Fan et al., 2018; Jian Xu et al., 2010;
32 Yu & Elderfield, 2008). In order to accurately reconstruct Mg/Ca temperatures, it is crucial to
33 clean foraminiferal tests to remove Mg-rich contaminants. Common Mg contaminants are clay
34 minerals, organic matter and ferromanganese oxides (Barker et al., 2003; Pena et al., 2008). The
35 removal of clay minerals and organic matter are the most important steps in Mg/Ca cleaning
36 (Barker et al., 2003) and are always included in cleaning methods. In order to remove oxides, a
37 reductive step must be added to the cleaning protocol, with the use of a hydrous hydrazine
38 solution mixed with citrate acid and buffered with ammonia (Boyle & Keigwin, 1985; Martin &
39 Lea, 2002; Yu et al., 2007). Benefits and pitfalls of removing oxides with such a chemical
40 treatment have been widely debated over these last years, some studies including this reductive
41 step, whereas others preferring not to (Martin & Lea, 2002; Barker et al., 2003; Yu et al., 2007;
42 Bian & Martin, 2010). Indeed, despite the ammonia buffering, several studies have provided
43 evidence of dissolution of foraminiferal tests due to this reductive cleaning, resulting in a
44 decrease of Mg/Ca ratios compared to those obtained from a cleaning protocol without a
45 reductive step (e.g. Rosenthal et al., 2004; Johnstone et al., 2016). According to several authors,
46 this decrease in Mg/Ca reflects the inhomogeneous distribution of Mg in the tests and the
47 preferential dissolution of Mg-enriched parts during the reductive cleaning (Barker et al., 2003;
48 Yu et al., 2007). However, in some sedimentary environments, not removing contaminant oxides
49 can result in erroneously high Mg/Ca values (Pena et al., 2005, 2008; Weldeab et al., 2006).
50 Paleoceanographers are caught, therefore, between a rock and a hard place on choosing the
51 cleaning protocol (i.e. with or without using a reductive step) and no general consensus has yet
52 emerged. As a result, reductive step is currently employed in some laboratories but omitted in
53 others. This lack of consensus can have significant impacts on paleoceanographic reconstructions
54 because, for instance, building databases of past ocean temperatures requires coherent datasets.
55 Addressing inter-laboratory biases such as the potential impact of cleaning protocol and how to
56 correct for this are thus key issues to homogenize the published Mg/Ca results.

57 Several studies have tried to address quantitatively the effects of reductive cleaning on
58 foraminiferal Mg/Ca but have reached contrasted conclusions. As part of an inter-laboratory
59 comparison exercise including a multi-planktonic species study, Rosenthal et al., (2004)
60 observed an average reduction of ~15% in Mg/Ca ratio obtained from foraminiferal tests cleaned
61 with a reductive step compared to those cleaned without it. Barker et al. (2003) had previously
62 reported a similar 10-15% shift in Mg/Ca between the two cleaning approaches. By studying
63 multi-benthic species, Elderfield et al. (2006) reported an average 0.2 mmol/mol Mg/Ca
64 reduction with a reductive step, and concluded that this value was similar to the 10-15%
65 difference found for planktonic foraminifera. When using a multi-planktonic species calibration
66 for Mg/Ca thermometry (Anand et al., 2003), a 10-15% relative loss is equivalent to 1.1-1.6°C
67 decrease. This amount is rather large considering, for instance, that the glacial-interglacial sea
68 surface temperature change in the tropics could be in the order of 3-4°C (e.g. Linsley et al., 2010).
69 Based on these early studies, a 10% loss is usually used for correcting the cleaning induced
70 Mg/Ca offset. However, following studies on specific planktonic species revealed considerable
71 differences. For instance, Xu et al. (2008) reported much smaller cleaning-induced Mg/Ca
72 differences and suggested that Mg-loss could be species dependent, with a ~6.6% Mg/Ca relative
73 decrease for *G. ruber*, and a smaller ~1.8% loss for *Pulleniatina obliquiloculata*. Fan et al.
74 (2018), on the other hand, recently reported a stronger ~6% Mg/Ca decrease for *P.*

75 *obliquiloculata* when a reductive step was added to the cleaning protocol. To make things even
76 more puzzling, [Johnstone et al. \(2016\)](#) recently reported no Mg/Ca change for *P. obliquiloculata*
77 after reductive cleaning, and relative Mg/Ca losses of ~4% for *G. ruber*, ~10% for
78 *Neogloboquadrina dutertrei* and ~5% for *Globigerinoides sacculifer*.

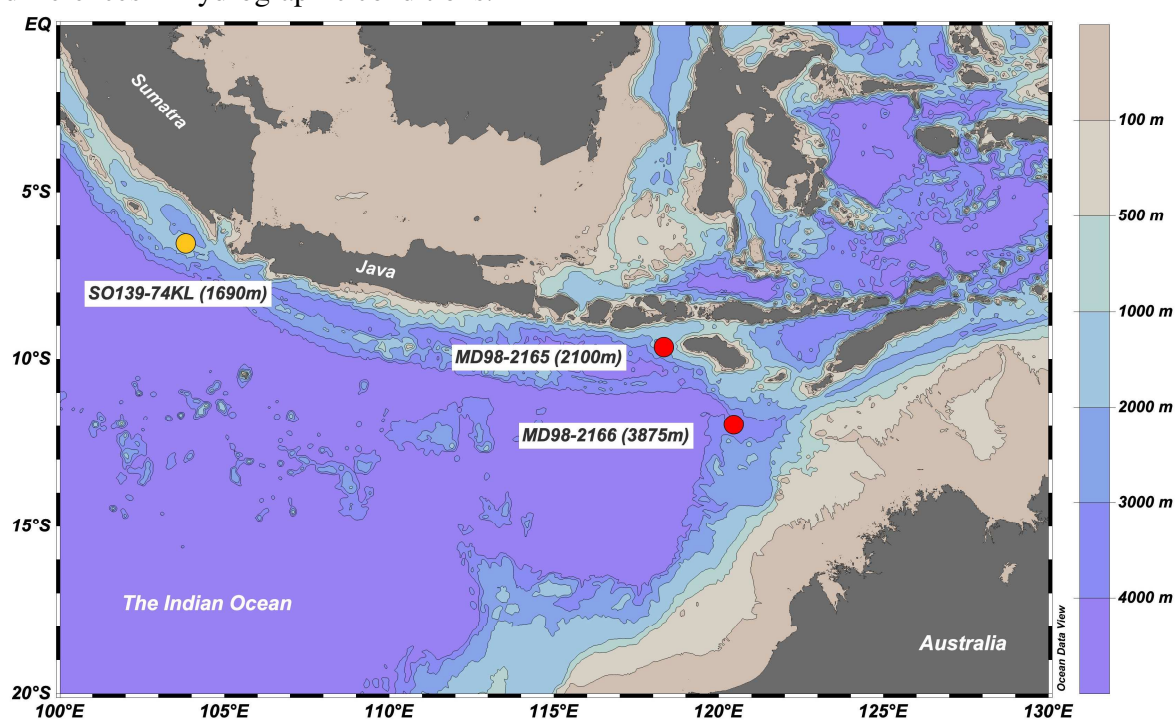
79 As can be seen for these few examples, published data are confusing, fueling a never-
80 ending debate about cleaning methodologies and how to compare or even combine Mg/Ca
81 results obtained from different laboratories. Difficulties in drawing consistent conclusions from
82 previous works come from the fact that no comprehensive study of cleaning effects has yet
83 combined all the following characteristics: (1) the two cleaning protocols should be applied on
84 the same samples, within the same laboratory, to limit other potential sources of bias, (2) the
85 cleaning protocols should be applied to different planktonic species picked from the same
86 samples in order to test for inter-species differences, (3) the potential impact of the preservation
87 state of foraminiferal tests should be addressed by comparing sedimentary material retrieved at
88 different water depths, (4) the study should include a downcore evaluation of Mg/Ca offsets
89 related to cleaning methods. Such a downcore comparison is mandatory if one wants to decipher
90 whether the cleaning offsets may vary through time or depth, along the same sedimentary record,
91 as a result of changes in the abundance of oxides (reflecting past variations in bottom water
92 oxygenation), past changes in calcite preservation conditions at the seafloor, and/or diagenetic
93 effects. This downcore study is particular important on assessing whether a constant correction
94 factor can be applied along a core over different time periods (e.g. through a glacial-interglacial
95 cycle). In addition, downcore studies provide a large number of samples, making it possible to
96 derive more statistically robust evaluations of cleaning effects.

97 The present study aims at unraveling the species and preservation dependencies of Mg/Ca-offsets
98 resulting from using non-reductive and reductive cleaning methods. For this purpose, we picked
99 three planktonic foraminifer species (*G. ruber*, *G. menardii* and *P. obliquiloculata*) exhibiting
100 different sensitivities to dissolution for assessing species-dependent effects. Because the
101 preservation state of foraminifer tests is chiefly related to the water depth of deposition
102 ([Regenberg et al., 2014](#)), we used two cores located in the same area but retrieved at different
103 water depths for addressing the dissolution issues. Although being close from one another, the
104 sites display slightly different hydrographic settings today and past temperature evolution were
105 not identical. Differences in Mg/Ca values between the two sites related to slightly various
106 environmental settings will not be the focus of the present paper, which chiefly deals with
107 cleaning-related Mg/Ca offsets.

108 **2 Materials and Methods**

109 We selected two adjacent sediment cores retrieved from different water depths in the
110 western Timor Sea ([Figure 1](#)): core MD98-2165 (9.65° S, 118.34° E) located at 2100m of water
111 depth, and core MD98-2166 (11.96° S, 120.46° E) located at 3875m of water depth. This choice
112 has been driven by one of our goals in this experiment: to test whether the preservation state of
113 foraminiferal tests exerts an impact on the relative Mg/Ca changes induced by adding a reductive
114 step to the cleaning protocol. The preservation state of planktonic foraminiferal Mg/Ca ratios is
115 controlled by the calcite-saturation state of bottom water ([Regenberg et al., 2014](#)). [Regenberg et](#)
116 [al. \(2014\)](#) reported a global decrease in planktonic foraminiferal Mg/Ca ratios of ~0.054
117 mmol/mol per $\mu\text{mol/kg}$ below a threshold for dissolution of ~21.3 $\mu\text{mol/kg}$. At the two studied
118 sites in this paper, we calculated the calcite-saturation state of bottom waters ($\Delta[\text{CO}_3^{2-}]$) – the

119 difference between in situ $[\text{CO}_3^{2-}]$ and saturation state of $[\text{CO}_3^{2-}]$ – using the CO2Sys software
 120 (Lewis et al., 1998) fueled with total alkalinity, temperature, salinity, pH and nutrient values
 121 obtained from the nearest GLODAP sites (Sabine, et al., 2005). Estimated $\Delta[\text{CO}_3^{2-}]$ is about +10
 122 $\mu\text{mol/kg}$ at the water depth of core MD98-2165 and -11 $\mu\text{mol/kg}$ at the water depth of core
 123 MD98-2166. As expected, the difference between the two sites and the under-saturation at site
 124 MD98-2166 suggest a much stronger dissolution effect on foraminifer tests at the deeper site.
 125 This is in agreement with the higher proportion of foraminifer fragments observed in samples
 126 from the deeper core MD98-2166. To better constrain the impact of the preservation state on the
 127 Mg loss associated to the reductive cleaning step, we will chiefly look at relative cleaning related
 128 Mg/Ca loss in %, and we will also provide the absolute cleaning related differences of Mg/Ca
 129 values in mmol/mol between the two sites. It is out of the scope of this study to distinguish in
 130 what extent the Mg/Ca differences between the two sites reflect in-situ dissolution or/and
 131 differences in hydrographic conditions.



