Journal of Environmental Management 345 (2023) 118899

Contents lists available at ScienceDirect

Journal of Environmental Management

journal homepage: www.elsevier.com/locate/jenvman

Research article

Influence of pH and dissolved oxygen control strategies on the performance of pilot-scale microalgae raceways using fertilizer or wastewater as the nutrient source

Rebecca Nordio [a,b,*], Emanuele Viviano [d], Ana Sánchez-Zurano [a,b], José González Hernández [c], Enrique Rodríguez-Miranda [c], José Luis Guzmán [c], Gabriel Acién [a,b]

[a] Department of Chemical Engineering, Universidad de Almería, E04120, Almería, Spain
[b] CIESOL Solar Energy Research Centre, Joint Centre University of Almería-CIEMAT, 04120, Almería, Spain
[c] Department of Informatics, Universidad de Almería, E04120, Almería, Spain
[d] Department of Science, University of Basilicata, Potenza, Italy

ARTICLE INFO

Handling Editor: Raf Dewil

Keywords:
Microalgae
Automatic control
pH
Dissolved oxygen
Productivity
Raceways
Wastewater

ABSTRACT

Dissolved oxygen concentration and pH are controllable and cost-effective variables that determine the success of microalgae-related processes. The present study compares different control strategies for pH and dissolved oxygen in pilot-scale microalgae production systems. Two 80 m^2 raceway reactors were used, one operated with freshwater plus fertilizer and the other with wastewater as the nutrient source. Both were in semi-continuous mode at a fixed dilution rate of 0.2 day^{-1}. A comparison between the classical On-Off and more advanced pH control strategies, such as PI and Event-based control, was performed, focusing on biomass productivity and the influence of all the process parameters on microalgae growth; "No control" of pH was also assayed. The results show that Event-based control was the best algorithm when using freshwater plus fertilizer. In contrast, no significant differences were observed using the different control strategies when wastewater was the nutrient source. These experiments were performed through selective control strategy, prioritizing pH over dissolved oxygen; however, it was demonstrated that they did not allow to achieve satisfactory dissolved oxygen removal results, especially for the fertilizer system. After modifying the gas diffuser configuration and improving the mass transfer, independent on-off strategies have been developed, permitting effective control of both variables and increasing productivity by up to 20% in both systems. Concluding, a detailed analysis of the energy demand for each strategy implemented in terms of gas consumption and gas flow to biomass ratio is provided.

1. Introduction

Over the last decades, the production of microalgae has gained increasing interest worldwide because of their potential application in so many different fields - from the production of complementary human foods to applied products in agriculture (biofertilizers, biostimulants and biopesticides); they can even be applied in remediation processes for wastewater or agricultural/animal rising (Yap et al., 2021). Microalgal biomass is attracting the interest of new emerging sectors since it can be included within the context of “green technologies”, defined as any technology that helps environmental conservation and minimizes the impact of human activity (Bhowmik and Dahekar, 2014). Microalgae represent a significant opportunity for developing sustainable processes and products; thus, intensive studies have been carried out to evaluate their potential, for example, for lipid extraction and biofuel/biogas production, nutrient recovery, and wastewater treatment (Odjadjare et al., 2017). The most used photobioreactor design is the open raceway because it entails low capital costs and can be easily operated (Rayen et al., 2019). However, the main disadvantages of this technology are limited control in contamination, pathogen attack, and certain process variables, such as light and temperature, that mainly depend on the environmental conditions, pH and dissolved oxygen (DO) (Narala et al., 2016).

At the laboratory scale, many studies can be found demonstrating the considerable potential of microalgal biomass applications and the advantages of producing them in freshwater or wastewater. However, pilot

* Corresponding author. Department of Chemical Engineering, Universidad de Almería, E04120, Almería, Spain.
E-mail address: rnordio@ual.es (R. Nordio).

https://doi.org/10.1016/j.jenvman.2023.118899
Received 30 May 2023; Received in revised form 14 August 2023; Accepted 27 August 2023
Available online 4 September 2023

and industrial-scale microalgal production still has cost and efficiency challenges that need to be addressed (Molina Grima et al., 2002). For this reason, over recent decades, the focus has been on reducing the gap between laboratory and large-scale production (Bernard et al., 2016), developing new reactor designs, improving them, and building new models and control strategies. Modelling in particular is a powerful tool as it allows one to study the process parameters that impact productivity, while the control strategies allow one to maintain these parameters at the optimal levels. There are many examples of microalgae growth models, depending mainly on light, temperature, pH, dissolved oxygen concentration, and substrates (Lee et al., 2015). Nevertheless, there is a noticeable lack of data on the validity of these models at a large scale, meaning that certain complex mechanisms might be neglected (i.e., adaptation) because these are not relevant over long time series (Darvehei et al., 2018). For this reason, simple large-scale models can be used to optimize the environmental and operational variables to make them more applicable to industrial-scale production.

