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Statistical Calibration of a Portable pH Sensor for Coastal Monitoring: A Case Study in Mauritius

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Abstract: The application of sensors for measuring physico-chemical parameters like sea surface temperature and pH enables rapid, reliable long-term data collection. This is particularly crucial for Small-Island Developing States, whose marine ecosystems are vulnerable to climate change and ocean acidification. This study compared the pH data recorded by a portable pH sensor to those determined in the laboratory (UV-Vis spectrophotometric method) at Flic-en-Flac, a lagoon in the west coast of Mauritius. The two methods were compared using the Bland-Altman analysis and a multiple linear regression. It was noted that without adjusting for temperature differences between the pH sensor and a temperature probe, the pH results indicate a Pearson's correlation coefficient of 0.473 ($r^2 = 0.224$, $p = 0.00135$). In contrast, when temperature discrepancies between the pH sensor and the temperature probe were accounted for, the two methods yielded comparable results (Pearson's $r = 0.919$, $r^2 = 0.959$, $p < 0.001$). With the temperature adjustments, the data provided by the pH sensor are considered reliable and can be a complementary method to laboratory-based pH measurement.

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Keywords: Ocean acidification; portable pH sensor; UV-Vis spectroscopy; SIDS; Mauritius

1. Introduction

As the major sink of atmospheric carbon dioxide, the oceans convert dissolved CO_2 into carbonic acid, which further dissociates into protons (H^+), bicarbonate (HCO_3^-), and carbonate (CO_3^{2-}) ions [1,2]. However, high levels of carbon dioxide in the atmosphere have contributed to both a reduction in sea water pH by about 0.1 units since more H^+ ions are released; a phenomenon referred to as ocean acidification (OA), and to an increase in sea water temperature due to enhanced greenhouse effect [3–7]. Adverse impacts of OA on the marine ecosystem include the lowering in the calcification rates of shell-forming organisms and coral bleaching, jeopardizing food security and causing a significant loss in revenues [3,8–10].

Research on the monitoring and evaluation of OA in terms of physico-chemical parameters (mainly salinity, sea surface temperature and pH) is of paramount importance for understanding the present status and foreseeing future challenges related to ocean health, [11], consistent with Sustainable Development Goal 14.3.1 (SDG 14.3.1) [12]. In most cases the available in situ oceanographic data are mainly scarce and/or virtually non-existent for modelling studies [13]. In addition, low spatial and temporal resolution of satellite data contributes to poor OA prediction in the long run [9,11]. In situ and laboratory measurements of these parameters are labor intensive and time consuming. Sensors may provide a sustainable alternative in the continuous collection of reliable spatial and temporal data over a defined time span [14,15]. In the field, glass electrodes are commonly used to monitor pH, but they are quite fragile, costly and have to be often calibrated. On the other hand, it is reported that spectrophotometric sensors using an indicator dye have higher precision and accuracy in measuring pH while accounting for ionic strength and turbidity [16–20].

Like all other Small Island Developing States (SIDS), Mauritius is not spared of the impacts of OA. With its huge exclusive economic zone (2.3 million km^2), ocean economy has become a key economic pillar and the activities involved are fisheries, aquaculture, tourism and shipping [21]. Moreover, Mauritius exports a variety of seafood products such as canned tuna, salted fish, smoked fish and frozen shrimps [22]. The effects of OA can indeed be detrimental to such economic pillar. Consequently, it is vital for a continuous monitoring of the physico-chemical parameters mentioned above. However, sample collection cannot be carried out on a 24-hr period, creating a gap in the dataset and, therefore, the variation of these parameters during the diurnal cycle cannot be studied. This is where sensors play a key role in obtaining the missing data. The data collected can be translated in a way that policy makers understand the impending threat of OA and eventually, amend

and adopt relevant policies, and launch sensitization campaigns at an early stage to avoid an OA crisis. In so doing, Mauritius can engage in capacity building to help other SIDS in the region to tackle OA.