132
 133 **Figure 1.** Locations of sediment cores discussed in this study; MD98-2165 (red dot, this study),
 134 water depth 2100m; MD98-2166 (red dot, this study), water depth 3875m; SO139-74KL (yellow
 135 dot)(Wang et al., 2018), water depth 1690m.

136 For core MD98-2165, we focused on the upper part of the core down to 779 cm. Age
 137 model of the upper part of MD98-2165 was developed by Waelbroeck et al. (2006) based on 20
 138 ^{14}C dates. Two anomalous ^{14}C values (inversion) suggested that the upper 55 cm of the
 139 sedimentary are allochthonous and were deposited rapidly (Waelbroeck et al., 2006). We focused
 140 our study, therefore, on the interval 110-779cm, covering the time interval from ~ 21 ka Cal BP
 141 (Last Glacial Maximum) to the Late Holocene (~2 ka Cal BP). Samples for the present study
 142 were taken every 5 to 10 cm (corresponding to a time resolution average on 300 yr).

143 For core MD98-2166, we studied the upper sedimentary section down to 540 cm. We
 144 sampled the sediment series every 10 cm. The age model for MD98-2166 was developed by

145 [Moreno et al. \(2008\)](#) by correlating the *G. ruber* $\delta^{18}\text{O}$ record of MD98-2166 to the high-
146 resolution (i.e., 300 yr mean resolution) *G. ruber* $\delta^{18}\text{O}$ record of core MD98-2165. This age
147 model indicates that our Mg/Ca records cover the interval from the last glacial period (~35 ka
148 Cal BP) to the middle Holocene (~ 2 ka Cal BP).

149 We picked three planktonic foraminifera species for this study: *G. ruber* (white), *P.*
150 *obliquiloculata* and *G. menardii*. *G. ruber* and *P. obliquiloculata* are two species commonly used
151 in the Indo-Pacific Warm Pool region for reconstructing surface and upper thermocline water
152 temperatures, respectively (e.g. [Xu et al., 2010](#); [Bolliet et al., 2011](#); [Fan et al., 2018](#); [Wang et al.,](#)
153 [2018](#)); *G. menardii* is a deep-dwelling species, which has been used to reconstruct past changes
154 in the thermocline structure ([Regenberg et al., 2010](#)). These three species have different textures
155 and morphological structures. *P. obliquiloculata* is characterized by a glassy, outer crust
156 ([Steinhardt et al., 2015](#)); *G. menardii* displays a large and thick surrounding keel and a dense
157 outer crust ([Regenberg et al., 2010](#)); *G. ruber* has a thin and porous test. The outer crusts (*P.*
158 *obliquiloculata* and *G. menardii*) and the keel of *G. menardii* are characterized by lower Mg/Ca
159 ratios compared to inner layer parts ([Sadekov et al., 2005](#); [Sadekov et al., 2009](#); [Steinhardt et al.,](#)
160 [2015](#)), and are known to be more resistant to dissolution occurring on the sea floor or during the
161 cleaning in laboratory ([Brown & Elderfield, 1996](#); [Johnstone et al., 2016](#)). Because of these
162 characteristics, *P. obliquiloculata* and *G. menardii* are dissolution-resistant species, whereas *G.*
163 *ruber* tests are more easily removed by dissolution in deep marine sediments.

164 For each sample, about 60-80 individuals of *G. ruber* and *P. obliquiloculata* were hand-
165 picked from the 250-355 μm size fraction and about 30 individuals of *G. menardii* were hand-
166 picked from the 355-450 μm size fraction. The samples were gently crushed between two glass
167 plates under a binocular and thoroughly mixed to insure optimum homogeneity of sample
168 aliquots. Samples were then divided as evenly as possible to obtain two sub-samples (Group A
169 and Group B) and transferred to acid-cleaned, 500 μl centrifuge tubes for cleaning.

170 Group A and Group B sub-samples were cleaned following the protocol presented in
171 [Barker et al. \(2003\)](#), and a reductive step was added for samples from Group B. Here is a brief
172 description of cleaning, for further details, please refer to [Barker et al. \(2003\)](#). Samples were
173 ultrasonically cleaned in Milli-Q water (5 times) then in methanol (2 times) for removing clay
174 minerals. Two additional rinses in Milli-Q water were performed for removing any remaining
175 methanol. Samples were then checked under a binocular and any coarse silicates and dark-
176 colored particles were removed before continuing with the oxidative cleaning step. For Group B
177 samples, a reductive step was added prior to the oxidative cleaning. For this reductive step, we
178 followed the protocol initially developed by [Boyle and Keigwin \(1985\)](#) and later used or slightly
179 modified in other studies (eg. [Martin & Lea, 2002](#); [Yu et al., 2007](#); [Brian & Martin, 2010](#)).
180 Samples were put in 0.12M citrate buffered with ammonium hydroxide and hydrazine solution in
181 a boiling water bath for 30 minutes. Samples were ultra-sonicated every 5 minutes for promoting
182 the reaction. Samples were then rinsed twice with Milli-Q water. For the rest of cleaning
183 procedure, samples in Group A and Group B were treated the same way. They were soaked into
184 alkali-buffered 1% H_2O_2 solution in a boiling water bath for 15 minutes (1 time). Every 3
185 minutes, samples were put into an ultrasonic bath for a few seconds for releasing CO_2 bubbles
186 building up during the oxidative reaction. Samples were then rinsed twice with Milli-Q water for
187 removing remaining oxidative reagent. The cleaning protocol ends up with a rapid leaching in a
188 diluted nitric acid solution (HNO_3 0.001M) for removing any adsorbed contaminants, followed

189 by thorough rinsing with Milli-Q water. All the water was removed through pipetting and
190 samples left in the centrifuge tubes.

191 Prior to analysis, each sample was dissolved in 0.075M HNO₃ and then centrifuged for
192 removing any undissolved particulate impurities. The supernatant solution was transferred to a
193 new acid-cleaned, 500 µl centrifuge tube. Mg/Ca ratios were measured by an inductively coupled
194 plasma mass spectrometry (ICP-MS, Analytik Jena PlasmaQuant MS Elite). Aliquots of samples
195 were first analyzed for Ca concentrations and the remaining solutions were diluted to reach a Ca
196 concentration of ~100 ppm in order to minimize possible matrix effects. A standard solution was
197 analyzed every 3 samples to monitor the instrument stability and correct for drifts (Yu et al.,
198 2005). The long-term reproducibility of Mg/Ca measurements estimated from replicate analyses
199 of a standard solution (Mg/Ca=3.69 mmol/mol) is ±1.4% (n=144 1σ relative standard deviation).
200 In each run, the Group A and Group B subsamples of the same initial sample were analyzed
201 sequentially (i.e. A₁B₁, A₂B₂, ..., A_nB_n) for insuring a better inter-comparison of paired-results.
202 By doing this, we aimed at minimizing potential differences in Mg/Ca ratios between paired-
203 subsamples that could result from instrumental instabilities. Together with Mg and Ca, we
204 measured Al and Fe, to check for potential contamination by clay minerals (e.g., Barker et al.,
205 2003), and we measured Mn, to detect the presence of oxide coating.

206 The impact of adding a reductive cleaning step on Element/Ca (e.g. Mn/Ca, Mg/Ca) has
207 been estimated by computing the relative Elements/Ca loss in % compared to non-reductively
208 cleaned paired samples. It is calculated as follow: Element/Ca-loss (%) = (Element/Ca_{without}
209 reductive step – Element/Ca_{with reductive step}) / Element/Ca_{without reductive step} *100. The species-specific
210 Mg/Ca temperature sensitivities of the three species studied in this paper are similar, around 9%
211 increase in Mg/Ca per 1 °C increase (Anand et al., 2003; Regenberget al., 2009). Thus, the
212 cleaning-related Mg/Ca-losses (%) are direct indicators of the impact of adding a reductive step
213 on the temperature reconstructions. In other words, the larger the Mg/Ca-loss (%) is, the larger
214 the temperature difference resulting from the two cleaning methods will be. Together with
215 relative Mg/Ca-losses (%), the absolute cleaning related Mg/Ca offsets in mmol/mol will be also
216 shown in this study. Comparing both relative (%) and absolute (mmol/mol) Mg/Ca-losses on the
217 same species from the two different sites makes it possible to better understand the importance of
218 *in situ* preservation on cleaning-related Mg/Ca differences.

219 3 Results

220 3.1 Contaminants

221 The low levels of Al/Ca (mean value ~ 0.01 mmol.mol⁻¹, maximum values < 0.2
222 mmol.mol⁻¹) indicate that the cleaning procedure successfully removed any clay phase in the
223 foraminifer samples. No relationships exist between higher concentrations of Al/Ca and high
224 Mg/Ca values.

225 Except for several reductively cleaned samples of *G. ruber*, most of Fe/Ca ratios rang
226 between 0.6 and 1.2 mmol.mol⁻¹ for the three species in core MD98-2165 (Figure 2-4). In
227 comparison, Fe/Ca ratios for the three species in core MD98-2166 are no higher than 0.6
228 mmol.mol⁻¹, with showing rather similar variations around a mean value at ~0.4 mmol/mol in all
229 three species (Figure 2-4). Fe/Ca values show little impact from reductive step in core MD98-
230 2165, adding reductive step result in averaged Fe/Ca decrease 7.2% for *G. ruber*, 2.9% for *P.*

231 *obliquiloculata* and 2.5% for *G. menardii*. In comparison, Fe/Ca values of core MD98-2166
232 show negligible impact from reductive step in all three species.

