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Review of the doctoral dissertation of M.Sc. Fernando Aguado Gonzalo

As a reviewer appointed by the Scientific Council of the Institute of Oceanology of the Polish Academy of Sciences I have reviewed the doctoral dissertation entitled "Characteristics of the marine CO_2 system in Arctic waters" by M.Sc. Fernando Aguado Gonzalo. It has been supervised by dr hab. Karol Kuliński, prof. IO PAN, Marine Biogeochemistry Laboratory, Institute of Oceanology, Polish Academy of Sciences.

1. Organization, scope and aims of the thesis

Fernando Aguado Gonzalo's doctoral thesis focuses on advancing the understanding, measurement, and modelling of the marine carbonate system (pH, pCO_2, total alkalinity, and dissolved inorganic carbon), with a particular emphasis on the Arctic and stratified coastal environments. The doctoral thesis is structured in the standard way, with the following sections: Cover page (in English and Polish), Table of Contents, Acknowledgements, Funding information, Lists of publications included in the thesis, Summary (in English and Polish), References and Reprints of three scientific works constituting the thesis together with statements of co-authors. The length of the dissertation is 102 pages including the three primary publications.

The work underlying the PhD thesis has been presented in three peer-reviewed papers published in reputable international journals (IF from 2.5 to 3.4) with broad visibility and appropriate scope and readership:

1) Aguado Gonzalo, F., Stokowski, M., Koziorowska-Makuch, K., Makuch, P., Beszczyńska-Möller, A., Kukliński, P., Kuliński, K., 2024. Key processes controlling the variability of the summer marine CO2 system in Fram Strait surface waters. Frontiers in Marine Science, 11, p.1464653. doi.org/10.3389/fmars.2024.1464653. IF=3.4; 100 pt. MNISW.
2) Aguado Gonzalo, F., Koziorowska, K., Szymczycha, B., Kuliński, K., 2026. The internal consistency between calculated and measured variables of the marine carbonate system in Arctic open and coastal waters, case study: Atlantic Arctic. Marine Chemistry, p.104604. doi.org/10.1016/j.marchem.2026.104604. IF=2.5; 140 pt. MNISW.
3) Aguado Gonzalo, F., Koziorowska, K., Bromboszcz-Szczypior, L., Loginova, A., Kuliński, K., 2026. High vertical resolution measurements of pH, pCO2, Total Alkalinity and Dissolved Inorganic Carbon using a new approach: the carbonate profiler. Oceanologia, 68(1), 68101. doi.org/10.5697/VMDA8631. IF=2.7; 100 pt. MNISW.

In all publications, the Candidate is the first author, and in Publications 2 and 3 is also the corresponding author. These articles are the result of collaborative work, and based on the attached statements, it is clear that the doctoral Candidate was the principal contributor (60% in Paper 1, 75% in Paper 2, and 70% in Paper 3) to all major aspects, including the formulation of the central scientific idea, the development and application of the research methods, data collection, interpretation and presentation, and the drafting and revision of both versions of the manuscript.

Publications included in the thesis are preceded by a short summary of approximately 8 pages (the limited length is partly due to the use of a relatively small font – 10 pt). The Summary is divided into the following subsections: "Introduction and Rationale", "Research hypotheses", "Goals", "Study area", "Methods", "Overview of the results" and "Conclusions".

The "Introduction and Rationale" is generally well prepared and logically structured. This three-page section provides a compact yet comprehensive overview of the fundamental principles of marine carbonate chemistry, the