1.1 Pilot study conducted in a Mauritian lagoon

As a pilot study, our aim was to assess the performance of a commercial spectrophotometric pH sensor, suited for shallow water (< 2 m) at Flic-en-Flac, (FF), a lagoon on the western coast of Mauritius. The sensor's operational salinity range (25–40) aligns with the salinity levels (around 35 ‰) commonly observed in Mauritian lagoons [23]. The objectives were to

- (i) have the sensor measuring pH ($\text{pH}_{\text{sensor}}$) at the same time samples were collected;
- (ii) compare and correlate $\text{pH}_{\text{sensor}}$ values to those measured in the laboratory (pH_{lab})
- (iii) conduct statistical analyses (multiple linear regression and Two-way ANCOVA) to compare the variability of $\text{pH}_{\text{sensor}}$ values with respect to both pH_{lab} values and seasons.

In terms of innovation, this study confirms whether reliable pH data are being collected by the sensor on a longer timescale and that in case of deviation from quality check (pH_{lab}), the sensor data can be re-calibrated statistically.

2. Methods

Sampling was conducted at FF (20.27°S, 57.37°E, Figure 1), a lagoon located on the western coast of Mauritius in 2023 - 2024. Being a tropical island, the climate is influenced by two seasons, summer (November to April) and winter (May to October).

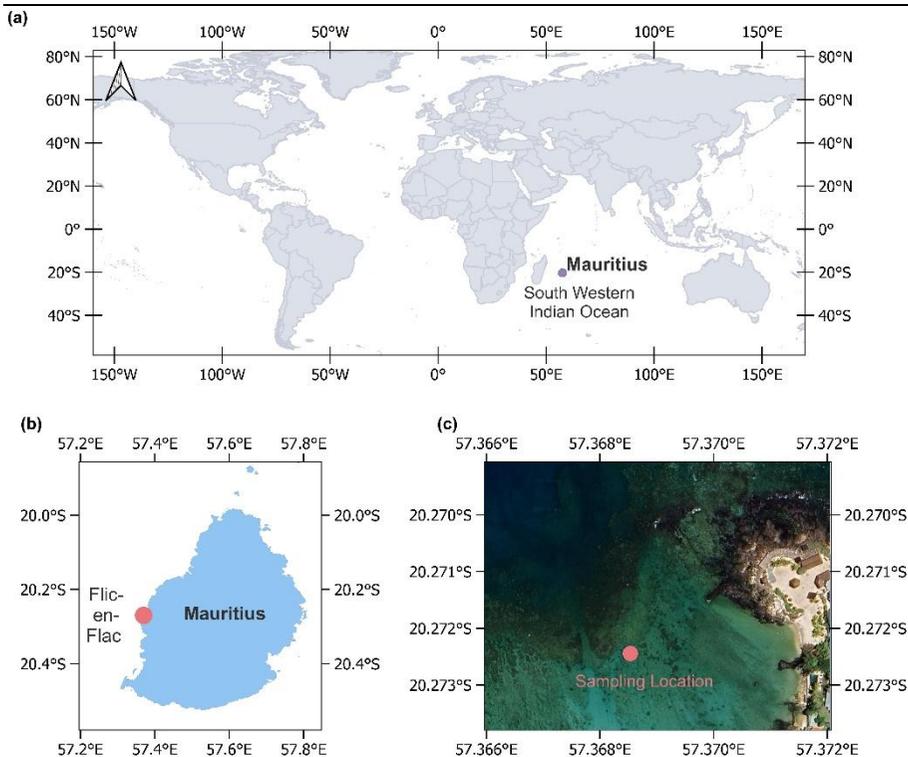


Figure 1. (a) The South Western Indian Ocean basin, (b) Map of Mauritius and (c) The specific sampling location at Flic-en-Flac