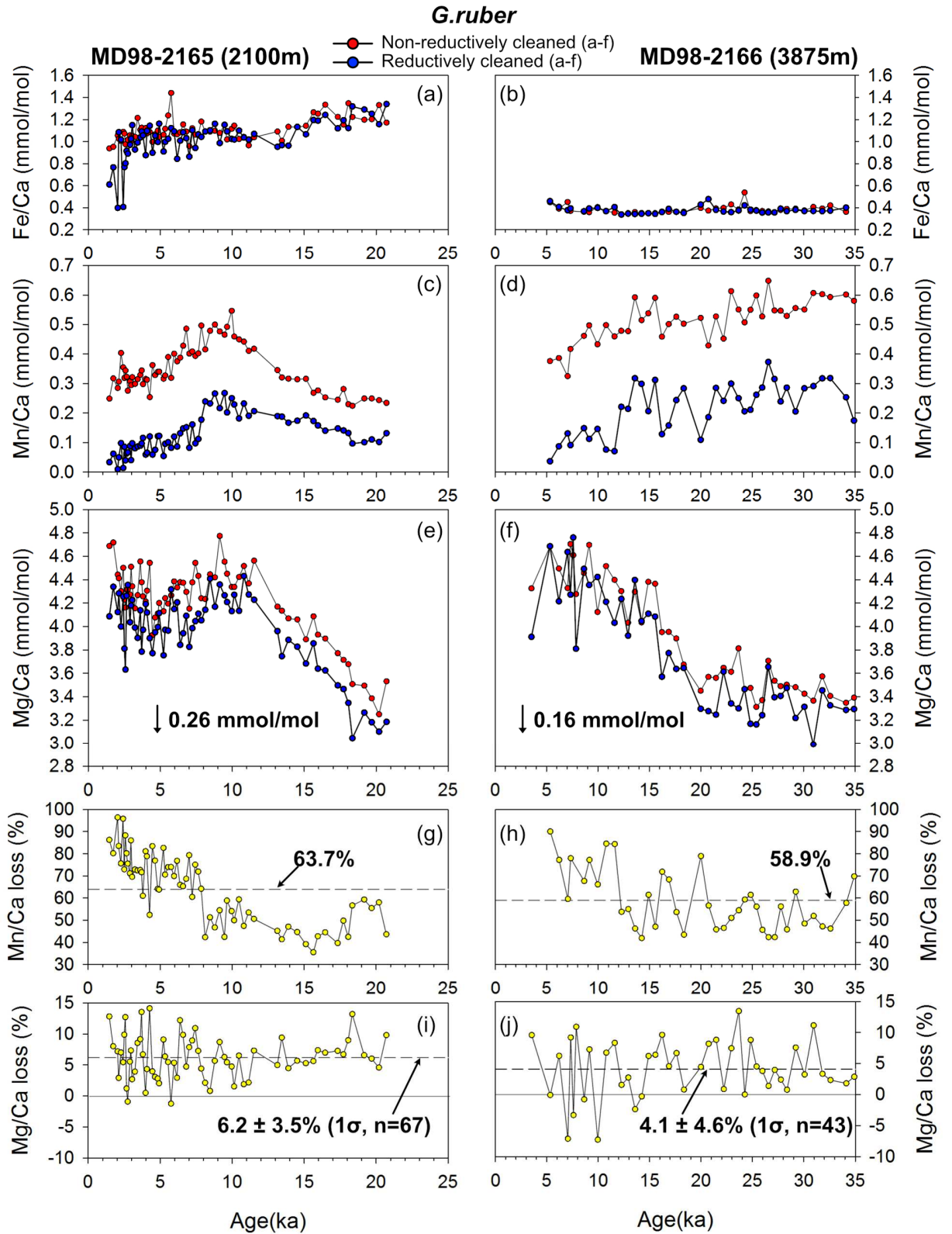
233 Overall, Mn/Ca ratios remain low, ranging from un-detectable up to a maximum value of
234 0.65 mmol/mol (non-reductively cleaned *G. ruber* in core MD98-2166) (Figure 2-4). Mn/Ca
235 values clearly show the impact of reductive cleaning on oxide removal. Adding a reductive step
236 results in a systematic 56 to 66% drop (average values) in Mn/Ca values in B sub-samples
237 compared to the A group aliquots cleaned with the non-reductive protocol (Figure 2-4). For
238 instance, average Mn/Ca in *G. ruber* from core MD98-2166 drops from 0.52 mmol.mol⁻¹ in the
239 non-reductively-cleaned, A-batch, to 0.22 mmol/mol in the reductively cleaned, B-batch (Figure
240 2d).

241 3.2 Mg/Ca results

242 3.2.1 *G. ruber*

243 For *G. ruber*, average Mg/Ca values range between ~ 3.2 and 4.7 mmol/mol (Figure 2e)
244 displaying a clear glacial/interglacial structure in both core MD98-2165 and MD98-2166, with
245 low Mg/Ca values during the glacial period increasing from around 20 ka Cal BP to reach a
246 maximum at the onset of Holocene (~11 ka Cal BP). Core MD98-2165, which has a more
247 complete Holocene section, shows decreasing Mg/Ca values towards a minimum centered
248 around ~ 5 ka Cal BP and then increasing upward (~ 4.5 mmol/mol at the core top). Overall, the
249 *G. ruber* Mg/Ca records from both cores are rather similar in terms of absolute Mg/Ca values
250 and relative glacial/early Holocene evolution. This suggests that sea surface temperatures might
251 have been slightly higher at the site MD98-2166, considering the fact Mg/Ca ratios at this deeper
252 site should have been more reduced due to the stronger *in situ* dissolution (Johnstone et al., 2011;
253 Regenberg et al., 2014).

254 As can be readily seen from Figure 2 (e-f, and i-j), except for a few odd samples, Mg/Ca
255 values obtained with the reductive cleaning protocol (blue curves) are systematically lower in
256 both cores than values obtained without the reductive step (red curves). Mg/Ca values decrease
257 in average by ~0.26 mmol/mol in core MD98-2165 when adding a reductive step, whereas the
258 Mg/Ca drop is only ~0.16 mmol/mol in core MD98-2166 (Figure e-f). This corresponds to a
259 relative Mg/Ca-loss averaging $6.2 \pm 3.5\%$ (1σ , n=67) in core MD98-2165, while it is $4.1 \pm 4.6\%$
260 (1σ , n=43) for the deeper core MD98-2166 (Figure 2 i-j). These results suggest that the reductive
261 step has a stronger impact on Mg/Ca ratios of *G. ruber* from the shallower core. In core MD98-
262 2166, there are a few, limited samples for which the offset is opposite (i.e. higher Mg/Ca values
263 for reductively cleaned samples), but these samples remain scarce and with limited impact on the
264 average Mg/Ca difference. Those odd values could result from the presence of small impurities
265 and/or result from analytical variability. The Mg/Ca differences do not display a clear pattern
266 with age (or depth) in cores MD98-2165 and MD98-2166.



267

268 **Figure 2.** Element/Ca of *G. ruber* from core MD98-2165 (a, c, e) and core MD98-2166 (b, d, f).

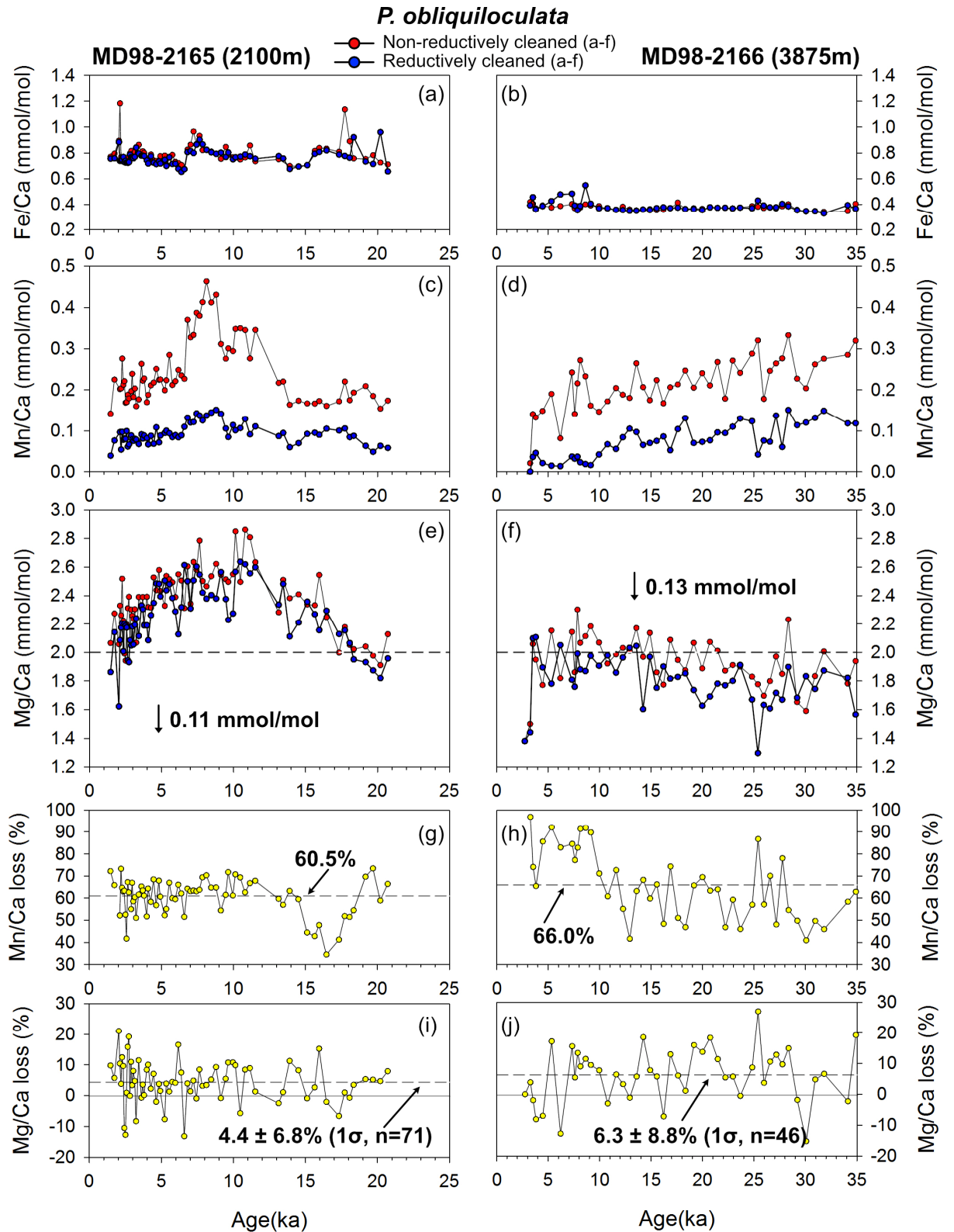
269 In (a)-(f), the red circles shows Element/Ca ratios obtained without the reductive step, the blue

270 circles corresponds to Mg/Ca ratios obtained with the reductive step. In (e) and (f), the numbers
271 on the graphs represent the average Mg/Ca decreases in mmol/mol due to the reductive cleaning.
272 (g)-(j), Mn/Ca and Mg/Ca loss in %; in (g) and (h), the values and the horizontal dashed line
273 indicate the average Mn/Ca-loss in %. In (i) and (j), averaged Mg/Ca-loss (%) \pm 1 sigma
274 uncertainty are displayed, with n the total number of samples; horizontal dashed line indicates
275 the average value, horizontal grey line represents the 0% loss.

