

## **FIRST REVIEWER**

### **GENERAL COMMENTS**

“A Holocene history of climate, fire, landscape evolution, and human activity in Northeast Iceland” by Ardenghi et al. is a complete and well written, well organized research paper that nicely integrates different approaches and proxies for the interpretation of human-environment dynamics during the Holocene. Islands are particularly fit for paleoenvironmental studies since changes are often more visible in lacustrine records and the source area is confined, as is demonstrated by the study of back trajectories reported in this manuscript. This work will certainly be of interest for the readers of *Climate of the Past*. I thus recommend publication after a minor revision.

We thank the reviewer for their positive feedback and insightful suggestions on our paper. We appreciate their recommendation for publication after a minor revision and address their valuable comments below.

### **SPECIFIC COMMENTS**

Line 109: six extraction cycles seem far too many compared to the usual three. Is there a particular reason for this? If so, please motivate in the text.

Sample size, matrix, and richness in organic compounds greatly varied along the core. Initial tests on a few samples occasionally showed organics being extracted even after 4-5 cycles. Thus, we decided to use a method with six short cycles as to maintain consistency in lipid yields throughout the core.

*Added to manuscript (now lines 114-116).*

Line 110: I believe ASE350 works at 1500 psi, not 2000. Please check.

*We did check and the reviewer is right; we will change to 1500 psi in the text.*

*Added to manuscript (now line 117).*

Lines 110-112: why adding the internal standards after the extraction?

It is standard practice in our laboratory to avoid using analytical standards before extraction, to avoid any risk, even if minimal, of cross contamination within the ASE350 extractor. Previous tests in our lab have shown that the extraction efficiency of a standard added to the ASE cell is very high, so adding it after extraction does not make a difference.

Although six extraction cycles certainly provide the highest yield for analytes, how do authors account for this?

As mentioned above, we noticed that the extract of some particularly rich samples were still colourful after 4-5 cycles, which likely indicated a non-negligible presence of organics.

Why using non-labelled standards here while adding deuterated ones as recovery and injection standards?

We used a column chromatography separation protocol designed to produce six lipid fractions. *n*-alkanes elute in the first fractions while PAHs in the second. We used non-deuterated standards to avoid the risk of future contamination, as even minimal amounts of these standard eluting into the wrong fraction (e.g., traces of d14 *p*-terphenyl for PAHs eluting in the first or third fractions) can easily create serious issues on an IRMS system. The deuterated standards, if necessary, were thus added after fraction separation, for example, as injection standards.

Line 129: were alkanes integrated using characteristic m/z ratios extracted from the full scan?

Yes, they were identified/integrated both by using the external standard mix, as well as by the characteristic m/z 57.

Why not using a SIM approach or FID then? What is the purpose of using a triple quadrupole in full scan mode?

Our lab has a triple quadrupole GC-MS system that can perform both untargeted (full scan) and targeted analysis (SRM). This instrument is perfectly suitable for the analysis of *n*-alkanes in full scan mode as reported in several recent publications from our group (e.g., Boudinot and Sepúlveda, 2020; Pérez-Angel et al., 2022). SRM is preferred over SIM mode for the targeted analysis of faecal sterols and PAHs because of the higher signal over noise ratio of this technique, which provides higher sensitivity. In addition, no GC-FID was available at the time in our lab.

Lines 211-225: are data coincident with the G10ka series tephra omitted due to age uncertainties or was it impossible to determine concentrations of chemical proxies in this layer? Please specify in the text to facilitate non expert readers.

The G10ka series, while being a very thick unit, is substantially like any other tephra. Its contents are almost 100% inorganic and thus we did not expect to find significant biomarker concentrations. Nevertheless, we tried analysing a couple of samples from the G10ka series but the yields of organics were too low to be reliable. We only included one sample at the bottom limit of the tephra and one at the upper limit.

*We have added this information in the revised manuscript (now lines 232-235).*

Lines 358–362: due to small concentrations of *n*-alkanes in this first part of the record, small changes (as the spike in C<sub>19</sub> at about 9 ka BP) may cause significant changes in CPI values. However, does a change from a dominant aquatic source to a dominant terrestrial source of *n*-alkanes justify the change in the correlation sign between CPI and  $\delta^{13}\text{C}$ ?