Discrete seawater samples ($n = 43$) were collected at a depth of 2 m, using 250-mL borosilicate bottles filled from a 2-L Niskin bottle. These samples were treated with 200 μL of saturated mercury (II) chloride solution [17]. The in situ sea surface temperature (SST) and sea surface salinity (SSS) were recorded using a handheld Fluke 52-II thermocouple ($\pm 0.3\text{ }^{\circ}\text{C}$) and a Milwaukee MA887 Digital Salinometer featuring automatic temperature compensation ($\pm 0.1\text{ }^{\circ}\text{C}$, $\pm 2\text{ }‰$), both of which were calibrated daily with deionized water. The pH sensor was also deployed at FF at 2 m depth to measure pH, denoted as $\text{pH}_{\text{sensor}}$. The latter was measured on the half hour mark as from 9.30 am for three hours. Sampling of sea water was also carried out in duplicate at the same time the sensor was measuring pH. The temperatures recorded by the Fluke 52-II thermocouple and the pH sensor are denoted as T_{probe} and T_{sensor} , respectively.

After sampling, the discrete samples were transported to the laboratory and analyzed on the same day. A UV-Vis spectrophotometric method was employed to determine pH, denoted as pH_{lab} , using a Biochrom Libra S22 UV-Vis spectrophotometer [24,25]. The in situ pH values were computed using CO2SYS (Excel v2.5) and the variables were the Fluke thermocouple measured temperatures and salinity [26]. The laboratory analyses were

validated using certified reference materials from the Scripps Institute of Oceanography, USA. The accuracy for pH was determined to be ± 0.0152 .

Bland-Altman analysis [27] was conducted to test the agreement between $\text{pH}_{\text{sensor}}$ and pH_{lab} , prior to performing a multiple linear regression analysis [28] in order to correct $\text{pH}_{\text{sensor}}$ values in case of deviation from pH_{lab} . The assumptions towards normality, homoscedasticity, and independence of residuals were systematically assessed using the Shapiro-Wilk test [29], the Breusch-Pagan test [30], and the Durbin-Watson test [31], respectively. In addition, assumption for multicollinearity between $\text{pH}_{\text{sensor}}$ and change in temperature was verified through the calculation of variance inflation factors (VIF), with values below 5 indicating acceptable levels of collinearity. To investigate variations of both laboratory based and sensor pH with respect to seasons (summer to winter), a two-way ANCOVA was performed.

3. Results

A salinity of almost constant 35 ‰ was noted at all times. Figure 2 below shows the variation of $\text{pH}_{\text{sensor}}$ and pH_{lab} against SST.

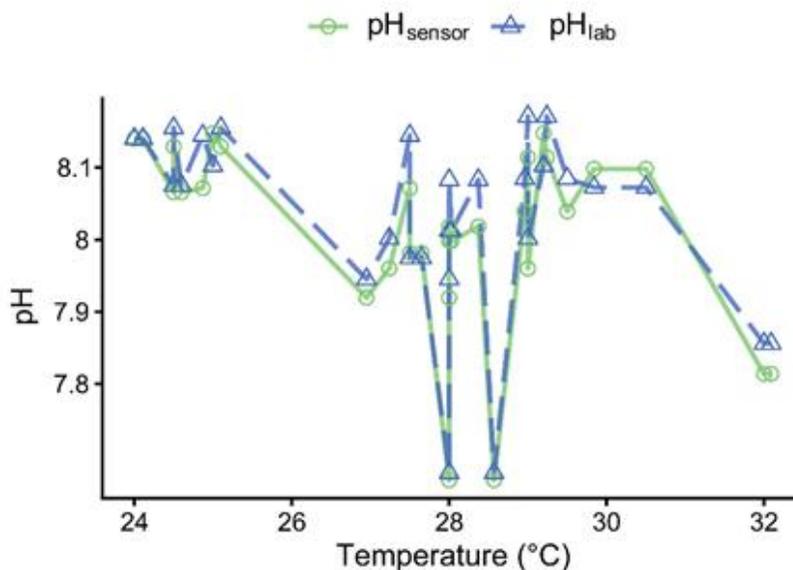


Figure 2. Variation of $\text{pH}_{\text{sensor}}$ and pH_{lab} against SST

From Figure 2, it can be noted that the variation trend of $\text{pH}_{\text{sensor}}$ against SST was similar to that of pH_{lab} throughout the sampling period.