276 **3.2.2 *P. obliquiloculata***

277 The *P. obliquiloculata* Mg/Ca record from core MD98-2165 displays a clear deglaciation
278 increase, with glacial values around 2 mmol/mol, increasing to \sim 2.7 mmol/mol at the beginning
279 of the Holocene (Figure 3e). Then, Mg/Ca values decrease upward to reach \sim 2.1 mmol/mol
280 (similar to glacial values) at the core top, with the most rapid drop in Mg/Ca occurring around 5
281 ka Cal BP. While the structure and values of Mg/Ca records were rather similar in both cores for
282 *G. ruber*, the Mg/Ca records from cores MD98-2165 and MD98-2166 cores are significantly
283 different for *P. obliquiloculata*. The deeper core MD98-2166 shows lower values than the
284 shallower core MD98-2165 (Figure 3e-f) and does not reveal a strong glacial/interglacial
285 structure, maximum Holocene Mg/Ca values (around 2 mmol/mol) remaining very similar to
286 glacial values, around 1.8 mmol/mol. Lower Mg/Ca in Core MD98-2166 may partly result from
287 the poorer preservation at this deeper site (Johnstone et al., 2011; Regenberg et al., 2014). We
288 interpret the different time-evolution of the two *P. obliquiloculata* Mg/Ca records as resulting
289 from the regional complexity of the thermocline structure and its specific evolution at the two
290 sites. This contrasts with the coherent evolution of sea-surface conditions at the two sites
291 revealed by the similarity of *G. ruber* Mg/Ca records.

292 As was already observed for *G. ruber*, Mg/Ca ratios obtained on aliquots treated with the
293 reductive step are lower in average than Mg/Ca ratios obtained on aliquots cleaned without the
294 reductive step, with showing an averaged loss of \sim 0.11mmol/mol decrease in core MD98-2165
295 and \sim 0.13mmol/mol decrease in core MD98-2166. The corresponding relative Mg/Ca losses
296 between the two cleaning methods average $4.4 \pm 6.8\%$ (1σ , n=71) for core MD98-2165, and 6.3
297 $\pm 8.8\%$ (1σ , n=46) for the deeper core MD98-2166 (Figure 3i-j). Those differences show,
298 however, a higher degree of scattering for *P.obliquiloculata* than for *G. ruber*. As can be
299 observed in Figure 3, part of that variability rests on a few “odd” samples for which reductively
300 cleaned Mg/Ca are higher than the non-reductively cleaned aliquots. The variation of cleaning
301 related Mg/Ca differences do not display a clear pattern with age (or depth) in cores MD98-2165
302 and MD98-2166 (Figure 3i-j).



303

304 **Figure 3.** Element/Ca of *P. obliquiloculata* from core MD98-2165 (a, c, e) and core MD98-2166

305 (b, d, f). In (a)-(f), the red circles shows Element/Ca ratios obtained without the reductive step,

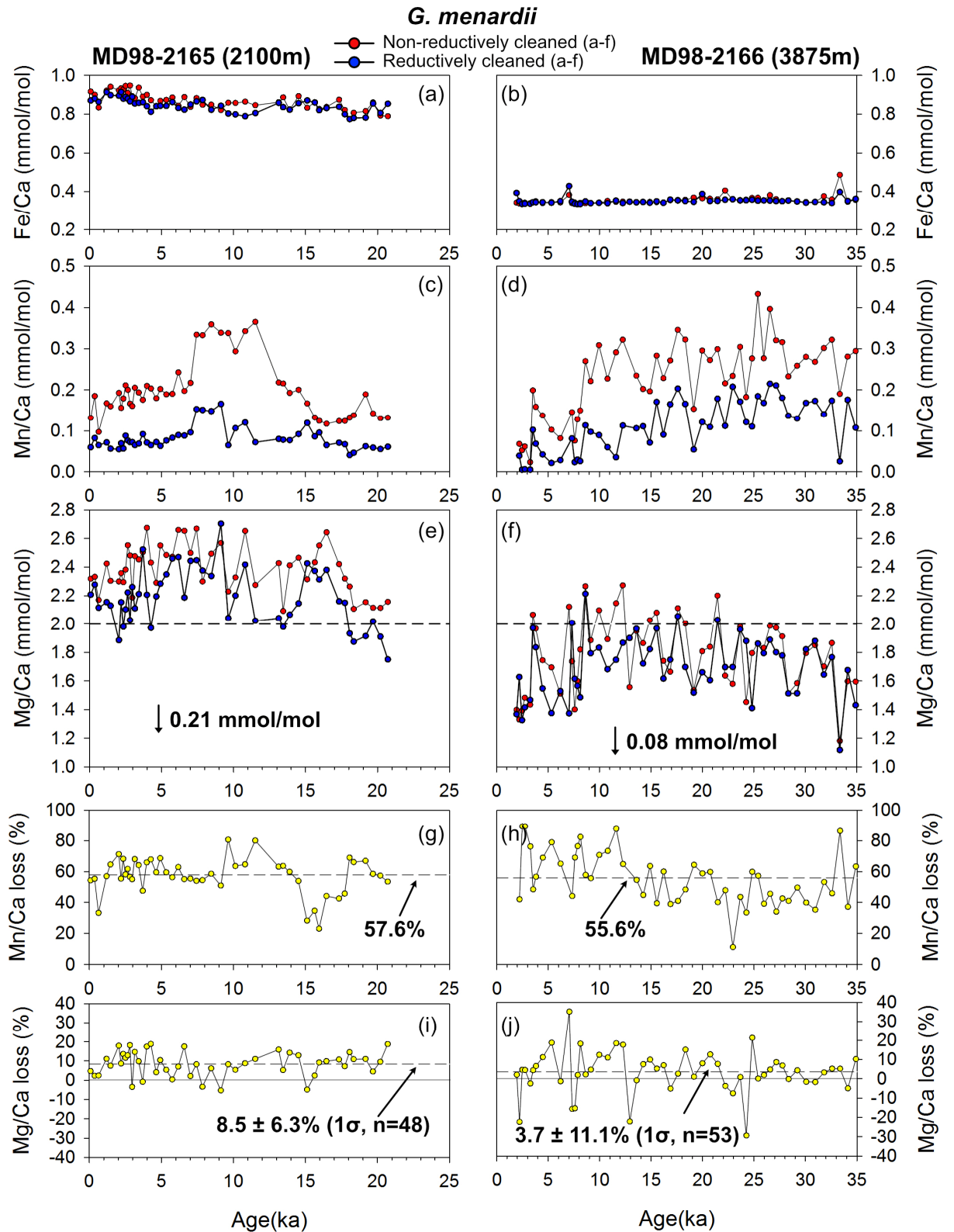
306 the blue circles corresponds to Mg/Ca ratios obtained with the reductive step. In (e) and (f), the
307 numbers on the graphs represent the average Mg/Ca decreases in mmol/mol due to the reductive
308 cleaning. (g)-(j), Mn/Ca and Mg/Ca loss in %; in (g) and (h), the values and the horizontal
309 dashed line indicate the average Mn/Ca-loss in %. In (i) and (j), averaged Mg/Ca-loss (%) \pm 1
310 sigma uncertainty are displayed, with n the total number of samples; horizontal dashed line
311 indicates the average value, horizontal grey line represents the 0% loss.

312 3.2.3 *G. menardii*

313 For *G. menardii*, averaged Mg/Ca values range between \sim 1.8 mmol/mol and \sim 2.7
314 mmol/mol in core MD98-2165, and between \sim 1.3 and 2.2 mmol/mol in core MD98-2166
315 (Figure 4e-f). No clear Mg/Ca evolution corresponding to the deglaciation can be seen in the
316 records, potential glacial/interglacial structure being overshadowed by short-scale, high
317 amplitude oscillations and rapid excursions. As was already observed for *P. obliquiloculata*, *G.*
318 *menardii* Mg/Ca values are significantly lower in core MD98-2166 than in the shallower core
319 MD98-2165 and their temporal evolutions are different too. As was concluded for *P.*
320 *obliquiloculata*, the lower Mg/Ca values in *G. menardii* from Core MD98-2166 may partly result
321 from the poorer preservation at this deeper site (Johnstone et al., 2011; Regenberg et al., 2014).
322 However, we interpret the different time-evolution of the two *G. menardii* Mg/Ca records as
323 resulting from the regional complexity of the thermocline structure and its specific evolution at
324 the two sites, which result in different temperature conditions recorded at our two sites.

325 Similarly, to what was observed for the two previous planktonic species, Mg/Ca ratios
326 obtained with the reductive cleaning protocol (blue curves) are usually lower than Mg/Ca ratios
327 obtained without the reductive cleaning protocol (red curves), showing an averaged \sim 0.21
328 mmol/mol decrease in core MD98-2165 and a smaller \sim 0.08 mmol/mol decrease in core MD98-
329 2166. However, the variance is higher than for the two other species. In the shallow core MD98-
330 2165, the Mg/Ca cleaning differences average $8.5 \pm 6.3\%$ (1σ , n=48), whereas they average
331 3.7% with a particularly high variance of $\pm 11.1\%$ (1σ , n=53) in the deeper core MD98-2166
332 (Figure 4i-j). Such particularly strong variance in the deeper core is largely controlled by a few
333 odd samples, which display much higher reductively-cleaned Mg/Ca values than their non-
334 reductively cleaned aliquots (up to -30% difference; Figure 4 j). Similarly, to what we observed
335 for *P. obliquiloculata* in the deeper core MD98-2166, the combination of high variance in the
336 Mg/Ca difference, and the low amplitude evolution of Mg/Ca along the core result in a very poor
337 linear relationship between the reductively cleaned and non-reductively cleaned Mg/Ca values in
338 MD98-2166 ($R^2=0.24$).

339 Overall, when considering the three planktonic species together, Mg/Ca ratios from
340 Group B (with reductive step) are significantly reduced compared to those from Group A
341 (Figures 2, 3 and 4), and Mg/Ca ratios from Group B aliquots being lowered by $6 \pm 7\%$ (1σ ,
342 n=328) compared to aliquots from Group A.



343

344 **Figure 4.** Element/Ca of *G. menardii* from core MD98-2165 (a, c, e) and core MD98-2166 (b, d,
 345 f). In (a)-(f), the red circles shows Element/Ca ratios obtained without the reductive step, the blue

346 circles corresponds to Mg/Ca ratios obtained with the reductive step. In (e) and (f), the numbers
347 on the graphs represent the average Mg/Ca decreases in mmol/mol due to the reductive cleaning.
348 (g)-(j), Mn/Ca and Mg/Ca loss in %; in (g) and (h), the values and the horizontal dashed line
349 indicate the average Mn/Ca-loss in %. In (i) and (j), averaged Mg/Ca-loss (%) \pm 1 sigma
350 uncertainty are displayed, with n the total number of samples; horizontal dashed line indicates
351 the average value, horizontal grey line represents the 0% loss.