specific environmental factors that make the Arctic Ocean particularly vulnerable to ocean acidification, and the current modelling and technological limitations that motivated this research. The Author demonstrates a good familiarity with the relevant literature in his field of research. The justification for the research is strong and well grounded in current research challenges. The section clearly identifies the main research gaps, including the poor quantification of individual drivers of surface pH change in the Arctic, the lack of dedicated assessments and clear guidelines for calculating carbonate-system variables (pH, pCO_2 – carbon dioxide partial pressure, TA – total alkalinity, and DIC – dissolved inorganic carbon) in complex coastal waters, and the absence of high-resolution sampling systems capable of resolving steep biogeochemical gradients in highly stratified waters where meltwater, runoff, and primary production interact. Taken together, these points make the need for the study convincing. Overall, I assess this section positively, but I do have a few minor remarks. The statement about the pH decrease being smaller due to the presence of "strong bases" in seawater (page 5) may require clarification. The author refers to CO_3^{2-} (via Eq. 7); however, within the carbonate system, CO_3^{2-} is the conjugate base of a weak acid within the carbonate system (HCO_3^- / H_2CO_3) and therefore behaves as a weak base. Its role in seawater buffering is more accurately explained through carbonate equilibria, including protonation of CO_3^{2-} and shifts among $CO_{2(aq)}$, HCO_3^-, and CO_3^{2-}, rather than as a direct neutralization of H^+ by a "strong base". Additionally, the statement regarding biological production (page 6) is somewhat general. While biological activity can contribute to an increase in surface pH over relevant timescales, it does not remove the long-term tendency toward pH decline associated with anthropogenic CO_2 uptake and subsequent carbon cycling and remineralization processes. Finally, several literature-based statements, particularly those in the second paragraph on page 6 and the second and third paragraphs on page 7, would benefit from the inclusion of appropriate references.

The hypotheses and research goals are clearly stated and logically follow the gaps identified in the "Introduction and Rationale". Three research hypotheses are formulated, which are as follows:

1) "Despite the direct influence of sea ice meltwater on TA and DIC, NPP is the main driver of pH changes in the surface layer of the Arctic open waters during the summer season."
2) "The accuracy of the calculated carbonate system parameters using ion-pairing models is reduced in Arctic coastal areas compared to open ocean waters, limiting the quality of OA studies."
3) "High-resolution vertical sampling of the marine carbonate system parameters reveals fine-scale biogeochemical variability in the surface layer induced by continental runoff and sea ice/glacier meltwater in Arctic coastal areas."

Hypothesis no. 2 is a fairly general and quite well-anticipated claim – coastal waters and marginal ice zones are more complex than open-ocean waters because of freshwater influence and salinity and temperature gradients, so the accuracy of carbonate-system calculations often decreases (e.g. Chen et al., 2015; Woosley et al., 2017). In that sense, the hypothesis is scientifically plausible, but it is not formulated in the most precise way, since it states a broad expectation rather than a sharply defined testable prediction.
The aims are clearly formulated. In short, the dissertation aims to better understand how the marine carbonate system varies in understudied Arctic waters, especially in coastal areas where freshwater input and stratification complicate the chemistry. More specifically, it seeks to: (1) describe the spatial and interannual variability of carbonate-system parameters in the Fram Strait and identify the main physical and biological drivers; (2) assess how reliable calculated carbonate variables are in open and coastal Arctic waters and provide guidance for better calculations; and (3) develop a way to measure carbonate-system parameters with high vertical resolution in complex Arctic coastal settings.

The "Study area" section clearly explains why the Greenland Sea, Fram Strait, and Svalbard shelf/fjords are ideal natural laboratories for this dissertation. It shows the contrast between open-ocean conditions and coastal settings, and it links the regional hydrography, sea-ice influence, freshwater input, and productivity to the carbonate-system studied in the thesis. One minor comment regarding this section: I believe it would be better to link Publications 1, 2, and 3 more directly to the hypotheses or research aims rather than to the "Study area". I understand that the references in "Study area" help connect the regions to the actual datasets and field contexts, but they should be included in the hypotheses/aims section, to show which publication addresses which hypothesis or objective.

The "Methods" section is concise but sufficient for a dissertation summary. It gives the main information about the data sources, sampling areas, analytical techniques, and calculation tools used in the three publications, so the reader gets a general picture of how the research was conducted. I consider the methods to be generally well chosen for the aims of the dissertation.

