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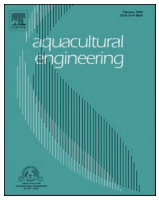

# Hydrogen sulphide dynamics in recirculating aquaculture systems with moving or fixed bed biofilters: A case study in two commercial salmon smolt producing farms in Norway

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ABSTRACT

Sudden mass mortalities of fish reared in recirculating aquaculture systems (RAS) have occurred in recent years. High total dissolved sulphide ($H_2S + HS^- + S^{2-}$) levels in the rearing water have been suggested as an underlaying factor for such mass mortalities. However, limited information is available regarding $H_2S$ dynamics in commercial aquaculture production facilities. In this case study, we present $H_2S$ dynamics in the rearing water of two commercial salmon post-smolt (150–250 g) RAS facilities equipped with different biofilters: one RAS with fixed bed biofilters (fRAS) and the other RAS with moving bed biofilters (mRAS). The farms operated at different water exchange rates and cumulative feed load but were otherwise comparable in terms of biomass and feed loading throughout the monitoring period. Self-calibrating, automatic gas-phase $H_2S$ sensors were installed at three locations per farm: after the fish tanks, after the biofilters and after the degassers and operated for a period of approximately 70 days in both farms. $H_2S$ was observed at maximum daily average of 0.6 μg/L in all locations monitored in the two RAS facilities and no significant fish mortality was reported during the monitoring period. In the fRAS, $H_2S$ concentration dynamics showed that there was a net concentration increase after the fish tanks and after the biofilters, and a net concentration decrease after the degassers. Furthermore, in the fRAS, backwashing of fixed bed biofilter chambers caused a slight increase in $H_2S$ after the biofilters. In the mRAS, there was a net positive increase in $H_2S$ after the fish tanks, and a net concentration decrease after the biofilters and degassers. Moreover, generally, $H_2S$ concentration in RAS seemed to be unrelated to feeding or fish biomass. Thus, this study suggests that the main contributing factors to $H_2S$ dynamics in RAS are biofilter design, system, and tank water exchange rates and, and potentially aeration and turbulence within each compartment.

## 1. Introduction

Intensification of fish production practices has led the industry to invest in more sustainable, controllable, and biosecure options compared to traditional open-net pen farms or flow-through systems. An alternative to complete fish production cycles in open water farms is to produce the fish in land-based, intensive systems that require less use of new water. Recirculating Aquaculture Systems (RAS) have been implemented in several Nordic countries to produce numerous marine and freshwater species (Dalsgaard et al., 2013). Norwegian salmon producers have made considerable investments in RAS to mitigate sea lice issues, prevent fish escapes, achieve better utilization of fish farming licenses in the fjords, improve water quality control and attain closer market access (Hagspiel et al., 2018).

Hydrogen sulphide ($H_2S$) has been associated with sudden mass mortalities of fish in RAS in recent years (Sommerset et al., 2022). Some literature is available on critical $H_2S$ levels associated with sudden mass mortalities, and the general recommendation is that $H_2S$ levels should be kept below 2 μg/L (Rosten et al., 2004; Sommerset et al., 2022), while concentrations below 60 μg/L are enough to hamper the fish respiratory capacity (Bergstedt and Skov, 2023). Sulphides are produced by anaerobic-anoxic microbial consumption of organic matter, a process which occurs in environments where oxygen, nitrite and nitrate are quickly depleted, and sulphate is abundant (Henze et al., 2008; Sikora et al., 2017). Sulphate is a precursor of sulphide in microbial respiration, which means that the risk for sulphide production is higher in more

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saline environments (Letelier-Gordo et al., 2020). In aqueous solutions, sulphide species can exist as either $H_2S$, $HS^-$ or $S^{2-}$, depending on pH, temperature and salinity of the water (Eaton et al., 2000). pH is the strongest driver of the sulphides speciation, and in typical aquaculture rearing water pH (6.5–8.0), the dominant sulphide species will be the toxic $H_2S$ or $HS^-$. There is, limited information regarding background $H_2S$ levels in commercial aquaculture operations, even though it is known that potential $H_2S$ production can occur within the whole RAS, e.g., from accumulated sludge, non-moving biofilter media, deep anoxic biofilm layers (e.g. Letelier-Gordo et al., 2020; Rojas-Tirado et al., 2021; Schwermer et al., 2010).