352 **4 Discussion**

353 **4.1 Statistical robustness of our estimates of cleaning-related Mg loss**

354 Considering the rather high scattering of datasets, it is important to decipher first whether
355 we can confidently compare and rank the averaged Mg/Ca loss in both % and mmol/mol
356 between the three species within a specific core and between the two sites for a given species.
357 We applied Shapiro-Wilk tests to our data. Results indicate that Mg/Ca differences between
358 paired A-B aliquots are random, showing normal distributions. Thus, we are allowed to run t-test
359 to check for the statistical robustness of similarity/dissimilarity between average Mg/Ca losses.
360 Prior to performing those tests, we run F-tests to decipher whether variances of the paired
361 datasets we want to compare are similar or dissimilar, which made it possible for us to select the
362 most appropriate t-test (i.e. Student test for equal variance cases, Welch's t-test for unequal
363 variance cases).

364 When comparing two species at a time, those t-tests show that the averaged Mg/Ca-loss
365 in % obtained for *G. ruber* and *P. obliquiloculata*, and for *P. obliquiloculata* and *G. menardii* at
366 site MD98-2165 are significantly different from one another (at a 95% confidence level), as are
367 the averaged Mg/Ca-loss in mmol/mol. However, the Mg/Ca loss of *G. ruber* (0.26 mmol/mol)
368 and *G. menardii* (0.21 mmol/mol) cannot be significantly discriminated from one another. For
369 site MD98-2166, the t-tests show that the averaged Mg/Ca-loss in % obtained for the three
370 species are not significantly different, except for *G. ruber* (0.16 mmol/mol) and *G. menardii*
371 (0.08 mmol/mol), which can be significantly discriminated.

372 As for the comparison between sites of the three species, Mg/Ca loss in both % and
373 absolute mmol/mol of *G. ruber* and *G. menardii* are significantly different and higher at the
374 shallower site MD98-2165 than at the deeper site MD98-2166. The t-test indicate, however, that
375 the difference in Mg/Ca loss between the two sites is not significant for *P. obliquiloculata*.

376 **4.2 Analytical scattering, instrumental stability, inhomogeneity of foraminiferal tests**

377 Despite careful sample preparation and analytical precautions (i.e. sequential ICP-MS
378 analyses of paired Group A and Group B aliquots, drift correction), Mg/Ca differences show
379 scattering and variability, which are of the same order of magnitude and sometimes higher than
380 the averaged offsets, and which are much larger than long-term instrumental uncertainty (i.e.
381 \pm 1.4%, 1σ). The lowest variability is observed for *G. ruber*, which shows a standard deviation of
382 \pm 3.5% (1σ) around the 6.2% mean difference at site MD98-2165 (Figure 2i) and shows a
383 standard deviation of \pm 4.6% (1σ) around the 4.1% mean at site MD98-2166 (Figure 2j). On the
384 other side of the spectrum, the highest variance is observed for *G. menardii*, with a standard
385 deviation of \pm 6.3% (1σ) around a mean value 8.5% at site MD98-2165 (Figure 4i), and a
386 staggering standard deviation of \pm 11.1% (1σ) around a mean value of 3.7% at the deeper site
387 MD98-2166 (Figure 4j).

388 As can be readily seen in [Figures 2, 3 and 4](#), standard deviations, in each data set,
389 integrate a few “anomalous” data points for which the batch A-batch B aliquot differences are
390 negative, meaning that the reductively cleaned sub-samples show higher Mg/Ca ratios than their
391 corresponding non-reductively cleaned aliquots. These anomalous values are particularly high in
392 the *G. menardii* dataset from core MD98-2166, with one sample showing a non-reductively
393 cleaned A-aliquot with 29% less Mg than the corresponding B-aliquot ([Figure 4j](#)).

394 Our results also show that there is no apparent co-variance between relative Mg/Ca loss
395 in the three planktonic species along each two cores (see panels (i) and (j) in [Figures 2, 3 and 4](#)).
396 It has been estimated that the $\Delta[\text{CO}_3^{2-}]$ change in the deep Indian Ocean water over a glacial-
397 interglacial cycle was about 7 $\mu\text{mol/kg}$ (Kerr et al., 2017). This is only about a third of the ~21
398 $\mu\text{mol/kg}$ $\Delta[\text{CO}_3^{2-}]$ offset that exists today between the two studied sites as a result of their
399 difference in water depth. The largest, average difference in Mg/Ca-loss that we attribute to
400 dissolution difference between cores MD98-2165 and MD98-2166 is ~ 4.8% for *G. menardii*.
401 Taken at face value, this suggests that the glacial-interglacial change in bottom water $\Delta[\text{CO}_3^{2-}]$
402 may have only accounted for change of ~ 1.6% (one third of 4.8%) along the cores. Due to the
403 rather large analytical uncertainties, such a glacial-interglacial variance in the Mg/Ca-cleaning
404 offset cannot be resolved from our data as is clearly indicated by our observations, which failed
405 to highlight a coherent downcore evolution of the Mg/Ca-cleaning offset.

406 The above observations suggest that most of the variance that we observe in cleaning A-
407 B Mg/Ca differences along a given core is random, reflecting inhomogeneities between aliquots
408 and analytical uncertainties, and does not reflect any strong, stratigraphic pre-conditioning of the
409 samples (e.g. through evolution of dissolution conditions at the seafloor during the deposition of
410 sedimentary material). Such random scattering around the mean values is also confirmed by
411 Shapiro-Wilk tests (i.e. null hypothesis of normal distribution with $p>0.05$), which indicate that
412 Mg/Ca differences between paired A-B aliquots can be considered as normal distributions. So,
413 our results suggest that past changes in the carbonate preservation state at either site were not
414 strong enough to strongly imprint downcore changes in the cleaning-related Mg loss. We have to
415 stress out, however, that such a result may not necessarily apply to sediment material from other
416 areas of the deep ocean.

417 Although we cannot reject the possibility that scattering and these anomalous negative
418 values (i.e. Mg/Ca in non-reductively cleaned samples < Mg/Ca in reductively cleaned samples)
419 result to some extent from analytical noise, this should be only marginal as there is no evidence
420 of severe and rapid instable ICP-MS behavior during runs. Anomalous Mg/Ca paired-values are
421 not associated, for instance, with anomalous values of other elements measured together with Mg
422 and Ca. Thus, we believe that variances of several percent around the averaged Mg/Ca
423 differences chiefly result from inhomogeneity between aliquots from the same samples, despite
424 our efforts to mix thoroughly crushed foraminifer tests before splitting them into paired-aliquots.

425 In order to avoid excessive loss of calcite material during the ultra-sonification of the
426 cleaning protocols, foraminifer tests were only slightly crushed to open shell chambers but not
427 finely ground ([Barker et al., 2003](#)). It is known that Mg is heterogeneously distributed in the
428 shells of the three species used in this study: *G. ruber* ([Eggins et al., 2003](#); [Sadekov et al., 2009](#)),
429 *P. obliquiloculata* ([Sadekov et al., 2009](#); [Steinhardt et al., 2015](#)) and *G. menardii* ([Sadekov et al.,](#)
430 [2005](#)). Large fragments of shell chambers may not allow for a thorough enough homogenization
431 and could result in slightly different Mg/Ca composition between A and B aliquots. It is
432 noteworthy that the two species of our data sets (namely *P. obliquiloculata* and *G. menardii*)

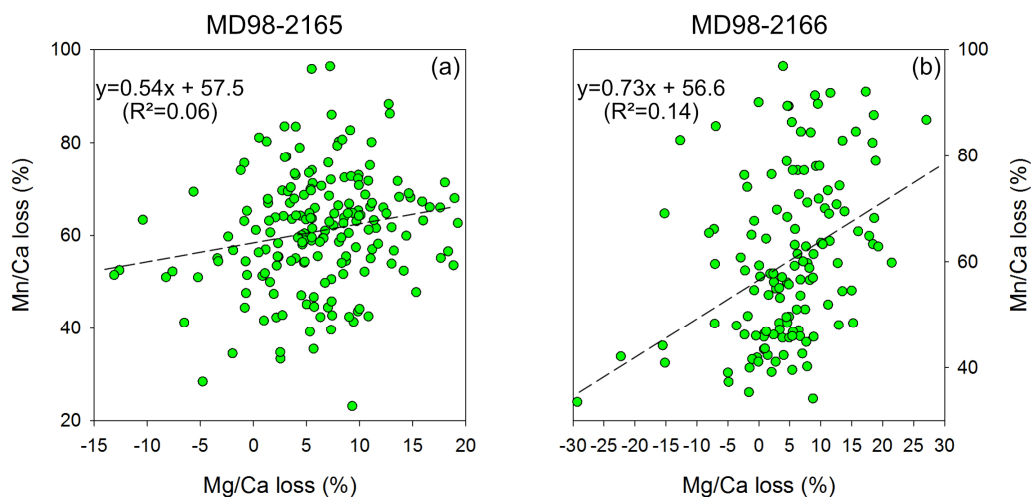
433 showing the highest variance in terms of Mg-cleaning loss and extreme anomalous negative
434 values, are two species with complex structures characterized by a very heterogeneous
435 distribution of Magnesium in the tests. *P. obliquiloculata* has a thick and dense outside cortex
436 with lower Mg/Ca than the inside of the test (Steinhardt et al., 2015). *G. menardii* shows a heavy
437 outer crust as well as thick keel (Regenberg et al., 2010), which are characterized by low Mg/Ca
438 ratios (~ 2 mmol.mol⁻¹) that contrast with the higher Mg/Ca (ranging from 4 to 6 mmol.mol⁻¹) of
439 the inner chamber calcite (Sadekov et al., 2005). *G. ruber*, on the other hand, is a bi-lamellar
440 species: each new layer of calcite is constituted by an inner, Mg-rich primary layer and an outer,
441 Mg-poorer layer. Thus, contrary to *G. menardii* and *P. obliquiloculata* – whose heterogeneity
442 occurs at the test-scale - Mg heterogeneity in *G. ruber* occurs at the wall-scale, and breaking
443 open the tests should not result in significant Mg/Ca differences between the large fragments.
444 This may explain the lower standard deviation shown by Mg-difference between aliquots of *G.*
445 *ruber* samples compared to the two other species.

446 We conclude from the above observations that Mg/Ca differences between paired A-B
447 aliquots of a given species chiefly reflect the excess-dissolution of Mg-rich layers induced by the
448 reductive cleaning procedure (Barker et al., 2003; Rosenthal et al., 2004; Yu et al., 2007), with
449 no clear impact from the removal of oxides (i.e. no relationship between Mg and Mn relative
450 losses). Our data show that a rather large scattering can be superimposed on the relative Mg/Ca
451 offset between the cleaning methods. We attribute the particularly high scattering to
452 inhomogeneities in Mg distribution within the foraminifer test (i.e. and, thus, between the
453 fragments), which randomly affect the Mg difference between A and B aliquots, leading (i) to a
454 higher scattering in *G. menardii* and *P. obliquiloculata* datasets than in *G. ruber* dataset and (ii)
455 resulting in odd, negative offsets for some samples (i.e. Mg/Ca in A aliquots < Mg/Ca in B
456 aliquots).