Thank you for the observation. This is possible but, however, we think it is unlikely.

First of all, our CPI<sub>21-33</sub> does not include *n*-C<sub>19</sub> nor *n*-C<sub>20</sub>; we calculated also CPI<sub>19-35</sub> and, as we did not detect any relevant change in the pattern of the CPI curve, we opted to use CPI<sub>21-33</sub>, excluding homologues missing too many datapoints. In addition, in figure A3, the scale of *n*-C<sub>19</sub> y axis is ca. 10-fold smaller than for the other *n*-alkanes; this could mislead to identify a relatively negligible increase in *n*-C<sub>19</sub> concentration at ca. 9 ka BP (a pattern similar to all the other odd and even homologues) as a “major” peak. This would be unlikely to lead to a major shift in CPI, let alone a change in the correlation between CPI and  $\delta^{13}\text{C}$ . On the contrary, we think that the stability of the initial CPI- $\delta^{13}\text{C}$  anticorrelation (high/low CPI - low/high  $\delta^{13}\text{C}$ ) before 9 ka BP is likely resulting from these proxies recording changes between aquatic dominated and terrestrial dominated sources. Thus, we think it is unlikely that these same changes be what modified the sign of the CPI-  $\delta^{13}\text{C}$  correlation itself (from negative to positive) after 9 ka BP.

If the terrigenous input increases after 8 ka BP, together with *n*-alkanes related to terrestrial plants and arboreal pollen, will this explain the change in correlation? Perhaps adding ACL or similar index may help the interpretation.

Thank you for the suggestion. However, we think it is unlikely.

The ACL curve has been omitted as it mimics the pattern of the P<sub>aq</sub> curve and it would not have provided additional info.

Pollen does contain *n*-alkanes. While the *n*-alkane distribution of pollen is still dominated by odd-numbered homologues, it does usually contain a relatively important amount of even-numbered homologues (more than the usual leaf; Hagenberg et al., 1990; Bashir et al., 2013). However, relative to other plant organs (e.g., leaves, stems, flowers, fruits, roots) pollen is likely to contribute only a minor/negligible portion of the *n*-alkanes found in sediment. Thus, pollen contribution is likely too low to visibly influence *n*-alkanes distribution in sediments. In addition, while CPI steadily decreases after 8 ka BP, pollen from nearby Ytra-Åland (Karlsdóttir et al., 2014) shifts between different plant groups repeatedly, thus making it unlikely to be the source of the change in the CPI- $\delta^{13}\text{C}$  correlation.

At present, there is only one mechanism that could tentatively explain, at least partially, this change in CPI- $\delta^{13}\text{C}$  correlation. If we assume that no C<sub>4</sub> plant is present in Iceland in any relevant amount,  $\delta^{13}\text{C}$  likely traces aquatic/terrigenous sources. In contrast, CPI behaviour could be influenced by shifts in aridity/seasonality, which seem to decrease CPI of *n*-alkanes of plant organs (e.g., leaves) while also increasing soil *n*-alkane aerobic exposure (and degradation), thus leading to lower sedimentary CPI values (Ardenghi et al., in review). Nevertheless, as mentioned in the text, there are too many variables influencing the behaviour of CPI and proposing such a hypothesis in the text without additional data would, most probably, only generate more confusion about the interpretation of the proxy curves. Therefore, we prefer to leave the text as is.

Lines 469–470: the discussion of the role of volcanic eruptions as a possible source of PAHs to the lake is limited to one sentence. I believe the manuscript will benefit of a short discussion to support the interpretation of measured PAHs as mainly deriving from fires, e.g. what compounds may be more related to eruptions and which ones are unambiguously related to biomass burning?

That is a good point, thank you.

As discussed below, we feel we can safely exclude that volcanic eruptions had major direct impacts on the amount and distribution of PAHs in the SVID archive.