The "Overview of the results" shows three main things. First, in the Fram Strait and East Greenland Sea, the carbonate system is strongly shaped by sea-ice meltwater, but the main driver of pH variability is net primary production (NPP). That is the key scientific message of Publication 1. TA and DIC follow salinity very closely, which supports conservative mixing and confirms the strong role of meltwater in setting the background carbonate chemistry. However, pH changes are more strongly linked to biological activity, so the chemical impact of meltwater and the biological control on pH are partly decoupled. Second, the calculated carbonate-system variables in Arctic waters are not always equally reliable, especially in coastal settings (Publication 2). This is important because a lot of work on Arctic acidification depends on values derived from thermodynamic models rather than direct measurements. The dissertation therefore does not just describe the system, it also proposes practical guidance for improving data quality. Third, the dissertation addresses a methodological gap: the lack of high-resolution vertical sampling. The result here is less a biogeochemical conclusion than a technical one – the new approach allows finer-scale profiling of TA, DIC, pH, and pCO_2, which is important in stratified coastal waters where steep vertical gradients may be missed by traditional Niskin bottle sampling (Publication 3). In my view, the greatest strength of the dissertation lies in this combination of process understanding and method development: it shows both what controls Arctic carbonate system variability and how it can be measured more effectively.

The "Overview of the results" section is informative and shows that the dissertation addresses its aims in a coherent way, although the depth and emphasis are not fully balanced throughout. It clearly presents the main findings of Publication 1 and links them well to the first hypothesis. The discussion of Publication 2 is also useful, since it highlights the limitations of calculated carbonate-system variables and the practical value of the proposed guideline. The contribution of Publication 3 is acknowledged, but it receives somewhat less attention than the other two studies, and its scientific significance should be highlighted more clearly.

Overall, it is a solid summary, but a few aspects merit further improvement. First, the section is quite data-dense, and much of the numerical detail is embedded in the text. The inclusion of dedicated figures or tables for this section could improve readability and make the main findings easier to grasp at a glance. Second, the summary could be further strengthened by briefly highlighting possible future research directions. A short closing remark indicating potential next steps and remaining research challenges would help place the presented results in a broader scientific context. Finally, a few minor editorial and stylistic issues are present in this section, including some inconsistencies in terminology and sentence formulation. These do not affect the interpretation of the results but could be corrected to further improve the clarity and readability of the summary.

The "Conclusions" are generally appropriate and logically follow from the results presented earlier. They are well aligned with the dissertation aims and hypotheses. They are also clearly formulated, although in some places they read more like a summary of results than a refined synthesis.

Millero, 2007 is not cited in the summary. Kerr et al., 2021 and Yang et al., 2015 appear in the English version, but are absent from in the Polish summary.

2. Evaluation of individual papers included in the thesis

I have no major reservations regarding the scientific content of the three individual papers published in peer-reviewed journals. The methods are presented in sufficient detail, the statistical analyses are sound, the results are clearly presented, the supplementary materials are informative, and the conclusions are generally well supported by the data. My questions and comments are mainly supplementary in nature; they arise from a desire to clarify certain aspects or from a desire for further elucidation. I provide my comments, questions, and minor critical remarks below, separately for each publication.

Publication 1: Aguado Gonzalo, F., Stokowski, M., Koziorowska-Makuch, K., Makuch, P., Beszczyńska-Möller, A., Kukliński, P., Kuliński, K., 2024. Key processes controlling the variability of the summer marine CO_2 system in Fram Strait surface waters. Frontiers in Marine Science, 11, p.1464653. doi.org/10.3389/fmars.2024.1464653

The first paper aimed to identify the main physical and biological drivers of marine carbonate-system variability in the Fram Strait surface waters. It was based on observations collected during summer cruises in 2019, 2020, and 2021. The research focused on quantifying how processes such as mixing of Atlantic water and sea ice meltwater, and primary production influence TA, DIC, pCO_2 and pH. The authors found that TA and DIC are predominantly controlled by the mixing of Atlantic water and sea ice meltwater. In contrast, NPP is the dominant factor driving the variability of pH and pCO_2, followed by temperature changes and freshening. The study identified interannual shifts in phytoplankton communities: for instance, colder years with reduced sea ice coincided with potential increases in calcifying species relative to non-calcifiers. The results suggest that sea ice extent and associated spread of Polar Surface Waters may be important factors shaping primary production, and thus pCO_2 and pH in the study area. Surface waters were consistently found to be undersaturated in CO_2 relative to the atmosphere, meaning the region acts as a carbon sink.