For ammonia transformation into nitrate, the majority of commercial aquaculture sites uses either fixed bed biofilter technology, moving bed biofilter technology, or a combination of both (Eding et al., 2006; Gutierrez-Wing and Malone, 2006). The two types of biofilter are different due to the way that water circulates within them and the positioning of the biofilter media: fixed beds employ static biofilter media with a plug-flow type of water movement, while mechanical stirring or strong aeration keep the media and water completely mixed in moving bed biofilters (Malone and Pfeiffer, 2006; Rusten et al., 2006). The mixing in moving beds causes constant shearing of biofilm within the biofilter media, maintaining mostly active nitrifying biofilm within its boundaries but releasing particles in the process (Fernandes et al., 2017; Pulkkinen et al., 2019; Rusten et al., 2006). A combination of static media with a plug-flow type of water circuit, creates good conditions for particle entrapment and extra bacterial growth within fixed beds (Fernandes et al., 2017; Golz et al., 1999; Pulkkinen et al., 2019). Generally, fixed beds have higher nitrification capacity than moving beds due to particle entrapment and the potential extra biofilm growth, but also a higher potential for heterotrophic processes, such as sulphate reduction to sulphide (Rojas-Tirado et al., 2021; Suhr and Pedersen, 2010). To mitigate the negative effects of uncontrolled biofilm growth, fixed beds are periodically backwashed – a routine that is associated with short-term reduction in nitrification efficiency and the potential release of particles into the system. (Eding et al., 2006; Golz et al., 1999; Gutierrez-Wing and Malone, 2006). Considering the above studies, it is hypothesized that $H_2S$ production can be linked to biofilter mode of operation (fixed or moving bed), and that backwashing of fixed bed biofilters may also influence $H_2S$ levels.

In this case study, hydrogen sulphide was continuously monitored at the inlet and outlet of the fish tanks and after the biofilter in two Norwegian commercial low salinity (< 3ppt) smolt producing RAS facilities. The farms monitored were nearly identical in terms of design and operational parameters, except for the use of fixed beds (fRAS)or moving beds (mRAS) biofilters. The main objective of this monitoring study was to determine background levels of hydrogen sulphide in commercial aquaculture facilities during approximately one growth cycle from pre-smolt to post-smolt.

## 2. Material and methods

### 2.1. Site selection

Two commercial RAS farms in Norway producing smolts at low salinity (< 3 ppt), were chosen as the case study sites. Water quality, in specific hydrogen sulphide in the water phase, was monitored in the smolt to post-smolt growth phase at each site and lasted for approximately 70 days in each RAS. The main equipment differences installed at each site were related to the biofilter mode of operation, and the main design criteria differences are summarized in Table 1: fRAS used fixed bed biofilters split into eight parallel chambers, totaling 993 $m^3$ volume, and 64% volumetrically filled with media (800 $m^2/m^3$); mRAS used moving bed biofilters totaling 850 $m^3$ volume, and 35% volumetrically filled with media (900 $m^2/m^3$). Operationally, the main differences between the two sites were the system hydraulic residence time (9 and 3 days in fRAS and mRAS, respectively) and the fish tank hydraulic residence time (36 and 70 min in fRAS and mRAS, respectively). Make-up water was added in the drum filter as backwash water in fRAS, and directly into the biofilter in mRAS. All eight biofilter chambers in fRAS were backwashed twice (one chamber per day) during the monitoring period in two different intervals (days 22–32 and days 50–59).

