Reviewer comments – BG2025988 – Mineral Formation during Shipboard Ocean Alkalinity Enhancement Experiments in the North Atlantic

Overview:

The manuscript from Hashim et al. presents results from an OAE experiment conducted with liquid alkalinity (i.e., NaOH solution) during a research expedition from 2023. Using natural seawater, the alkalinity was increased using a 1M NaOH solution and the carbonate chemistry was measured throughout the experiment. Finally, the data explore the formation of CaCO₃ and compare the results with available data from the literature.

Overall, the manuscript is very reader friendly. The setting, experimental design, results and discussion are easily understandable, and the data presented here match other data available from the literature. One very interesting aspect is that in the manuscript introduces for the first time (as far as the reviewer knows) the proposed carbonate chemistry sampling techniques from Schulz et al., 2023, available in the Guide to Best Practices in Ocean Alkalinity Enhancement Research. Furthermore, the authors focused on the mineralogy of precipitated material with continuous XRD analysis of the precipitated CaCO₃. Finally, the data were fitted with other work available in the literature (Burton and Walter, 1987, Moras et al., 2022, and Mucci et al., 1989) which allow for an easy and effective comparison of the precipitation of CaCO₃ under various seawater conditions. The reviewer is supportive of the publication of the research after discussing and/or addressing the various comments and questions below, and believes that after these minor revisions, the manuscript should be considered for publication.

Comments:

Lines 37-38: I agree with the terminology "unseeded". However, I am questioning the term (pseudo)homogeneously. The use of unfiltered seawater (line 25) suggests that there may be some particles in suspension, which could have acted as seed. I think that the terminology should be slightly reviewed, emphasizing in the text that the term "unseeded" refers to the absence of CaCO₃ seeds but that there might still be some resuspended particles that could have been used as precipitation nuclei for CaCO₃

Line 38: I believe the right spelling would be "homogeneous" rather than "homogeneous"

Line 40: I believe the word "correlated" was intended rather than "correlate"

Line 85: for consistency, the TA unit should be reported as µmol kg⁻¹ throughout the text

Line 89: same as line 38

Line 105: I rather use the term magnesium hydroxide here. While I agree that brucite is the mineral form of magnesium hydroxide, Mg(OH)₂ can in some instances precipitate I an amorphous form which is not considered brucite. For ease, I would stick to magnesium hydroxide throughout the text

Line 115: I believe the sentence should read "... the ones that are more likely..."

Line 132: were the incubated water in the bags exposed to any movement (floating around, boat rocking, etc.) or was it considered static? Such absence or presence of movement may have affected the CaCO₃ precipitation kinetics and should be mentioned explicitly

Line 133: was the unfiltered seawater passed through a 1 or 2 mm mesh to get rid of bigger particles or was it fully unfiltered?

Line 147: I believe the standard notation for TA concentration is μmol kg⁻¹ without the "." in between. May need to be edited throughout the text

Line 154: same as line 85; also, it would be beneficial to have a column with the measured ΔTA to show the maximum TA reached, as well as indicate whether there are some discrepancies (maybe from early CaCO₃ precipitation after addition?)

Line 156: how was salinity measured? Because salinity does not have unit if measured on the practical salinity scale of 1978

Line 178-179: how exactly were the DIC samples taken? For stable DIC sampling, it is advised to sample the DIC in a borosilicate vial as described here using a peristaltic pump with the tubing placed at the bottom of the vial, and allowing at least half of the vial volume of overflow (Dickson et al., 2007). This section might need slightly more details.

Line 191: was the titrant ionic strength adjusted to match the samples' ionic strength?

Line 217: wouldn't calculating the various Ω at 27 °C (line 132) instead of 25 °C more suitable considering the experiments were run at ~27 °C? Or are the differences negligible?

Line 322: in caption, 3rd line, I believe there is a letter "r" missing, it should read "DIC decrease"

Line 336-337: the sentence reads that magnesite is both highly ($\Omega > 100$) and moderately ($\Omega \sim 10$) supersaturated. Please edit

Line 354: here, the figure 3 is discussed. There is one pattern that I noticed and seems interesting to me. It appears that from figure 3, the aragonite A111 and A021 signals decrease at 8.8h and15.8h after TA addition. While I may not be the more familiar with XRD analyses, I would like to have some more details as to why there is such pattern? If all the XRD samples have been handled the same way, why is there a slight decrease at these points in time? Was CaCO₃ precipitation halted during these times? Or is it only a sample artefact? I am not sure whether it is worth mentioning in the manuscript, but I would like to have the authors point of view on such pattern.

Line 372: see line 105 comments. It would be more justified to use the term magnesium hydroxide here as well

Line 388-389: if CaCO₃ coated the Mg(OH)₂ crystals, could this also explain why XRD did not reveal any? Even if the XRD covers the 2θ range of Mg(OH)₂, if these are coated with CaCO₃, the analysis result would only show CaCO₃, right?

Line 402-410: this comment does not need to be addressed, but I wanted to highlight that I really appreciated the review of the methodology and the suggested work around this unexpected loss of DIC

Line 426: same as line 38

Line 426-427: here, the work of Marion et al., 2009 could be used to determine a more accurate threshold for homogeneous precipitation given the experiment salinity and temperature

Line 433-434: same as line 38

Line 453: same as line 38

Line 463: same as line 38

Line 524-526: some works are available in the literature where they report on the inhibitory effect of various compounds under both natural and OAE setting, and could be considered as references in the manuscript (Chave and Suess, 1970, Moras et al., 2024, Pan et al., 2021, Pytkowicz, 1965)

Line 571: same as line 38