To check for agreement between $\text{pH}_{\text{sensor}}$ and pH_{lab} , a Bland-Altman analysis was carried out. The mean $\text{pH}_{\text{sensor}}$ (8.085 ± 0.1350) and mean pH_{lab}

(8.074 ± 0.0941) were so well in agreement, as shown in the Bland-Altman plot (Figure 3).

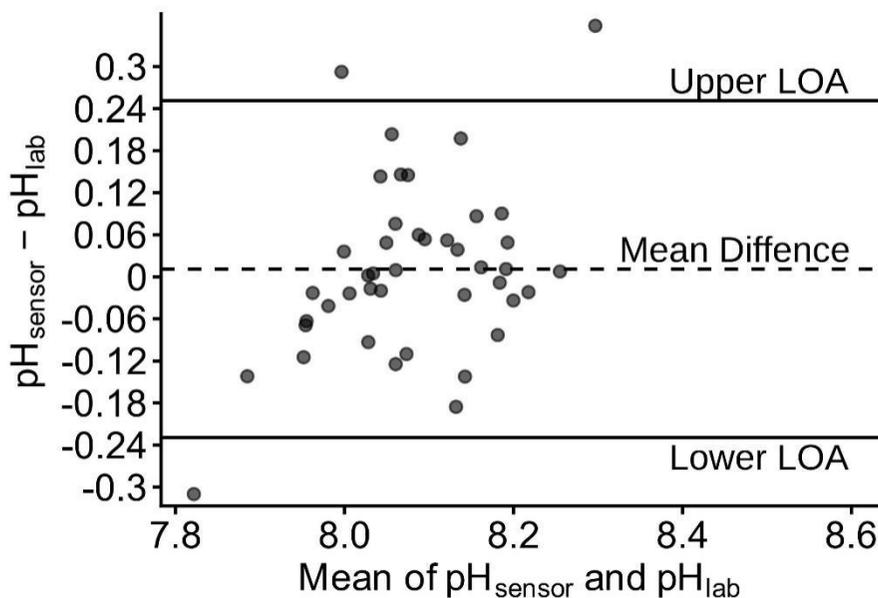


Figure 3. The Bland-Altman plot between $\text{pH}_{\text{sensor}}$ and pH_{lab}

From Figure 3, the mean difference between the two pH sets was 0.0109, indicating some systematic difference. The limits of agreement (LOA) ranged from -0.230 to 0.251 . From the plot, there is no clear pattern or proportional bias, indicating a consistent agreement across the range of pH values. Discrepancies arose mostly due to the difference in temperature measurements of the pH sensor (27.6 ± 4.15 °C) and the handheld thermocouple (27.8 ± 2.82 °C) since salinity was almost constant at 35 ‰ and that other factors are considered to have negligible impact on the pH differences observed between the two methods of pH measurement.

Repeating the Bland-Altman agreement analysis with $\text{pH}_{\text{sensor}, T_{\text{probe}}}$ ($\text{pH}_{\text{sensor}}$ corrected according to temperature of handheld Fluke 52-II thermocouple) instead of $\text{pH}_{\text{sensor}}$ reveals that the systematic difference has improved to 0.00340, with the LOA ranging from -0.237 to 0.244 , as shown in Figure 4.