**Table 1**
Design criteria and system production metrics of the two RAS monitored within this trial.

| Parameter | Symbol | Equation | Unit | fRAS | mRAS |
|---|---|---|---|---|---|
| Total system volume | $V_s$ | - | $m^3$ | 6 426 | 5 300 |
| System flow | $Q_s$ | - | $m^3/h$ | 8 538 | 4 038 |
| MUW flow | $Q_m$ | - | $m^3/d$ | 722 | 2 026 |
| Recirculation rate | RER | $(1-Q_m/Q_s)*100$ | % | 92 | 50 |
| Water Exchange Rate | WER | $Q_m/V_s*100$ | %/d | 11 | 38 |
| System Hydraulic Residence Time | $HRT_s$ | $V_s/Q_m$ | d | 9 | 3 |
| System turnover rate | - | $Q_s/V_s$ | 1/h | 1.33 | 0.76 |
| Biofilter type | - | - | - | Fixed bed | Moving bed |
| Total biofilter volume | $V_b$ | - | $m^3$ | 993 | 850 |
| Biofilter volumetric filling rate | FR | - | % | 64 | 35 |
| Total biomedia volume | $V_{media}$ | $V_b*FR$ | $m^3$ | 636 | 298 |
| Specific Surface Area | SSA | - | $m^2/m^3$ | 800 | 900 |
| Active Surface Area of biomedia | $A_{media}$ | $SSA/V_{media}$ | $m^2$ | 508,416 | 267,750 |
| Max feeding observed | $F_{max}$ | - | kg/d | 3 064 | 3 442 |
| Max cumulative feed burden | CFB | $F_{max}/Q_m$ | $kg/m^3$ | 4.24 | 1.70 |
| Rearing CFB (from $F_{max}$) | $CFB_s$ | $F_{max}/V_s$ | $kg/m^3$ | 0.48 | 0.65 |

Both farms produce salmon up to 150–250 g and can hold up to ca. 270 t of fish at peak production. During the monitoring period in each site, fish mortality remained under 0.1%. Fish health and welfare were monitored according to FISHWELL scoring system (Noble et al., 2020) and was generally good in both sites. This will not be discussed in this article.

### 2.2. Hydrogen sulphide monitoring

Aquasense™ (AQS,Searas AS, Bergen, Norway) equipment was deployed in three locations (before the fish tanks, after the fish tanks, after the biofilter), at each RAS, to monitor online the concentration of $H_2S$ (minimum detection limit of 0.1 μg $H_2S$/L, Lien et al., 2022). The system contains a gas-phase equilibrator where $H_2S$ is measured, auto-calibrates once a day and, due to intrinsic carbonate and sulfide speciation systems in water, corrects each gas measurement with online and continuous monitoring of pH, temperature, and salinity (Lien et al., 2022).

Each AQS sensor box was installed at each site as shown in Fig. 1. The locations were chosen based on potential gas production or removal locations within each farm: the fish tank, the biofilter, and the degasser. The setup, therefore, included the positioning of the AQS boxes before the fish tank/after the degasser (BFT), after the fish tanks (AFT), after the biofilter (ABF, fixed beds in fRAS and moving beds in mRAS). Make-up water (MUW) in each system was also included in the mass balance assessment, even though we did not assess the MUW quality via AQS measurements.