457 **4.3. What controls the relative loss of Mg during reductive cleaning?**

458 As indicated above, the reductive cleaning protocol results in a systematic and important
459 relative loss of manganese in batch-B aliquots compared to batch-A aliquots. The relative losses
460 of Mn/Ca range from 56% for *G. menardii* in core MD98-2166 (mean Mn/Ca dropping from
461 0.23 mmol/mol to 0.11 mmol/mol when adding the reductive step) up to 66% for *P.*
462 *obliquiloculata* in core MD98-2166 (the mean Mn/Ca dropping from 0.22 mmol/mol to 0.08
463 mmol/mol). The Mn losses average 61% when computing together data from the three species at
464 both sites. Such an important Mn loss traduces the efficient removal of Mn-rich oxides when
465 using the reductive cleaning protocol. The fact that reductively cleaned samples still contain a
466 small but measurable amount of Mn suggests that this element is also present in the calcite
467 lattice. Whether this Mn is located in the foraminifer test or in a diagenetic Mn-rich carbonate
468 phases (Boyle, 1983; Pena et al., 2005) is beyond the scope of this paper. Importantly, there is no
469 relation between Mn and Mg contents and there is no relationship between the relative loss of
470 Mn and that of Mg (Figure 5), suggesting that in our two cores the cleaning-related loss of Mg
471 does not chiefly result from the removal of Mn-rich oxides that may have contained Mg among
472 other metals. Thus, our data confirm that the decrease of Mg/Ca ratios associated with the
473 reductive cleaning likely results from cleaning-induced, calcite dissolution, which preferentially
474 removes the more labile and Mg-richer parts of foraminifer tests (Barker et al., 2003; Rosenthal
475 et al., 2004; Yu et al., 2007). Recently published scanning electron microscopy (SEM) images
476 provide direct evidence of such cleaning-induced dissolution effects on foraminiferal
477 microstructures (e.g., Johnstone et al., 2016; Peral et al., 2018).

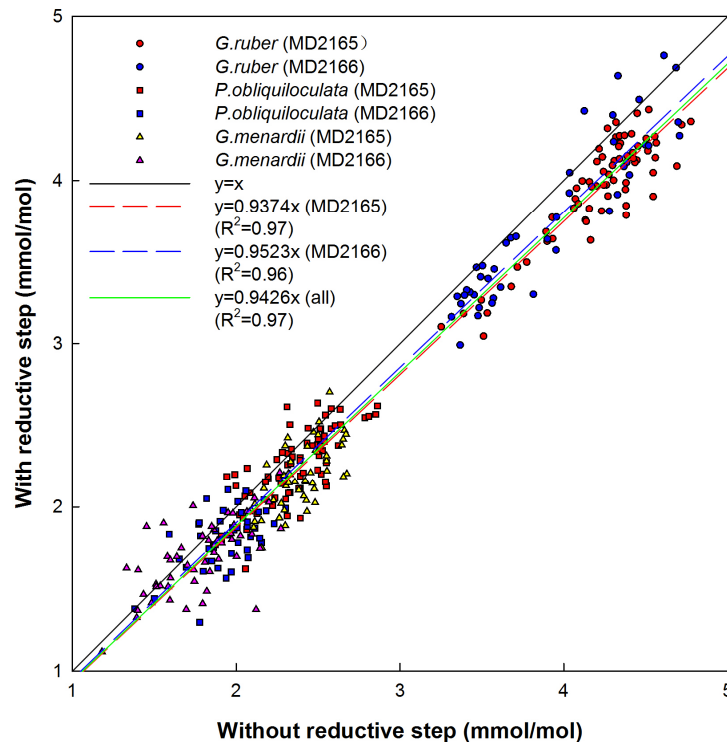
478 Mn/Ca ratios were often used to monitor cleaning efficacy. Low Mn/Ca ratios (<
 479 0.1mmol/mol) was suggested as a threshold for indicating negligible problem from oxide
 480 contaminants on foraminiferal Element/Ca analysis (Boyle, 1983). Our down core Mn/Ca ratios
 481 are usually higher than 0.1mmol/mol for non-reductively cleaned samples. Even for the samples
 482 cleaned with the reductive step, there is still a considerable number of samples with Mn/Ca ratios
 483 in excess of 0.1mmol/mol. However, there is no evidence showing that Mg/Ca ratios for the two
 484 studied cores are affected by oxide contaminants. This suggests that Mn/Ca values might not
 485 always be a good indicator for monitoring cleaning efficacy for Mg/Ca analysis.



486
 487 **Figure 5.** Comparison of Mg/Ca-loss (%) versus Mn/Ca-loss (%) of all three species (i.e. *G.*
 488 *ruber*, *P. obliquiloculata* and *G. menardii*) from core MD98-2165 (a) and core MD98-2166 (b).
 489 The dashed lines indicate the best linear fits. Note the absence of significant relationships
 490 between Mg/Ca-loss and Mn/Ca-loss at both sites.

491 **4.4 Cleaning effect on Mg/Ca: a multi-species perspective**

492 Figure 6 compares all our data in a X-Y plot, with reductively cleaned Mg/Ca values (Y)
 493 plotted versus non-reductively cleaned values (X). Since there is no evidence of a coherent
 494 evolution of A-B Mg/Ca differences along the cores (panels (i) and (j) in Figures 2, 3 and 4),
 495 such a synthetic plot provides the best graphic solution to summarize the distribution and relative
 496 contribution of each species and core to the overall estimate of Mg/Ca loss (Figure 6). The slope
 497 obtained through a simple linear regression approach has been used in many previous studies to
 498 provide an estimate of the average Mg differences between A and B aliquots (Barker et al., 2003;
 499 Rosenthal et al., 2004; Yu et al., 2007). When taking into account the three species together, at
 500 the two sites, the overall Mg-loss is ~ 5.7% (i.e. slope 0.943 of the green regression line, Figure
 501 6).



502

503 **Figure 6.** Comparison of Mg/Ca ratios obtained with cleaning methods including and excluding
 504 the reductive step. Solid line, $y=x$; dashed line, linear best fits forced through the origin for
 505 samples from MD98-2165 (red), from MD98-2166 (blue) and all the data from the two sediment
 506 cores (green).

507 This loss is much smaller than the 15% loss found by Rosenthal et al., (2004) from their
 508 multi-planktonic foraminifera study. It is noteworthy that their estimate of a 15% decrease due to
 509 reductive cleaning was obtained from a linear regression approach without forcing the intercept
 510 to zero, whereas we chose to force the intercept to zero in Figure 6. Forcing the regression to
 511 zero assumes that the linear tendency we observe over the 1.5-4.5 mmol/mol Mg/Ca range of our
 512 dataset can be strictly extrapolated down to a zero Mg loss in a pure calcite (i.e. no Ca
 513 substitution). By forcing the intercept to zero, the average loss we obtained through this approach
 514 is similar to that obtained by computing a simple arithmetic mean of the whole dataset. We used
 515 published data from Rosenthal et al. (2004, Figure 5 therein), and run a linear regression with an
 516 intercept forced to zero. The re-evaluated slope indicates a reductive Mg loss of 6% for the
 517 multi-species, foraminifer Mg/Ca database of Rosenthal et al. (2004); a result fully consistent
 518 with our own. This suggests that differences between published Mg-loss may not only result
 519 from different protocols or different species or assemblages being analyzed, but may also depend
 520 upon the approach used to estimate the average Mg/Ca decrease.

521 4.5 Species-dependence of Mg loss during reductive cleaning

522 Merging all the data together provides an estimate of cleaning Mg/Ca loss (~ 5.7%) that
 523 is coherent with the revisited Mg/Ca loss (6%) using the database of Rosenthal et al. (2004).

524 Such multi-species estimates of cleaning loss mask, however, species-dependent differences,
525 which appear statistically significant when applying Student or Welch's t-tests.

526 In order to highlight the potential species-dependent differences in Mg/Ca resulting from
527 the cleaning process, we focus on the data from the shallower of our two cores, MD98-2165,
528 which provides relatively well-preserved planktonic tests. Among the three species we studied,
529 *P. obliquiloculata* shows the smallest average loss of Mg (~ 4.4%, Figure 3i). This average Mg-
530 loss is significantly higher than the 1.8% Mg-loss found by Xu et al., (2008) and the 0% loss
531 estimated by Johnstone et al., (2016) for this species. But the average loss we found is slightly
532 smaller than the ~ 6% loss published by Fan et al. (2018) for *P. obliquiloculata* tests picked from
533 two Makassar Strait cores (Indonesian Archipelago) and retrieved at shallower water depths
534 (1185m and 1984m) than MD98-2165 (2100m).

535 Our data indicate that *G. menardii* shells lose almost twice as much Mg (8.5%) than *P.*
536 *obliquiloculata* (4.4%) in core MD98-2165 when a reductive step is added to the cleaning
537 protocol (Figure 4i). The third species studied, *G. ruber*, shows an intermediate, cleaning-related
538 Mg loss, which averages 6.2 %. This result is consistent with the 6.6% decrease observed by Xu
539 et al., (2008) using material from a nearby sediment core retrieved at a similar water depth
540 (1783m) in the Indonesian Archipelago. Interestingly, Xu et al. (2008) and the present study
541 obtain the same Mg-loss for *G. ruber* (~ 6%), whereas the two studies differ significantly with
542 regard to the *P. obliquiloculata* Mg-loss (see above).

543 The glossy *P. obliquiloculata* test is one of the most dissolution-resistant among
544 planktonic foraminifers (Berger, 1967; Johnstone et al., 2016). Increasing the substitution of Ca
545 by Mg is known to degrade calcite crystallinity and increase its sensitivity to dissolution
546 (Bassinot et al., 2004; Brown & Elderfield, 1996; Nouet & Bassinot, 2007). We believe that the
547 smaller decrease of Mg during the reductive cleaning (i.e. higher resistance to dissolution) shown
548 by *P. obliquiloculata* compared to the two other species, results from the lower Mg content of its
549 calcite test (with Mg/Ca ranging from ~ 2.0 mmol.mol⁻¹ to 2.7 mmol.mol⁻¹ in core MD98-2165).