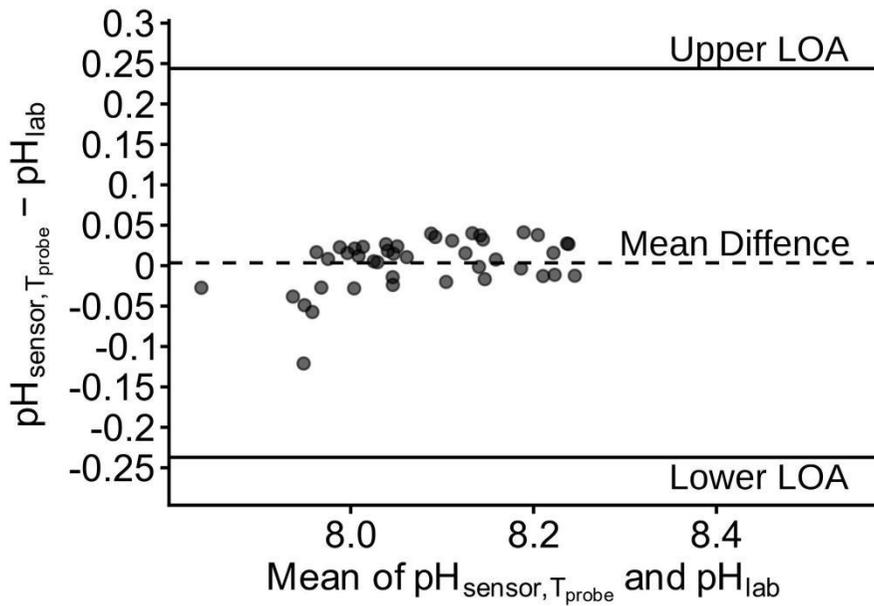


Figure 4. The Bland-Altman plot between $\text{pH}_{\text{sensor}, T_{\text{probe}}}$ and pH_{lab}

Now these findings suggest that the sensor provided pH values comparable to the laboratory pH measurements.

Consequently, in the first place, a linear correlation was performed between $\text{pH}_{\text{sensor}}$ and pH_{lab} (Figure 5), which resulted in a rather poor Pearson's correlation coefficient of 0.473 ($r^2 = 0.224$, $p = 0.00135$).

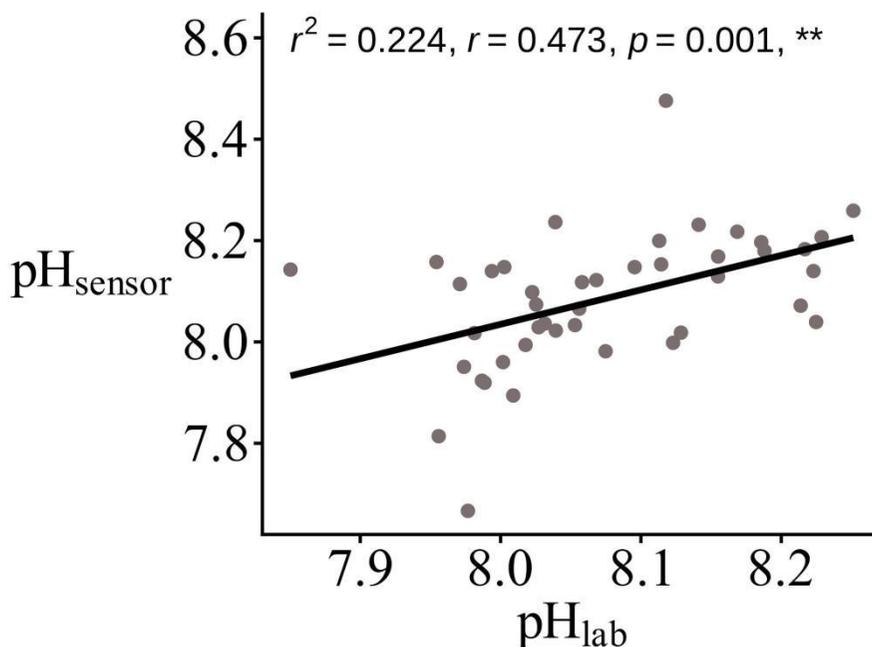


Figure 5. Linear correlation between $\text{pH}_{\text{sensor}}$ and pH_{lab}

This result conveys an important deduction. The two methods cannot be compared directly without accounting for the different measured temperatures, T_{sensor} and T_{probe} .