### 2.3. Calculations and statistical analysis

$H_2S$ data were downloaded into.csv files, processed in Microsoft

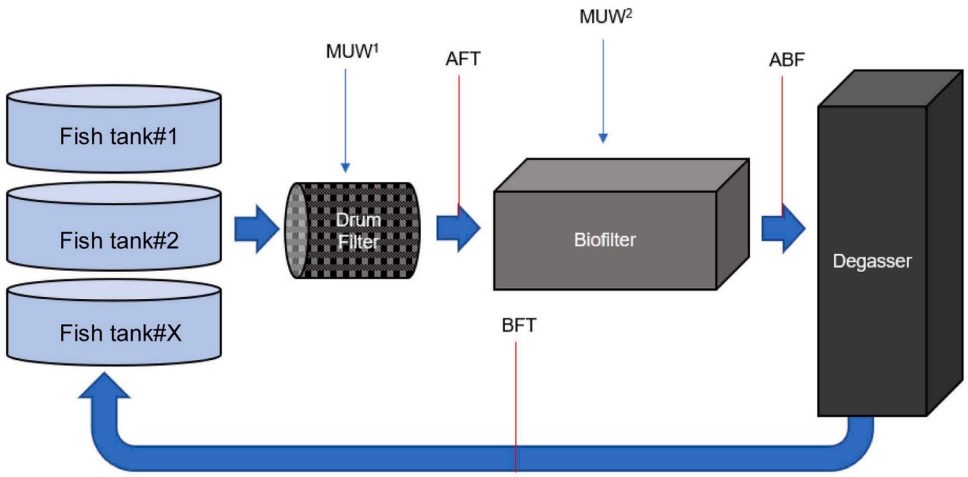


**Fig. 1.** General layout of the two RAS monitored and sampling locations of AQS equipment (red lines) and make-up water point of entry into system 1 ($MUW^1$) and system 2 ($MUW^2$). AFT – After the fish tank; ABF – After the biofilter (fixed bed in site 1 and moving bed in site 2); BFT – Before the fish tank/after the degasser.

Excel and exported into R for statistical analysis. $H_2S$ concentration data were clustered into daily averages for whole-period graphical representation, while individual data points were used for box-plot representation of backwashing effects.

Mass balances were calculated at four locations per farm, representing net differences in sensor data from each three locations of each farm, multiplied by the corresponding flow of water. There was no measurement of the MUW, but the net decrease or increase of $H_2S$ by MUW could be calculated via the $H_2S$ concentration data from each location (Fig. 1) and multiplied by the reported MUW flow. The general mass balance (MB) calculation formula for the different RAS components (fish tank, biofilter or degasser) was $MB = (C_{out}\text{-}C_{in})*Q$, with $C_{out}$ as $H_2S$ concentration measured after the component (in µg/L or $mg/m^3$), $C_{in}$ as $H_2S$ concentration measured before the component (in µg/L or $mg/m^3$), and Q as the flow of water through the component (in $m^3/h$ or $m^3/d$). In general, a positive MB result represents the increase or production of $H_2S$, while a negative MB result represents the removal or decrease of the parameter. The final mass balance formulae used for individual locations at each farm are shown in Table 2.

Data between systems were compared by one-way ANOVA and post hoc compared with Tukey HSD to detect significant differences between groups. A non-parametric ANOVA (Kruskal-Wallis) was used to compare the effects of backwashing and post hoc compared with Dunn test for between-groups analysis. A maximum *p*-value of 0.05 was used to determine statistical differences between groups.

## 3. Results

Hydrogen sulphide concentrations in fRAS (Fig. 2) were relatively stable over time at an average of 0.32 ± 0.05 µg/L, 0.40 ± 0.08 µg/L and 0.12 ± 0.02 µg/L after the fish tank, the biofilter and the degasser, respectively. On average $H_2S$ net mass increased daily by an average of 38.03 g/d and 17.78 g/d within the fish tanks and biofilters, respectively (Fig. 3). The make-up water removed 0.13 g $H_2S$/d, while the degasser removed 55.80 g $H_2S$/d.

In mRAS, $H_2S$ averaged at 0.33 ± 0.15 µg/L, 0.16 ± 0.02 µg/L and 0.03 ± 0.01 µg/L, after the fish tanks, after the biofilter and after the degasser, respectively (Fig. 2). In general, there was a net mass decrease in $H_2S$ of 16.15 g/d, 13.08 g/d and 0.33 g/d caused by the biofilters (moving beds), degassers and make-up water, respectively (Fig. 3). On the other hand, there was an average daily $H_2S$ increase of 29.56 g/d through the fish tanks.