550 Interestingly, *G. menardii* not only losses twice as much Mg (8.5%) than *P.*
551 *obliquiloculata* (4.4%) when cleaned with the reductive protocol, but it also losses more Mg than
552 *G. ruber* (6.2%). This higher relative loss of Mg from *G. menardii* seems to be at odd with the
553 fact that *G. ruber* is considered a dissolution-sensitive planktonic species, whereas *G. menardii* is
554 considered a dissolution-resistant one (Berger, 1967). However, it is important to recall that the
555 capacity of *G. menardii* to resist dissolution is chiefly due to the presence of a thick and low-Mg
556 keel (Sadekov et al., 2005). This keel can be preserved in sediments as easily identifiable,
557 crescent-shaped fragments, even when the chamber walls have long been dissolved and *G.*
558 *menardii* tests reduced to fragments (Mekik & François, 2006; Mekik et al., 2007). If not for this
559 resistant keel, *G. menardii* should not be considered a dissolution-resistant species since its
560 walls, which are two to three times richer in Mg than the keel (Sadekov et al., 2005), can show
561 very early signs of dissolution (i.e. holes starting to develop early in the external walls of the last
562 foraminifer chambers and rapidly increasing in size with proceeding dissolution). The early
563 impact of dissolution on its chamber walls, associated with the resistance of its keel make *G.*
564 *menardii* an interesting candidate for developing semi-quantitative proxies of dissolution
565 intensity at the seafloor (Mekik & François, 2006). We believe that it is this capacity of chamber
566 walls to dissolve rapidly that explains why *G. menardii* tests from core MD98-2165 losses more
567 Mg than *P. obliquiloculata* and even more than *G. ruber* when subject to the reductive cleaning.

568 **4.6 The effect of in situ dissolution in modulating Mg/Ca cleaning reduction**

569 When considering separately Mg/Ca data from the two cores (Figure 6), planktonic
570 foraminifers picked from the shallow core MD98-2195 show a slightly higher Mg-loss during
571 reductive cleaning (slope of 0.937, i.e. loss of ~6.3%) than foraminifers from the deeper
572 MD98-2166 core (slope of 0.952, i.e. ~4.8% loss). The difference is small but consistent with
573 the hypothesis that foraminifers having undergone *in situ* dissolution are likely to lose less Mg
574 during cleaning process than better-preserved tests (Barker et al., 2003). However, the impact of
575 *in situ* dissolution is actually a bit more complex than just a reduced cleaning loss of Mg with
576 increasing water depth of deposition. Our data suggest, here again, that there are some species-
577 related differences in the way *in situ* dissolution impacts Mg loss during cleaning.

578 As just seen above, *G. menardii* tests from the shallow core MD98-2165 show a
579 significantly higher cleaning-related Mg/Ca decrease (8.5%) than the other two species (6.2% for
580 *G. ruber*, 4.4% for *P. obliquiloculata*). In the deeper core MD98-2166, however, *G. menardii*
581 exhibits a similar or lower cleaning-related relative Mg/Ca decrease (3.7%) than the other two
582 species (4.1% for *G. ruber*, 6.3% for *P. obliquiloculata*). This implies that different preservation
583 states of foraminiferal tests between the two sites exert an impact on Mg loss during the
584 reductive cleaning step. Both the relative (%) and absolute (mmol/mol) Mg/Ca losses for *G.*
585 *menardii* are significantly higher for the shallower core MD98-2165 (8.5%, 0.21 mmol/mol) than
586 the deeper core MD98-2166 (3.7%, 0.08 mmol/mol) (Figure 4 e-f and i-j). We interpret the
587 strong difference in *G. menardii* cleaning Mg-loss between the shallow core MD98-2165 and the
588 deep core MD98-2166 as an indication that *in situ* dissolution, which has presumably removed
589 some part of Mg-rich chamber walls (and, thus, has increased the relative contribution of the
590 Mg-poor, dissolution resistant keel), significantly reduces the capacity of *G. menardii* to lose
591 further Mg during the reductive cleaning.

592 Similar to what we observe for *G. menardii*, *G. ruber* data reveal a potential “pre-
593 conditioning” effect of *in-situ* dissolution on the cleaning-related loss of Mg. Our data show that
594 cleaning-related offsets in Mg/Ca ratios are smaller for *G. ruber* (4.1%, 0.16 mmol/mol) in the
595 deeper core MD98-2166 than from the shallower core MD98-2165 (6.2%, 0.26 mmol/mol). This
596 difference is seen as statistically robust when running a t-test. This suggests that, similarly to
597 what we observed for *G. menardii*, the tests of *G. ruber* from the deeper core, which have
598 experienced more intense dissolution on the seafloor than the tests from the shallow core, are less
599 prone to lose further Mg during reductive cleaning.

600 Contrary to what is observed for the two other species, *P. obliquiloculata* exhibits a
601 stronger, cleaning-induced Mg/Ca decrease in the deeper core MD98-2166 (6.3%, 0.13
602 mmol/mol), than in the shallower core MD98-2165 (4.4%, 0.11 mmol/mol) (Figure 3). But this
603 difference is not statistically robust when running a t-test. This suggests that the decrease in
604 Mg/Ca due to reductive cleaning step is insensitive to the preservation state for *P.*
605 *obliquiloculata*.

606 **4.7. Including or not the reductive step in the cleaning method?**

607 There is no doubt that using a reductive step during cleaning is necessary for those
608 specific elements that are abundant in oxide coatings (e.g. Cd, U and Ba; Boyle & Keigwin,
609 1985; Yu et al., 2007), as well as, apparently, for Mg/Ca analyses performed on samples
610 particularly affected by oxide contamination, such as those from in the Panama basin (Pena et al.,

611 2005, 2008). For most of non-problematic regions, like our study area in the Indonesian
612 Archipelago, adding a reductive step does not improve Mg/Ca measurements. We observed no
613 relationship between cleaning-related loss of Mg and Mn, and there are no outliers showing
614 anomalously high Mg/Ca ratios associated to high Mn/Ca values in the non-reductively cleaned
615 (A) samples. This suggests that the amount of Mg associated to oxides is negligible compared to
616 the amount of Mg that is contained in the foraminiferal calcite, an observation already made by
617 [Barker et al. \(2003\)](#). Reductive cleaning does not apparently improve Mg/Ca results with respect
618 to the removal of oxides, but it significantly reduces the Mg/Ca ratios through the preferential
619 dissolution of Mg-rich parts of the foraminiferal tests. In addition, as can be readily seen from
620 Figures 2, 3 and 4, anomalous Mg/Ca values are more often found in the reductively cleaned
621 datasets, an observation which could reflect the fact that the reductive cleaning adds more
622 manipulations (i.e. increased likelihood of contamination) and is more difficult to conduct
623 properly (i.e. it requires a precise timing of the reductive step to avoid variability in dissolution-
624 induced changes). Thus, we believe that the reductive cleaning should be used only in sediments
625 with severe oxide coating of the particles and/or when the paucity of planktonic foraminifers
626 makes it impossible to conduct separate analyses for Mg/Ca and other elements that are
627 significantly contaminated by oxides (e.g. Cd, Ba). If one is interested chiefly in Mg/Ca, we
628 believe that it is best to use the non-reductive approach for cleaning the foraminiferal tests. In
629 cases when the reductive step must be included, attention should be paid to Mg/Ca results from
630 species having strongly non-homogeneous distribution of Mg. Our results show, for instance,
631 that in order to obtain reliable Mg/Ca-based temperatures from *G. menardii*, it is mandatory to
632 work on well-preserved samples, and the use of reductive step during cleaning should be better
633 avoided.

634 **4.8 Correcting for the Mg/Ca offsets induced by the cleaning methods**

635 When dealing with spatial temperature distribution and trying to map temperature
636 anomalies at a given period of the past (e.g. Last Glacial Maximum), it is crucial to insure the
637 compilation of the most coherent temperature estimates possible. In such cases, efforts should be
638 made to use as much as possible Mg/Ca data obtained with the same cleaning protocol.
639 However, to date, no agreement has been reached regarding the cleaning protocol for Mg/Ca
640 analyses. Some laboratories add a reductive step, while others do not include it. As a
641 consequence, when comparing data from different laboratories or dealing with the construction
642 of Mg/Ca-T° databases, one has to cope with the potential problem of mixing inhomogeneous
643 Mg/Ca datasets (e.g. [Xu et al., 2010](#); [Fan et al., 2018](#); [Wang et al., 2018](#)). This problem does not
644 only concern direct Mg/Ca-T° reconstruction. Mg/Ca-derived temperatures are also used, for
645 instance, to reconstruct other paleoceanography proxies, such as the $\delta^{18}\text{O}$ of seawater ([LeGrande
646 & Schmidt, 2006](#); [Thirumalai et al., 2016](#)), or to study the thermocline structure based on the
647 Mg/Ca-T° gradient between shallow and deep-dwelling species such as *G. ruber* and *P.
648 obliquiloculata* (e.g., [Holbourn et al., 2011](#); [Fan et al., 2018](#); [Hollstein et al., 2018](#); [Wang et al.,
649 2018](#)). In all these cases, cleaning-induced differences in Mg/Ca can bias the interpretations. It is
650 therefore crucial to decipher how we can minimize cleaning-induced Mg/Ca differences between
651 datasets.

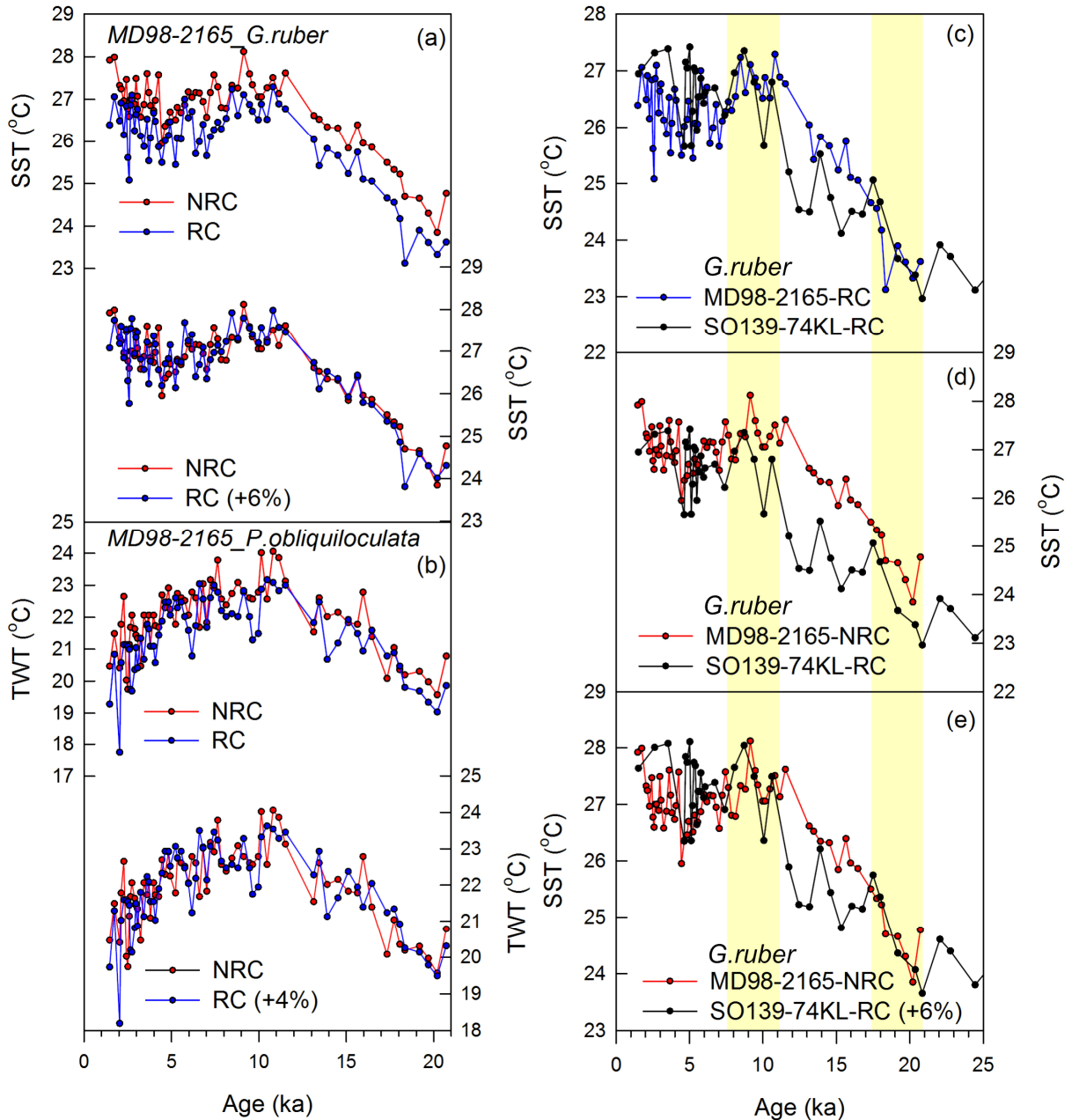
652 Our results and those from previous studies show that simple linear corrections can be
653 applied. In [Figure 7 \(a-b\)](#), we compare temperature records obtained in core MD98-2165 using
654 the initial Mg/Ca results for *G. ruber* (panel a) and *P. obliquiloculata* (panel b) converted to
655 temperatures using the empirical equation $\text{Mg/Ca} = 0.38 \exp(0.09 \cdot T^\circ)$ for *G. ruber* and Mg/Ca