Volcanic activity does produce PAHs (both in the gaseous and in the particulate phases) but generally their long term contribution is considered negligible, particularly in the modern world where the main PAHs source is the burning of fossil fuel (Kozak et al., 2017; Guíñez et al., 2020).

However, in an environment such as Iceland, is legitimate to ask if the several and frequent eruptions might have had a significant impact in the PAHs’ natural background and if this is detectable in our records.

Overall, due to the high temperatures involved, volcanic eruptions tend to produce medium-high molecular weight PAHs in gases and particulates (Ilyinskaya et al., 2017; Guíñez et al., 2020). Volcanic layers can contain pyrogenic (unsubstituted but also alkylated) PAHs with a molecular weight distribution resembling modern fossil fuel combustion, dominated by unsubstituted forms (Murchison and Raymond, 1989) as well as traces of nitro- and oxy-PAHs (Guíñez et al., 2020). We do not see this distribution in any of our samples, not even when they include parts of tephra layers. This suggests that (1) either our sampling method does not capture volcanic layers (maybe due to its resolution) or that (2) there is no such signal in the SVID archive.

Regarding the first hypothesis (sampling method not capturing volcanic PAHs), Kozak et al. (2017) analysed the impact of 2010 and 2011 Icelandic eruptions in Svalbard (Arctic Norway), finding high abundances of 4-5 ring PAHs in volcanic mud, great variations in the contribution of different eruptions to the total PAHs detected in sampled water, significant increases in the total abundance of PAHs during eruption years, but also that this increase in PAHs abundance as well as shifts in PAHs distribution does not seem to last beyond the eruption years. Considering the temporal resolution of our record (max 10-50 years), it is unlikely that any eruption would have impacted our record significantly: from a geological point of view, eruptions tend to be short-living, and unless they relate to a massive eruption sustained over a long period of time, they are unlikely to have a strong impact on a sample that represents 10-50 years of sedimentation. No discernible correlation arose between the PAHs curve and the detected tephra, the only exceptions being the Hekla 4 and 3 tephra layers. However, these major volcanic events, besides marking the beginning of major shifts in most proxies, correlate to the initial phase of major shifts in low (and not high nor medium) molecular weight PAHs. This suggests that, if a connection between these two eruptions and PAH shifts exists, it must be indirect and, more likely, the two events acted as a general destabilising factor in an environment already subjected to increasing cooling and erosion.

The only possible example of an increased PAH concentration due to volcanic sources could come from the sample obtained from the beginning of the G10ka tephra, which has a different composition compared to the rest of the organic-rich samples in our record. In fact, HMW PAHs seem to spike here, even if they still exhibit an overall lower concentration than LMW PAHs; this could be due to its massive nature (Óladóttir et al., 2020).

Regarding the second hypothesis (missing signal of volcanic PAHs), it is possible that no detectable volcanic PAHs were preserved in the SVID archive due to (1) its geographical location, relatively far from volcanic sources and formations (Hjartarson and Sæmundsson, 2014), and (2) the same nature of Icelandic volcanic eruptions, which are characterised by relatively low-viscosity basaltic lava rather than highly explosive pyroclastic flows (Thordarson and Höskuldsson, 2008), thus reducing the chance of ash production and deposition, particularly in distal locations such as SVID.

*We have added this information to the revised manuscript (now lines 489-524).*

Lines 454-475: observing the record, both peaks in pyroPAHs coincide with drops in *Betula* pollen, although the one at 2.8 ka BP seems unrelated to human presence since no peak in fecal markers is recorded here, while the second one at 1.5 ka is accompanied by an increase in stanols. In addition, the latter occurs in a cooler (although dry) period. Based on these observations, the first peak could be interpreted as driven by natural causes, as opposed to the second, likely also including some human influence in fire activity (see comments to figure A1), although authors are rightly very cautious in this interpretation. This would also agree with the subsequent reduction in wood availability and fire suppression discussed at the end of the section, in line with several records from the last 2 ka, where initial burning by early settlers is followed by strong changes in fire regimes and/or fire suppression/management, leading to a general decrease in fire tracers prior to the industrial era.