There were two backwashing periods during the assessment of fRAS where one biofilter chamber was washed per day. The first period occurred between days 22–32, while the second period occurred between days 50–59. Both backwashing periods had a significant, although weak, effect on $H_2S$ concentrations after the biofilter (Fig. 4). In both events, the background $H_2S$ concentration increased after the biofilters during backwashing. Before the first event (days 22–32) the $H_2S$ concentration after the biofilters was 0.39 ± 0.02 µg/L, increasing to 0.42 ± 0.01 µg/L during the backwashing period, and remaining at 0.42 ± 0.01 µg/L the week after backwashing ($\chi^2_{Kruskal\text{-}Wallis} = 13\ 569$, $n_{obs} = 31\ 724$, $p < 0.001$). The results of the statistical analysis suggested that there was a significant, although weak, $H_2S$ increase between the week before and the week during backwashing ($p_{Holm\text{-}adj.} < 0.001$), and a weak significant decrease between the backwashing week and the ensuing week ($p_{Holm\text{-}adj.} = 0.031$). Statistically, the ensuing week values were still higher than the levels in the week before backwashing ($p_{Holm\text{-}adj.} < 0.001$). Before the second event (days 50–59), the $H_2S$ concentration after the biofilters was 0.40 ± 0.02 µg/L, increasing to 0.42 ± 0.02 µg/L during backwashing, and remaining at 0.42 ± 0.03 µg/L in the ensuing week ($\chi^2_{Kruskal\text{-}Wallis} = 8\ 179$, $n_{obs} = 28\ 875$, $p < 0.001$). The results of the statistical analysis suggested that there was a significant, although weak, $H_2S$ increase between the week before and the week during backwashing ($p_{Holm\text{-}adj.} < 0.001$), and a significant $H_2S$ decrease between the backwashing week and the ensuing week ($p_{Holm\text{-}adj.} < 0.031$). Statistically, the ensuing week values were still higher than the levels in the week before backwashing ($p_{Holm\text{-}adj.} < 0.001$).

**Table 2**
Mass balance formulae used per location per farm, as defined by the measuring positions of AQS equipment. AFT - After the fish tank; BFT - Before the fish tank; ABF – After the biofilter; Qs – System flow; Qm – Make-up water flow.

| Location | Abbreviation | fRAS | mRAS |
|---|---|---|---|
| Fish tank | FT | = ([AFT]-[BFT])*Qs-MUW | = ([AFT]-[BFT])*Qs |
| Biofilter | BF | = ([ABF]-[AFT])*Qs | = ([ABF]-[AFT])*Qs-MUW |
| Degasser | DG | = ([BFT]-[ABF])*Qs | = ([BFT]-[ABF])*Qs |
| Make-up water | MUW | = ([AFT]-[BFT])*Qm | = ([ABF]-[AFT])*Qm |

## 4. Discussion

$H_2S$ was continuously present in all measured locations of the two RAS, at concentrations below 1 µg/L. Some reference values exist for acute toxicity of $H_2S$ in different fish species and life stages (Rosten et al., 2004), and little information exists on how sublethal $H_2S$ affects salmon in the parr to smolt life stage (e.g. Bergstedt and Skov, 2023). Still, $H_2S$ concentration < 2 µg/L have been suggested to not affect fish