To account for the influence of temperature differences on pH calculations, $\text{pH}_{\text{sensor}}$ was recalculated at T_{probe} using CO2SYS, and is referred to as $\text{pH}_{\text{sensor}, T_{\text{probe}}}$. This adjustment results in a closer linear relationship between the two means, with $\text{pH}_{\text{sensor}, T_{\text{probe}}}$ averaging 8.078 ± 0.1070 , compared to pH_{lab} (8.074 ± 0.094). The linear correlation between $\text{pH}_{\text{sensor}, T_{\text{probe}}}$ and pH_{lab} is shown in Figure 6.

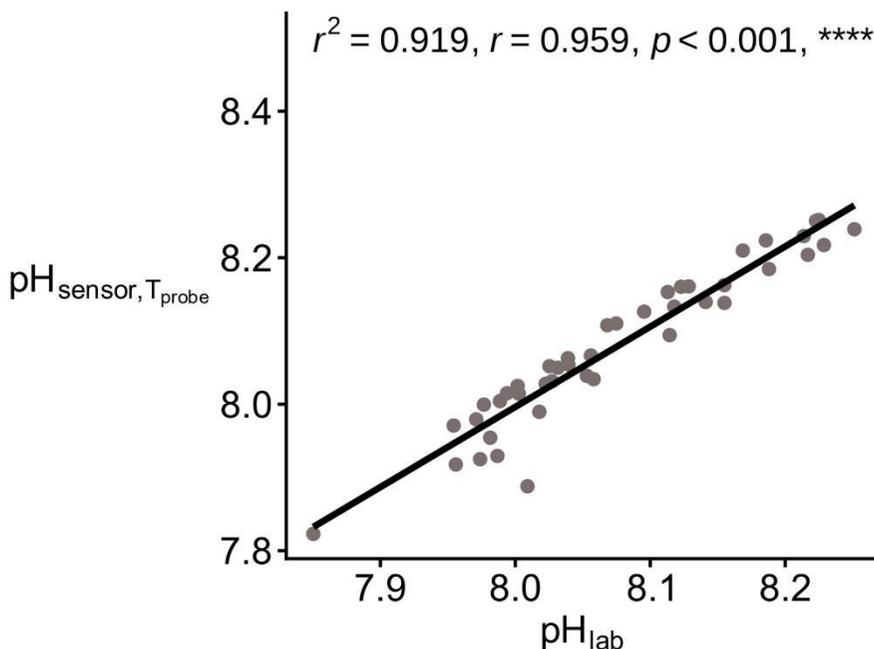


Figure 6. Linear correlation between $\text{pH}_{\text{sensor}, T_{\text{probe}}}$ and pH_{lab}

From Figure 6, it can be observed that a significant linear relationship ($p < 0.001$) is obtained between $\text{pH}_{\text{sensor}, T_{\text{probe}}}$ and pH_{lab} , with a Pearson's correlation coefficient of 0.959 ($r^2=0.919, p < 0.001$).

To correct for the disparity in $\text{pH}_{\text{sensor}}$ data compared to pH_{lab} through statistical calibration, a multiple linear regression was conducted to account for the temperature differences ($\Delta T = T_{\text{sensor}} - T_{\text{probe}}$). All assumptions (Shapiro-Wilk test, Breusch-Pagan test and Durbin-Watson test) of multiple linear regression were met. The Shapiro-Wilk test indicated that the residuals were normally distributed ($p = 0.852$). The Breusch-Pagan test confirmed homoscedasticity ($p = 0.536$), and the Durbin-Watson test suggested independence of residuals ($p = 0.736$). All VIF values were below 5 ($\text{pH}_{\text{sensor}} = 1.00$, and $\Delta T = 1.00$), indicating no issues in terms of collinearity. The overall regression model was statistically significant $F_{2,40} = 254.2, p = 2.20 \times 10^{-16}$, with the inclusion of ΔT improving the fit, resulting in an adjusted R^2 of 0.927. The analysis revealed that $\text{pH}_{\text{sensorcorrected}}$, equivalent to pH_{lab} , can be calculated by including the significant predictors, namely, $\text{pH}_{\text{sensor}}$ value ($p < 0.001$) and ΔT ($p = 0.0403$). $\text{pH}_{\text{sensorcorrected}}$ can thus be calculated using only $\text{pH}_{\text{sensor}}$, T_{sensor} and T_{probe} . The regression equation developed was:

$$\text{pH}_{\text{sensorcorrected}} = 1.304 + (0.8398 \times \text{pH}_{\text{sensor}}) + (-0.00457 \times \Delta T)$$