Comments and questions:

1) Could you explain the rationale for selecting 2014 as the reference year? Why this particular winter was chosen as a baseline for comparison?
2) In this study, oxygen saturation was interpreted as an indicator of net primary production. However, oxygen saturation is a relative indicator rather than a direct measure of primary production, as it is also influenced by temperature, mixing, and air–water gas exchange. In Section 3.2.2, the relationship between pCO_2, salinity, and O_2 saturation is discussed, but only for one transect from 2021. Was a similar relationship observed in 2019 and 2020? If not, could interannual differences reflect the varying influence of physical versus biological processes? Have you considered using the O_2/Ar approach to separate biological oxygen signals from physical effects (e.g. Craig and Hayward, 1987), to quantify the biologically-driven supersaturation of O_2? This approach could strengthen the interpretation of the observed relationships within the study area.
   Moreover, oxygen supersaturation integrates the net balance between O_2 production and O_2 consumption. Would it therefore be more appropriate to interpret oxygen saturation as a proxy for net community production (the gross photosynthetic production by autotrophs minus community respiration by autotrophs and heterotrophs) rather than as a proxy for net primary production?
   I would appreciate the Candidate's comments on this issue during the public defense.
3) In the other two papers included in this thesis, pCO_2 data were normalized to 1 atm, whereas in this manuscript only temperature correction was applied. Could you clarify why the atmospheric pressure correction was not applied here?
4) How transferable are the conclusions of this study beyond the Atlantic Arctic, for example, to other Arctic shelves or fjord systems? Considering that the carbonate system in Fram Strait is strongly influenced by the interaction between Atlantic and Arctic waters, sea-ice processes, and biological activity, which aspects of your findings do you consider to be region-specific and which could be applied more broadly?

Publication 2: Aguado Gonzalo, F., Koziorowska, K., Szymczycha, B., Kuliński, K., 2026. The internal consistency between calculated and measured variables of the marine carbonate system in Arctic open and coastal waters, case study: Atlantic Arctic. Marine Chemistry, p.104604. doi.org/10.1016/j.marchem.2026.104604

In the second paper, the authors evaluated the "internal consistency" of the carbonate system – the accuracy of calculating two variables when the other two are measured, and proposed guidelines for improving the quality of such calculations. The study was carried out in summer 2021 in the Fram Strait and on the west coast of Svalbard, including coastal fjords. In open-ocean waters, calculated values generally show good consistency with observations. However, in coastal waters, calculations were significantly less accurate: it was only possible to accurately calculate pH when using pCO_2 together with either TA or DIC as input parameters, and to accurately calculate pCO_2 using DIC and pH as input parameters. Therefore, in complex coastal environments, pCO_2 should be measured directly rather than calculated. Importantly, the authors found that using the TA estimated from its correlation with salinity together with pCO_2 allows accurate pH values to be calculated in both coastal and ocean waters, which opens new perspectives for long-term studies of OA.

Comments and questions:

1) One of the aims of this study was to estimate the errors in calculated marine carbonate system variables resulting from the omission of silicate and phosphate as input parameters. Could you explain the rationale behind selecting these two nutrients? In particular, did you consider whether other nutrients, such as ammonia, might have a stronger influence on carbonate system calculations due to their higher concentrations in fjord environments? The choice of phosphate, in particular, is not entirely justified, as its concentrations in seawater are generally low. Ammonia may be a more relevant parameter, especially in fjord systems around Svalbard, where its concentrations can be several times higher than those of phosphate. For example, in Kongsfjorden, during the period May-August 2024, phosphate concentrations were 0.31 ± 0.18 µmol dm^{-3} (range: 0.02–0.81 µmol dm^{-3}), whereas ammonia concentrations were 2.19 ± 1.09 µmol dm^{-3} (range: 0.01–4.58 µmol dm^{-3}) (Assmy et al., 2024).
2) Interpretation of the sign of ΔTA in waters collected close to sea ice: The discussion does not sufficiently explain how the presence of ikaite could lead to negative ΔTA values. The proposed explanation for the negative ΔTA observed near sea ice is "the presence of ikaites (a form of carbonates) in seawater"; however, it remains unclear whether this refers to an analytical artifact caused by particulate ikaite during sample storage and analysis (as described by Chen et al., 2015), or to the effect of *in situ* ikaite dissolution.
   The sampling and analytical procedure raise additional questions regarding the possible influence of ikaite or other particulate $CaCO_3$ phases on the calculated carbonate system variables. Temperature, salinity, and pCO_2 were measured continuously using the underway system, whereas discrete samples for pH, TA, and DIC were collected separately. pH was measured immediately after sampling, while TA and DIC samples were poisoned with $HgCl_2$ and analysed several weeks later. Unfiltered samples collected close to sea ice may have contained suspended mineral particles, including ikaite. If these particles dissolved during storage or TA/DIC analyses, both TA and DIC would increase, with an expected stoichiometric ratio of approximately ΔTA:ΔDIC = 2:1. However, the effect on the calculated TA depends strongly on the combination of input parameters used in CO2SYS, because pH and pCO_2 represent the original *in situ* state, whereas DIC and TA may represent a modified state after possible ikaite dissolution. In particular, the use of DIC and pH as input parameters could potentially result in negative ΔTA if the increased DIC is interpreted by the model as requiring higher alkalinity, whereas calculations based on pH and pCO_2 would be expected to produce positive ΔTA values because both parameters represent the original *in situ* conditions.
   For which carbonate system calculation pair(s) did the Candidate obtain the negative ΔTA values observed near sea ice, and does the Candidate interpret this effect as an in situ biogeochemical signal or as an analytical artifact caused by particulate ikaite in unfiltered samples?
3) Problematic citation in section 3.6: The authors state that "It is well known that there is a natural accumulation of PO_4^{3-} and DSi associated with the precipitation of ikaites during sea ice formation and their subsequent release into the surrounding waters during the melting season (Hu and Wang, 2020; Nedashkovskii et al., 2008); thus, the exceptionally high errors were a consequence of this process." The cited references do not adequately support this statement. Nedashkovskii et al. (2008) documented the enrichment and seasonal dynamics of phosphate and silicate in Arctic sea ice; however, the authors did not attribute these patterns to ikaite precipitation. Instead, they related phosphate enrichment to incorporation of the surface microlayer and subsequent biological and photochemical processes, while silicate dynamics were linked to biological uptake and biogenic silica cycling. Similarly, recent experimental evidence suggests that ikaite precipitation does not necessarily result in phosphate co-precipitation. Therefore, the statement linking phosphate and silicate accumulation specifically to ikaite precipitation is unsupported by the cited literature.

Publication 3: Aguado Gonzalo, F., Koziorowska, K., Bromboszcz-Szczypior, L., Loginova, A., Kuliński, K., 2026. High vertical resolution measurements of pH, pCO2, Total Alkalinity and Dissolved Inorganic Carbon using a new approach: the carbonate profiler. Oceanologia, 68(1), 68101. doi.org/10.5697/VMDA8631

The third paper aimed to develop and test a new high-vertical-resolution sampling approach for simultaneous measurements of pH, pCO_2, TA, and DIC. The authors describe a low-cost pumping system called the "carbonate profiler", designed to overcome the limitations of traditional sampling, which often lacks the vertical resolution needed to study highly stratified waters. The profiler was successfully tested in Arctic coastal waters around

Svalbard and in the Baltic Sea, effectively capturing steep changes in carbonate parameters within the upper 60 meters of the water column. The system can simultaneously collect discrete samples for TA, DIC, and pH while providing continuous pCO_2 measurements with a vertical resolution of 0.5 meters. Overall, the study highlights the need for a higher-resolution characterization of the carbonate system to improve predictions of the impacts of rising atmospheric CO_2. The measurements further illustrated that freshwater runoff in Arctic fjords reduces surface alkalinity, thereby lowering the capacity of water to absorb CO_2 and enhancing ocean acidification.

Comments and questions:

1) How does the Candidate see the possibility of extending the operational depth range of the carbonate profiler beyond the current limit of 60 m? In the southeastern Baltic Sea, the halocline typically occurs at a depth of approximately 60–80 m. Within and around the halocline, steep gradients in physical properties and seawater chemistry are expected. Although these deeper waters do not directly participate in air–sea CO_2 exchange, extending carbonate profiler measurements into these layers could provide valuable insights into the influence of processes occurring in the near-bottom water layer and surface sediments on the carbonate system throughout the entire water column. Furthermore, deep waters may be transported to the surface during upwelling events, thereby becoming directly involved in air–sea CO_2 exchange.
2) I understand that investigating the impact of diurnal changes in biological activity on the variability of the CO_2 system was not the focus of your study. However, in Section 3.2.1, you suggest that the relatively low surface pCO_2 reflects strong primary production. Since your measurements covered a 24-hour period with sampling every 6 hours, do you think that diel biological processes, including photosynthesis and respiration, could also have influenced the measured CO_2 system variables? If so, would you expect these processes to have a substantial effect relative to the variability associated with the Vistula River plume, or was the plume dynamics likely the dominant driver? I also noticed that Figure 4 does not indicate the time of day for each profile. Including this information might help readers assess any potential diel variability related to biological processes, even though this was not the primary objective of the study.