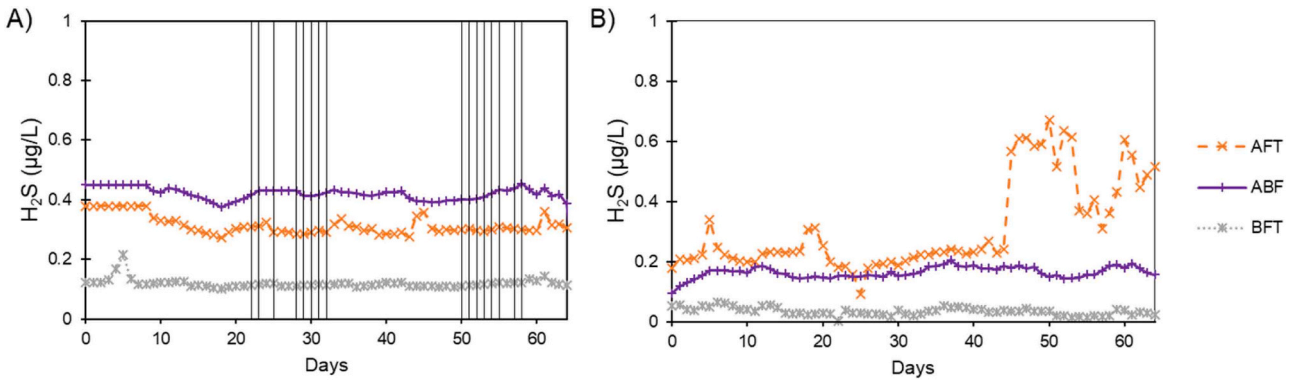


**Fig. 2.** Daily $H_2S$ average concentration at A) fRAS and B) mRAS. AQS measurement from each point represented in orange (AFT), purple (ABF) and grey (BFT). Black vertical lines represent backwashing days (one chamber per day) in fRAS.

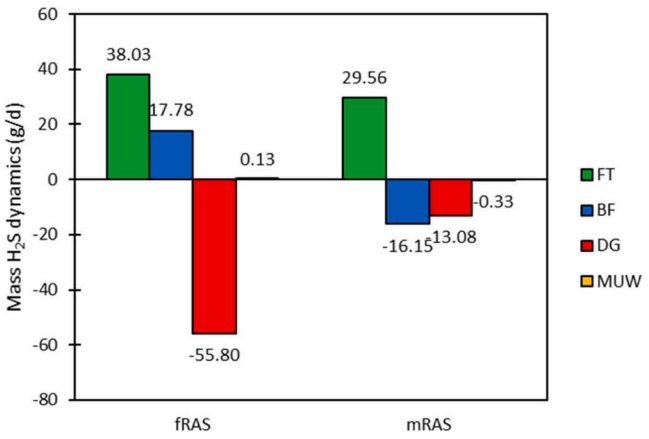


**Fig. 3.** Mass hydrogen sulphide dynamics through the fish tank (green bars), biofilter (blue bars), degasser (red bars) and make-up water (yellow bars), at fRAS and mRAS. Positive values mean net mass increase, and negative values mean net mass decrease.

health and welfare (Rosten et al., 2004; Sommerset et al., 2022). This is in line with the fact that no significant mortality or fish health and welfare issues were observed in this study. $H_2S$ levels were rather constant after the fish tanks in the fRAS over the post smolt production phase, suggesting that $H_2S$ levels are unaffected by the increase in biomass or feed load (data not shown). A similar pattern was observed in the fish tanks in the mRAS facility even though the hydraulic residence time was longer, which can lead to an increased proportion of settled solids within the tanks, and shortage of oxygen and increased anaerobic processes (Letelier-Gordo et al., 2020; Rojas-Tirado et al., 2021; Schwermer et al., 2010). Furthermore, a sudden drop in pH on day 44 caused an increase in $H_2S$ concentrations in mRAS. This is probably related to the fact that $H_2S$ is the dominant sulphide form at pH below 7 (Bergstedt et al., 2022; Lien et al., 2022; Lindholm-Lehto, 2023), since total sulphide concentration remained unchanged (data not shown).