That is a good observation.

However, assuming that the second PAH peak were related to human activity would mean that it predated human arrival for at least 400-500 years. This hypothesis would be revolutionary and would thus need exceptional proof, which at present, it cannot be sustained with these data. If on one hand this suggestion resembles the reasoning behind the 300-year predating of colonisation in the Faroes, on the other hand, the signal to noise ratio of faecal stanols is too low to draw conclusions. We suggest this should be the focus of future research in Iceland.

#### **Figure A1:**

Consider adding smoothing lines to PAH data to aid visual interpretation of the data.

We added 3 points running average curves for each compound in figure A1.

Is benzo(e)pyrene missing from the plot?

Yes. We do not have the data for benzo(e)pyrene. It has been removed from the plot. Thank you for pointing that out.

How do authors explain spikes of benzo(a)pyrene at about 9, 7 and 4 ka BP? Perhaps the axis break should be removed to avoid misinterpreting small fluctuations of the background.

Yes. Those are single datapoints and they are noticeable only because of the exaggerated scale. The scale has been adjusted to maximise interpretation.

*The new figure A1 has been added to the manuscript (now line 630).*

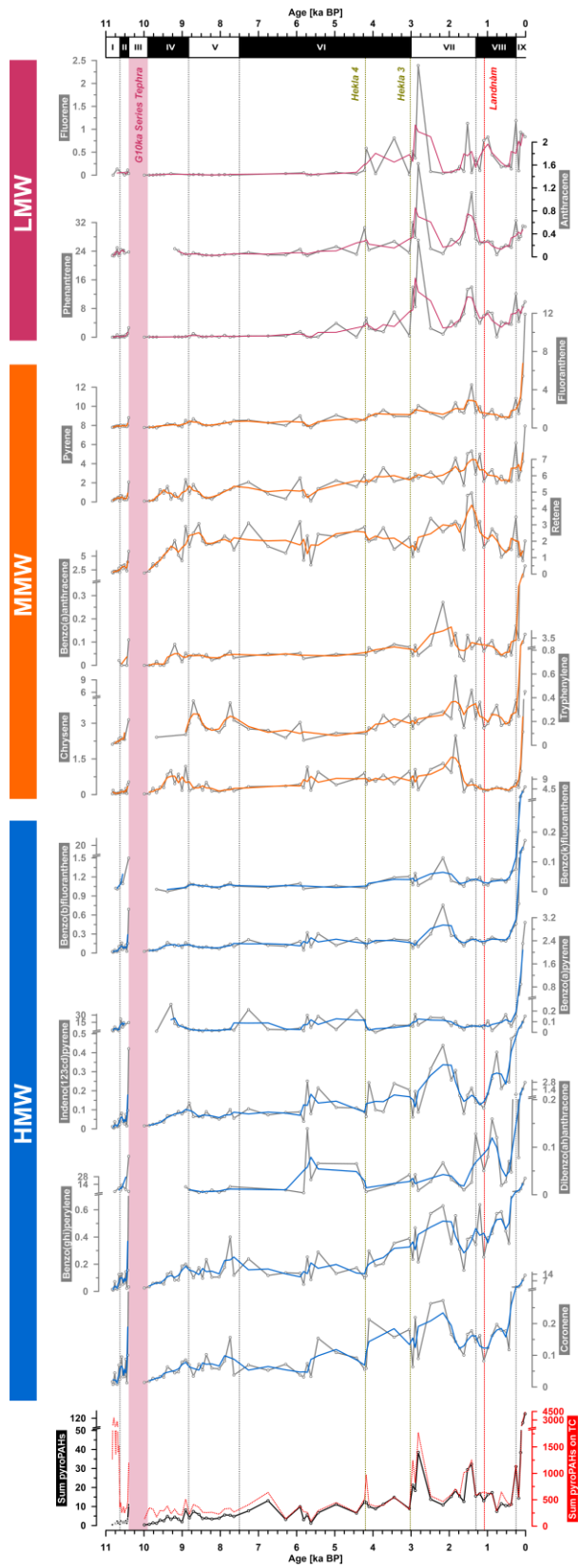


Fig. A1 updated

Retene is believed to be useful as a proxy of coniferous wood and its combustion. This compound is not considered in the discussion but looking at the plot, its trend seems different from others with a peak at the end of phase VII that is synchronous with a peak in LMW PAHs and in sterols/stanols. This peak is prior to the conventional colonization date and authors state at line 422 the stanol one does not have a sufficient s/n ratio to be confidently interpreted as a human signal. However, the synchronicity with peaks in other sites and with retene, considering the dilution effect due to the lake dimensions (resulting in low concentrations and small fluctuations from the background), might indeed link this to early occupation and deforestation of the site. Is any palaeoecological record including softwood species and charcoal available for the area?

That is a good observation.

We agree that retene's contribution to the second PAH peak is relevant. We are also aware that retene has often been linked to softwood combustion (e.g., Karp et al., 2020 and refs therein). However, softwood seems to be almost absent from Icelandic vegetational communities (e.g., Karlsdóttir et al., 2014), with the exception of a marginal presence of *Juniper* in SVID, but only from ~10 to 8 ka BP and not present during the Late Holocene (Harning et al., 2023). We thus concluded that at least in this environment, retene is not necessarily a reliable proxy for softwood burning.

## TECHNICAL CORRECTIONS

Please renumber manuscript sections starting from the Introduction (1), not the Abstract.

Yes. Thank you.

Corrected: section 1 is now the Introduction.

Line 136: "MS conditions were as same as" please correct to "the same as".

Yes. Thank you.