Statistically, the two-way ANCOVA showed a significant effect of seasons on pH ($F_{1,82} = 6.14$, $p = 0.0152$). However, the result was insignificant when the pH_{lab} values were compared to those of sensor ($F_{1,82} = 0.265$, $p = 0.871$), further supporting that the variability of the two data sets are statistically comparable to each other.

4. Discussion

The similar variation trend in the two pHs (pH_{lab} and pH_{sensor}) versus SST in Figure 2 indicate that these two sets of values are in accordance with each other. In addition, the Bland-Altman plot revealed that, upon correcting the sensor temperature with respect to the thermocouple, a much better alignment of the two sets of pH data were obtained. Furthermore, the strong correlation observed indicates that the pH sensor can reliably serve as an effective supplement to laboratory analyses. In our study, the observed differences are attributed to temperature variations as the salinity was almost constant at 35 ‰. Pressure variations between in situ conditions and the laboratory environment can also be reasonably disregarded. The equation from the multiple linear regression has also allowed to fine-tune the pH data from the sensor. This equation can be extrapolated to include the effects of other parameters such as when there is a more pronounced change in salinity.

Given the challenges of limited human resources and logistical difficulties in Mauritius, the pH sensor can be effectively utilized to support ocean acidification research. For instance, the pH sensor can be employed on a 24-hour or 48-hour basis, with readings at each hour or less, in order to monitor diurnal cycles of pH and SST. This will be helpful in the study of natural short-term variations in these parameters. The pH sensor can also be positioned at various strategically important sites, such as aquaculture zones or fish breeding areas. Deploying the pH sensor, the baseline data collected can be compared to global databases and incorporated into time-series analyses. The sensor can also be further coupled along with low-cost alternatives based on the Arduino platform, to allow simultaneous in situ detection of other OA-related parameters such as carbon dioxide concentration or alkalinity. This approach will help to clarify the rate of OA in the lagoons of Mauritius and identify which lagoons are more vulnerable. The Two-way ANCOVA also revealed that seasons affect pH in general.

5. Conclusion

In this study, we compared a pH sensor to the laboratory analysis of pH using UV-Vis spectrophotometry. It was observed that when not accounting for temperature discrepancies between the pH sensor's temperature measurement and using a temperature probe, the pH results had a Pearson's correlation coefficient of 0.473 ($r^2 = 0.224$, $p = 0.00135$). However, the two pH measurement methods produce similar results ($r = 0.919$, $r^2 = 0.959$, $p < 0.001$), when accounting for temperature discrepancies between the pH sensor's temperature measurement and a temperature probe. Performing the Bland-

Altman agreement analyses revealed that the two methods of pH measurements have good agreement once the temperature difference is adjusted, with a mean difference of 0.00340, with the limits of agreement ranging from -0.237 to 0.244. This study provides an insight on how the use of the pH sensor can effectively help in OA research in SIDS, as exemplified in Mauritius. We suggest deploying the pH sensor for autonomous pH monitoring in Mauritius, particularly for extended observation periods, such as for capturing diurnal or seasonal cycles. Furthermore, low-cost sensors could be tested and utilized to monitor additional physico-chemical parameters, including dissolved oxygen, nutrient levels such as nitrates and phosphates, and the partial pressure of carbon dioxide. These will be useful in understanding how the seawater of Mauritius is being affected by OA and climate change.

Supplementary Materials: Not applicable.

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