$H_2S$ concentrations were higher after the biofilters in fRAS compared to mRAS. This is probably due to a combination effect of dilution via MUW and degassing by aeration inside the moving beds (Summerfelt et al., 2000; Colt et al., 2009) in mRAS. In addition, there was a net production of $H_2S$ in fRAS. This is in accordance with the results from the study performed by Rojas-Tirado et al. (2021), showing that $H_2S$ production rate is higher in biofilters with non-moving media. Entrapment of particles and short-circuiting of water flows within fixed beds (Fernandes et al., 2017; Pulkkinen et al., 2019) leads to a higher heterotrophic microorganism activity rate, potentially also generating anaerobic or anoxic zones where $H_2S$ can be produced (Letelier-Gordo et al., 2020; Rojas-Tirado et al., 2021). In addition, the 3.5 times higher water exchange rate (38% daily water renewal in mRAS compared to 11% in fRAS) may have contributed to the lower $H_2S$ concentrations in mRAS. However, $H_2S$ was not measured in MUW samples, and the 3.5 times higher water exchange rate does not fully explain the differences in $H_2S$ dynamics through the fish tanks and biofilters in this study. The most likely explanation is that MUW always had a dilution effect, but the effect of each component (biofilter, fish tanks, drum filter) where MUW was added in each site, was stronger than that of MUW and, therefore, masked its effect.

$H_2S$ production was relatively constant over time in the fixed bed biofilters, with minor peaks associated with backwashing events. Generally, backwashing disrupts the outer layers of the biofilter biofilm, minimizing uncontrollable heterotrophic microbial growth, but may

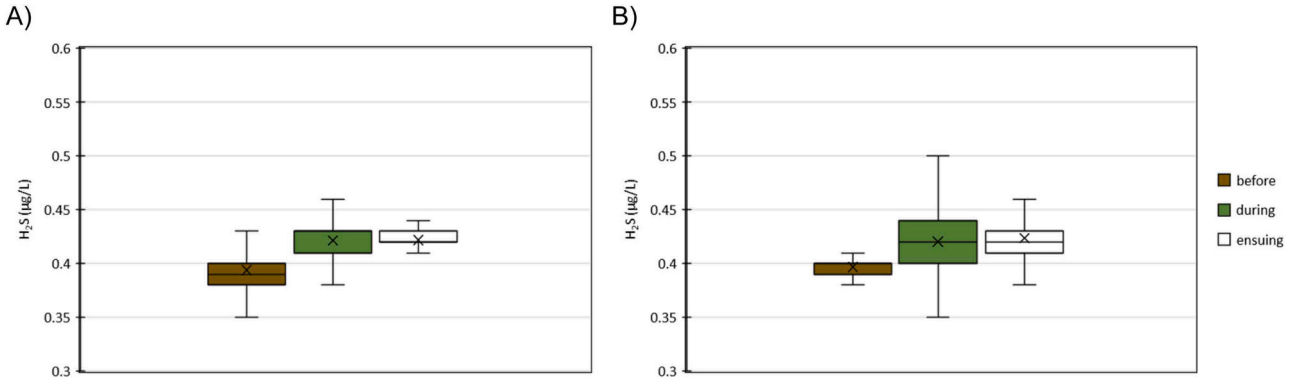


**Fig. 4.** Average $H_2S$ concentration measured after the biofilters of fRAS in the preceding (brown boxplot), during (green boxplot) or ensuing (white boxplot) weeks of backwashing, in the A) first backwashing period (days 22–32), and B) second backwashing period (days 50–59).

release particles back into the system. It may also promote the establishment of opportunistic bacteria in the newly disrupted niches within the biofilm (Blancheton et al., 2013; Golz et al., 1999). According to our results, backwashing of fixed bed biofilters seems to release $H_2S$ that may be trapped within backwashed material, or cause pulse production of $H_2S$ from released organic material. Backwashing of fixed bed biofilters is usually regarded as beneficial to the functioning of the reactors, and the benefits have historically outweighed the detrimental effects (Gutierrez-Wing and Malone, 2006; Malone and Pfeiffer, 2006). That our results demonstrate pulse $H_2S$ release from fixed bed media in association with backwashing stresses that potential release of $H_2S$ should be included in management of RAS using fixed bed biofilters.