Corrected (now line 144).

Line 170: please correct "digestive track" to "digestive tract".

Yes. Thank you.

Corrected (now line 178).

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## SECOND REVIEWER

The manuscript from Ardenghi and collaborators proposes a complete paleoenvironmental reconstruction (including climate, fires, and potential human impacts, among others), based on organic proxies, of a Holocene lacustrine sequence in Iceland. Taking into account the studied proxies, I miss the *n*-alkane isotopes of this sequence, at least the  $\delta D$ , in order to fully understand the climate dynamics in the region. In order to (kind of) solve this situation, authors used the  $\delta D$  record of leaf waxes recovered in a nearby marine core. This manuscript is suitable for *Climate of the Past* after minor revisions.

We thank the reviewer for their positive feedback. We appreciate their recommendation for publication and address their valuable comments below.

We agree with the reviewer regarding the great potential of *n*-alkane  $\delta D$  analysis on the SVID samples and we did indeed consider it. While we concur, it was deemed excessive for inclusion in this paper and could potentially be incorporated into a subsequent work.

I think that all the proxies were correctly interpreted, but my main concern is regarding the applied methodology for the bulk organic samples, that would limit their correct use. I'm sure that this can be solved by explaining the sample pre-treatment before the TC and  $\delta^{13}C$  analyses. The main problem is that authors referred to "total carbon" analyses in the samples (this would include a mixture of the TIC and the TOC), but in section 4.4 (bulk organic proxies) the TC is used to calculate the C/N ratio; therefore, it has been used as TOC. So, when authors refer to the TC, do they refer to the total organic carbon (TOC) or to the total carbon (TC). This issue needs to be clarified, otherwise the meaning of the TC, C/N ratio and the  $\delta^{13}C$  is not clear (mixture of inorganic and organic carbon signals). If authors meant total organic carbon, carbonates should have been removed from the samples (by acid digestion) to specifically quantify organic carbon content and analyse the isotopes of the organic carbon. However, this step is not mentioned in the methodology. If no carbonate removal was performed and the real TC was estimated, the TC and  $\delta^{13}C$  values would be pointless since there can be a mixture of sources: TOC (organic) and TIC (carbonates) in the carbon (isotopic) signal. Even if there is no local carbonate precipitation in the lake (biogenic or inorganic), the direct estimation of TOC as TC is not the best approach because aeolian (carbonate) inputs can occur (e.g., Saharan dust (Varga et al., 2021) that is rich in carbonates (Rostási et al., 2022)). In any case, there are organisms with carbonate shells in this lake (ostracods: Alkalaj et al., 2019) and although there is no information about other types of carbonate precipitation, the carbonate removal must be performed before the TOC and  $\delta^{13}C$  isotope analyses.