656 = $0.328 \exp(0.09 \cdot T^\circ)$ for *P. obliquiloculata* (Anand et al., 2003). Without any correction for
657 cleaning effects (upper curves), the average temperature difference between reductively cleaned
658 and non-reductively cleaned samples is $\sim 0.7^\circ\text{C}$ for *G. ruber* and $\sim 0.5^\circ\text{C}$ for *P. obliquiloculata*.
659 As expected these differences cancel out (lower curves) when correcting reductively cleaned
660 Mg/Ca values with species-specific offsets (i.e. +6% for *G. ruber* and +4% for *P.*
661 *obliquiloculata*) prior to temperature conversion. Using the same multi-species, average offset
662 factor (5.7%, Figure 6) to correct Mg/Ca data from *P. obliquiloculata* would have resulted in
663 slightly over-corrected *P. obliquiloculata* Mg/Ca-thermocline T° , thus reducing artificially the
664 vertical temperature gradients that can be deduced from comparing the temperatures obtained
665 with the *G. ruber* Mg/Ca-SST. Yet, using a common, multi-species correction factor does not
666 modify the downcore evolution of the *G. ruber* and *P. obliquiloculata* temperature records, and
667 does not affect, therefore, the interpretation of the relative evolution of vertical temperature
668 gradient at site MD98-2165.

669 The necessity for as precise as possible quantitative estimates of temperatures is linked to
670 our interest in comparing those temperatures between different sites of the same region. In order
671 to illustrate potential misinterpretations that may result from cleaning issues and the interest of
672 correction for these effects, we compared Mg/Ca-SST that we obtained from *G. ruber* in the
673 shallow core MD98-2165, with *G. ruber* Mg/Ca-SST obtained from nearby core SO139-74KL
674 (Wang et al., 2018) located to the South off Sumatra (Figure 1). Mg/Ca ratios in SO139-74KL
675 were measured from *G. ruber* in the 250-350 μm size fraction and the cleaning method included
676 a reductive step (Wang et al., 2018). Mg/Ca ratios were converted to SST by using, for both
677 records, the same empirical equation for *G. ruber*: $\text{Mg/Ca} = 0.38 \exp(0.09 \cdot T^\circ)$ (Anand et al.,
678 2003). As can be seen from Figure 7c, Mg/Ca-SST obtained using the same cleaning method (i.e.
679 with reductive step) indicate roughly comparable SST between the two cores during the early
680 onset of deglaciation (20 – 18 ka Cal BP) and the early Holocene (starting around 10ka), with a
681 strong temperature gradient developing during the deglaciation itself. However, when we
682 compare Mg/Ca-SST obtained using different cleaning methods (i.e. using MD98-2165 data
683 obtained without the reductive step), the data suggest that a lateral temperature gradient always
684 existed between the two sites since the LGM and only collapsed sometimes during the mid-
685 Holocene (Figure 7d). Thus, paleo-interpretation is clearly affected by the cleaning effects on
686 Mg/Ca records. We applied the *G. ruber* +6% correction to reductively-cleaned Mg/Ca data from
687 core SO139-74KL (Figure 7e). After correction, the evolution of both Mg/Ca-SST records is
688 fully coherent with the interpretation resulting the comparison of Mg/Ca-SST obtained with the
689 same cleaning technique (Figure 7c). This example demonstrates the necessity to adjust the
690 cleaning-offset when precise, absolute Mg/Ca-based water temperatures are needed (i.e., vertical
691 or lateral gradients) for paleo-interpretation.

692 Although a multi-species correction factor (i.e., 5.7%, see Figure 6) is practical to use to
693 minimize cleaning-offsets, we suggest using species-dependent correction factors when possible
694 (i.e. 6 % for well-preserved samples of *G. ruber* and 4 % for well-preserved samples of *P.*
695 *obliquiloculata*). For the poorly-preserved samples of *G. ruber* (such as the case in MD98-2166),
696 a smaller correction factor of 4% should be more appropriate. For samples of *G. menardii* and
697 the poorly-preserved samples of *P. obliquiloculata*, it would be inappropriate to correct the
698 cleaning-offset by a universal factor, as their large variance and weak correlation between Mg/Ca
699 ratios cleaned with and without the reductive step; in fact, it is as well as pointless to correct
700 them because they have lost the ability to reflect the water temperature due to the altered Mg/Ca

701 ratios caused by the *in situ* dissolution. For those other species not mentioned in this study, the
 702 multi-species correction factor of 5.7% could be employed.



703
 704 **Figure 7.** (a) Comparison of *G. ruber* Mg/Ca-SST with using different cleaning methods in core
 705 MD98-2165, and cleaning-offset correction; SST is the short of sea surface temperature; TWT is
 706 the short of thermocline water temperature; NRC represents non-reductively cleaned samples
 707 and RC represent reductively cleaned samples. (b) Comparison of reductively and non-
 708 reductively cleaned *P. obliquiloculata* Mg/Ca-TWT in core MD98-2165, and cleaning-offset
 709 correction. (c)(d)(e) Comparison of *G. ruber* Mg/Ca-SST between core MD98-2165 and SO139-

710 74KL (Wang et al., 2018) cleaned with and without reductive step and cleaning-offset correction.
711 The yellow bars show intervals that are discussed in the text.

712 5 Conclusions

713 In this study, we compared the Mg/Ca ratios obtained using two cleaning methods on
714 three planktonic species, *G. ruber*, *P. obliquiloculata* and *G. menardii*, picked from two
715 sediment cores retrieved at different water depths. The Mg/Ca records cover the last ~21ka Cal
716 BP for core MD98-2165 (2100m) and ~35ka Cal BP for core MD98-2166 (3875m).

717 Our results show that adding a reductive step decreases the Mg/Ca ratio for all three
718 species. The overall Mg/Ca loss averages 6% (0.16 mmol/mol). In the shallow, well-preserved
719 core MD98-2165, t-tests show that differences between averaged Mg/Ca % losses in the three
720 species are statistically robust, indicating that the cleaning loss is species-specific to some extent.
721 The cleaning-related differences in Mg/Ca (in both relative % and mmol/mol) of *G. ruber* and *G.*
722 *menardii* are higher in the shallower core MD98-2165 than in the deeper core MD98-2166,
723 suggesting that foraminifera tests which have experienced intense dissolution on the sea-floor are
724 less prone to lose further Mg during the reductive cleaning. *P. obliquiloculata*, however, show
725 non-significant difference in Mg/Ca loss between the two sites, indicating that cleaning related
726 Mg/Ca loss in this species is insensitive to the preservation state.

727 Well-preserved *G. menardii* shells retrieved in core MD98-2165 shows the strongest
728 cleaning-induced Mg/Ca decrease of our study (8.5%), which is likely due to the fragile nature of
729 the wall calcite that tends to dissolve easily. *G. menardii* specimens from Core MD98-2166,
730 which have experienced some dissolution at the seafloor, show a marked reduction in cleaning-
731 related Mg/Ca loss (3.7%) compared to specimens from the shallow core. We interpret this result
732 as being indicative of the strong resistance to dissolution of the remaining outer crust and keel,
733 characterized by low Mg/Ca composition (Sadekov et al., 2005). This suggests that well-
734 preserved *G. menardii* specimens (retrieved from shallow cores, always above the lysocline) are
735 mandatory for reconstructing unbiased Mg/Ca-based thermocline temperature, and non-reductive
736 cleaning procedure is clearly preferable for this species to prevent partial dissolution.

737 For all species studied, reductive cleaning results in a clear Mg/Ca decrease compared to
738 non-reductively cleaned values. However, there is no sign of significant changes in the relative
739 Mg/Ca variations along a given core and for a given species when comparing the curves obtained
740 by the two cleaning methods. This suggests that past changes in the carbonate preservation state
741 over a glacial-interglacial cycle at either site were not strong enough to result in noticeable
742 downcore changes in the cleaning-related Mg loss. In other words, relative seawater temperature
743 changes reconstructed along a given sediment records (e.g. the temperature change between
744 LGM and Holocene, for instance) are not affected by the choice of the cleaning method.

745 The averaged 5.7% correction factor that we obtained from merging together Mg/Ca-loss
746 from the three studied species at the two sites can be used for correcting cleaning effects and
747 may prove valid for other foraminifer species, although this assumption will need to be tested in
748 future studies. However, for minimizing the cleaning related Mg/Ca-offsets, we rather
749 recommend using species-dependent correction factors when possible, such as 6 % for well-
750 preserved samples of *G. ruber*, 4 % for well-preserved samples of *P. obliquiloculata* and 8.5%
751 for well-preserved samples of *G. menardii*.

752 It is the authors' contention that non-reductive cleaning approach should be preferred for
753 building inter-laboratory homogeneous datasets of Mg/Ca-derived temperatures, whereas the
754 reductive step should be only included when severe oxide coating is observed.

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