In terms of mass dynamics of $H_2S$, removal or generation were relatively low compared to production or consumption of other gases in RAS. At maximum biomass (270 t) in each RAS and assuming a standard metabolic rate of approximately 120 mg $O_2$/kg fish/h (Khan et al., 2018), the fish would have consumed ca. 778 kg $O_2$/d and produced ca. 1 070 kg $CO_2$/d. This represents $20 \times 10^3$ and $28 \times 10^3$ times larger $O_2$ and $CO_2$ budgets, respectively, than that for $H_2S$ in fRAS; and $26 \times 10^3$ and $36 \times 10^3$ times larger $O_2$ and $CO_2$ budgets, respectively, than that for $H_2S$ in mRAS. RAS are traditionally designed to cope with these oxygen requirements and degassing needs, and aeration and degassing mechanisms to sustain fish respiration and keep $CO_2$ at acceptable levels seem to have helped maintain $H_2S$ concentrations at low levels. Traditional degassing equipment seems to be able to remove $H_2S$ in low salinity water (Lien et al., 2022). The measured levels after degassing equipment in this study were relatively constant throughout the measurements in both sites. This means that, probably, the degassers of the two RAS assessed had reached a balance with the $H_2S$ production units. Due to $H_2S$ being more soluble in water than $CO_2$ and $O_2$, it is possible that, when designing degassers for a certain threshold of $CO_2$, the maximum design capacity is not able to cope with removing such low $H_2S$ concentrations as observed in the measurements presented in this study. Potentially, the results observed in this study would not translate into seawater systems, as 1) there is more sulphate (the precursor of $H_2S$ production via microbial sulphate reduction) in seawater (Letelier-Gordo et al., 2020); 2) decreased gases solubility at higher salinities (Duan et al., 2007); 3) decreases gas stripping efficiency by conventional degassers at higher salinities (Moran, 2010).

## 5. Conclusions

In this study, $H_2S$ was present at all times in all sampling points in two commercial RAS facilities producing salmon smolts in Norway. The levels were below the suggested chronic and acute toxicity thresholds. In accordance with this, there was no significant mortality or welfare issues observed, and fish appetite and biomass were reported according to the operational expectations in the two different RAS.

$H_2S$ was found in rearing water of both sites at concentrations that never surpassed 1 μg/L (maximum daily average of 0.6 μg/L). In this case study, $H_2S$ dynamics were affected by fish tank hydraulic residence time and water flows, biofilter mode of operation (fixed or moving bed) and degassing equipment: fixed bed biofilters produced more hydrogen sulphide than moving beds, potentially due to a combination of particle entrapment, increased organic matter consumption and uncontrolled heterotrophic microbial growth. Additionally, there was a short-term increase in $H_2S$ levels after backwashing of fixed beds in fRAS, potentially due to particulate material release, or disturbance of $H_2S$ production zones.

To our knowledge, this is the first study to showcase $H_2S$ concentration levels in commercial RAS facilities producing salmon smolts in low salinity environments. However, it is important to keep in mind that this case study included no control of variables or replication. Accordingly, further studies are needed to understand and generalize $H_2S$ levels and dynamics in commercial operations. Thus, this case study should be followed by deeper studies on the same technological effects on $H_2S$ dynamics, as well as monitoring background levels with other fish species, in different environments (e.g., higher salinities) and in other geographical regions, also including measurements on individual tanks, other technologies (e.g., ozonation, protein skimmers) and make-up water.

## CRediT authorship contribution statement

**Steigum Endre:** Formal analysis, Investigation, Writing – review & editing. **Fernandes Paulo Mira:** Conceptualization, Data curation, Formal analysis, Investigation, Methodology, Supervision, Visualization, Writing – original draft, Writing – review & editing. **Rojas-Tirado Paula Andrea:** Conceptualization, Funding acquisition, Investigation, Methodology, Writing – review & editing. **Höglund Erik:** Formal analysis, Funding acquisition, Visualization, Writing – original draft, Writing – review & editing. **Åtland Åse:** Conceptualization, Funding acquisition, Project administration, Resources, Writing – review & editing.

## Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

## Data availability

Data will be made available on request.

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