We thank the reviewer for allowing us to clarify this point.

We did not decalcify samples as the contribution of inorganic carbon to TC in Icelandic lake sediment is considered to be neglectable (Geirsdóttir et al., 2020). Iceland's bedrock is dominantly comprised of basaltic bedrock, including the catchment of SVID (Hjartarson and Sæmundsson, 2014), meaning there is negligible carbonate available. Although some dissolved inorganic carbon (DIC) has been measured in Icelandic rivers, it is greatly outweighed by organic carbon (Kardjilov et al., 2006), whereas the amount of inorganic carbon measured in soils is negligible (Mankasingh and Gísladóttir, 2019). This is important to consider as SVID has no river inflow and water inflow is dominated by runoff from the catchment through soil. The reviewer is correct that there is some evidence for the transport of Saharan dust to Iceland within the last decade, however, there is currently no evidence of such transport during the Holocene. The additional pools of inorganic carbon from aquatic invertebrates, such as ostracods, are also considered to be minimal. Ostracods crystallise their shells in very close equilibrium to the carbon isotopes of DIC (Decrouy, 2012), which for Iceland is notably enriched relative to bulk organic matter carbon isotopes (Sveinbjörnsdóttir et al., 2020). If ostracods were a substantial contributor to the total carbon pool, we would expect lake sediment carbon isotopes to deviate from modern terrestrial and aquatic plant carbon isotope values. As Icelandic lake sediment bulk geochemistry is consistent with the fields of modern plants (see Geirsdóttir et al., 2020), inorganic carbon from aquatic invertebrates are not considered a significant contributor to the total carbon pool. Collectively, we hope that this allays any concerns by the reviewer that pre-treatment is needed for our SVID samples.

*We have added this information to the revised manuscript (now lines 109-110 and 340-353).*

### **Other minor comments:**

Study site: I suppose that the catchment basin consists of volcanic rocks, please briefly explain the geology of the catchment.

Thank you for pointing that out.

The lake is surrounded by Quaternary age basaltic lavas and glacial hyaloclastites formed by subglacial eruptions, as well as some Holocene soil with several mm-cm thick tephra layers (Hjartarson and Sæmundsson, 2014).

*This information has been added to the revised manuscript (now lines 66-68).*

Line 71. Please, explain the coring technique followed to recover the core. Where was the core stored? At what temperature to prevent OM degradation? Please specify all these points.

All this information is available in Harning et al., (2023):

*"In winter 2020, we recovered a composite 8.93-m-long sediment core (20SVID-02) from 17.4 m water depth using lake ice as a coring platform. The sediment was collected in seven drives of ~150 cm each. The core sections were subsequently stored at 4°C before opening for sediment*

subsampling.”

The core was stored in a cool room (4 °C) at the Institute of Arctic and Alpine Research, University of Colorado Boulder.

*In addition to this reference, we have added some of this information to the method section of the revised manuscript (now lines 74-76).*

Age depth model: Please specify the version of Rbacon. Fig. 2, please add Rbacon instead of Bacon. Although the age-depth model is linear and seems to be correct, there is a period between 6.2 and 9.9 ka without dates, which would increase the uncertainty for this interval. Authors should mention this in the text.

We used rbacon Version 2.5.7.

*We added this information to the revised manuscript (now line 99).*

It is true there is a lack of age control in the Early Holocene; this has been already mentioned in the original manuscript in the Result section (lines 211-212).

Please, explain the acronyms the first time they are mentioned in the text, e.g., Paq ->line 261, but the acronym is explained in line 391.

Yes, thank you.

*We have changed the text accordingly (now line 196).*

Line 475. “Discussed in Section 5.4” is correct? Because section 5.4 is related to Faecal sterol/stanols

Yes, it is 6.4.

*It corrected itself after removing the abstract number; now it reads 5.4.*

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