Although I positively evaluate the scientific content of the three publications, I noted a relatively high number of editorial inconsistencies across the manuscripts. These include formatting errors in units and symbols, inconsistent terminology, missing spaces between values and units, and minor inconsistencies between text and figures (Fig. 9 in Publication 1 and Fig. 5 in Publication 3). These issues are purely editorial and do not affect the scientific conclusions; however, they should be carefully corrected to improve the overall quality and readability of the publications.

3. Coherence of the included papers towards verifying the hypotheses and conclusions reached

The individual papers are closely linked and complement one other in addressing the broader research question defined by the hypotheses. Taken together, they form a logical and coherent dissertation.

The dissertation has led to several important conclusions, whose scientific significance I briefly discuss below:

1) Mixing of sea-ice meltwater and Atlantic water is a key factor controlling TA and DIC variability in the studied Arctic surface waters. Although the conservative behaviour of TA during mixing is expected from its definition as a charge-balance property (e.g., Broecker, 1974), this is nevertheless an important finding because it identifies the principal physical control on the marine carbonate system and helps explain how Arctic surface-water chemistry responds to changing ice cover and water-mass structure. It is also highly relevant in the context of climate change, as ongoing ice loss and altered water-mass mixing may affect surface-water buffering capacity and CO_2 uptake, as well as the conditions for primary production.
2) Net primary production is the main driver of summer pH variability in surface waters. This result highlights the strong link between biological processes and carbonate chemistry, showing that surface water pH variability is shaped mainly by phytoplankton activity rather than by physical forcing alone. In a changing climate, this is especially important because reduced ice cover, longer open-water periods, and changing nutrient conditions are likely to modify bloom development and therefore influence surface pH and the local carbon cycle.
3) Coastal Arctic waters are more prone to errors in ion-pairing model-calculated carbonate-system variables than open-ocean waters. Only three input combinations appear to yield accurate results in coastal waters: TA and pCO_2, DIC and pCO_2, and DIC and pH; other combinations should be avoided, especially pCO_2

and pH. At the same time, TA derived from salinity together with pCO_2 measurements can be used to calculate pH with high accuracy, which creates new opportunities for long-term ocean acidification studies in Arctic waters. This is an important methodological contribution, providing practical guidance for future carbonate-system calculations and improving the reliability of ocean acidification studies in complex coastal environments.

4) High-resolution vertical sampling is needed to resolve fine-scale CO_2 system variability in stratified coastal environments. The proposed carbonate profiler offers a useful tool for simultaneous high-resolution measurements of CO_2 system parameters. This is significant because the profiler addresses a major observational gap and provides a practical solution for studying rapidly changing surface-layer processes that would otherwise be missed by conventional sampling.

Overall, the conclusions are consistent with the hypotheses and research aims, and they are supported by the results presented in the three included publications. It can be concluded that the main aims of the PhD thesis have been satisfactorily met.

References cited in the review:

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4. Recommendation

Having presented the foregoing, I hereby state that the evaluated doctoral thesis fully satisfies the requirements for doctoral dissertations set out in Article 187 of the Act of 20 July 2018 on Higher Education and Science, consolidated text: Journal of Laws of 2021, item 478, on academic degrees and scientific titles, as well as degrees and titles in the arts. The doctoral thesis presents a substantial set of new data and their interpretation, which constitutes an original solution to the research problem. The attached materials provide the basis for a positive evaluation of the Candidate's general knowledge in the field of Earth and Environmental Sciences and his ability to conduct independent research. Accordingly, I apply to the High Scientific Council of the Institute of Oceanology of the Polish Academy of Sciences to approve the dissertation and admit M.Sc. Fernando Aguado Gonzalo to the following stages of the doctoral process.

Yours sincerely,

Katarzyna Tukaszewska-Kowalczyk