On the other hand, control strategies need to be used to improve productivity, reduce energy demand, improve the environmental footprint, and permit an economically advantageous production process (De-Luca et al., 2019; Guzmán et al., 2021; Rodríguez-Miranda et al., 2021). Environmental and operational variables such as light, temperature, pH and dissolved oxygen determine the performance of microalgal cultures. Typically, in large-scale raceway reactors, sunlight is the energy source exploited for microalgal production while keeping the microalgal suspension at an equilibrium temperature with the surrounding environment. Despite temperature control strategies in raceway reactors being possible, none of them has been demonstrated to be economically feasible. In contrast, cost-effective pH and DO control have been widely described (Rodríguez-Miranda et al., 2022). Briefly, the dynamic of the pH depends on the equilibrium between the carbon elements and the capacity of microalgae to consume them during photosynthesis; it can be effectively controlled by on-demand CO_2 injections. Similarly, oxygen is produced by microalgae during photosynthesis and it can be delivered to the atmosphere through compressed air injections. Several studies have been carried out on the control of pH and dissolved oxygen (Fernández et al., 2010; Pawłowski et al., 2018; Pawlowski et al., 2015) that have defined different approaches for the controlled variables (static, dynamic, On-Off, PI) and for performing simulations. Other studies showed the possibility of improving biomass production in microalgae cultivations by implementing a selective control strategy of pH and DO, using an Event-based approach, achieving good control results (Pawlowski et al., 2015). However, the existing results were evaluated for short periods and no detailed comparisons were performed experimentally and considering the impact on microalgae growth. Therefore, the literature still lacks a comprehensive study of all the impacting parameters in the productivity in large-scale production over a long period, and still, there is an incomplete understanding of all the phenomena influencing biomass production. In evaluating the wastewater treatment in pilot-scale raceways proposed by Posadas et al., for example, the impact of three different pH and carbon sources (pure or from fuel gas) was explored. However, the overall effect produced by the joint effect of pH and DO on productivity was not assayed (Posadas et al., 2015a). Moreover, one can assume that the optimal approaches would be different depending on whether one is using fertilizer or wastewater as the nutrient source. In the first scenario, inorganic carbon is mainly provided through the on-demand injection of CO_2, with the photosynthesis rate determining this demand in parallel with oxygen production and then pH increase. In the second scenario, the presence of organic matter favours the presence of bacteria and influences the inorganic carbon concentration and pH, as well as the nutrient uptake efficiency, photosynthesis rate, and oxygen fluxes (Acién et al., 2016a). Consequently, a comparative study during at least weakly-period experiments would be helpful to highlight differences and to define the best adaptive approach.

In this study, two pilot-scale raceway reactors were used - one was provided with unprocessed wastewater as the culture medium and the other with freshwater plus chemical fertilizer; both were operated in semi-continuous mode at a fixed dilution rate of 0.2 day^{-1}. The study analyzed the effect of sequential pH and dissolved oxygen control when using different pH control strategies (On-Off, PI, Event-based, no control) on culture condition values for a long period (one week for each control approach), including also the influence of the inorganic carbon concentration. The temperature and nutrient availability were also recorded even though they are not directly related to the pH control strategy. The On-Off control is the most communly employed method in microalgae production due to its semplicity, yet it is often less effective regarding control performance and running costs (specifically CO_2 consumption). To adress this limitation, PI control is tested to improve the pH control accuracy. However, when moving from discontinuous to continuous controllers, such as On-Off and PI, the system performance is improved but the running costs remain high. To strike a balance between costs and performance, Event-based PI controllers are also tested as they allow finding a trade-off between costs and performance by just setting a single tuning parameter. Furthemore, it is important to note that this study not only assesses the performance of different pH control strategies but also examinates the consequences of the absence of pH regulation in large-scale production systems. Using the ABACO model (Sánchez-zurano et al., 2021), it was possible to indipendently evaluate the influence of culture conditions on the biological system's performance when employing various pH control strategies in both clean and wastewater systems. The data analysis allows us to elucidate the main phenomena taking place in both cases and the optimal conditions for each of them. Finally, we propose and evaluate a new system for simultaneous and indipendent control of pH and dissolved oxygen.

Concluding, this study centers on examining the impact of different pH/DO control strategies on the performance of pilot-scale microalgae productions. It elucidates the individual contribution of each controlling parameter and their effect on the overall process performance. This research was carried out using two large scale different culture media, such as clean water plus fertilizers and wastewater, thus providing valuable insight into the significant phenomena taking place when managing diverse biological systems. Accordingly, recommendations for pH and dissolved oxygen control strategies are presented for large-scale microalgae productions. Moreover, an optimized reactor configuration is proposed to imrpove the gases' mass transfer. Therefore, these results are considered a valuable contribution to the industrial development of microalgae-related processes.

2. Material and methods

2.1. Raceway reactors

The study was carried out using two identical 80 m^2 raceways reactors, located at the SABANA demo plant in the IFAPA research centre, La Cañada, Almería, Spain (Fig. 1A). The raceway photobioreactors comprised a double channel of 40 m in length, a paddlewheel connected to an electric motor to recirculate the culture, and a 0.59 m^3 sump where the CO_2/air was injected. The culture's depth was fixed at 15 cm, and the process was carried out in semi-continuous mode with a fixed dilution rate of 0.2 day^{-1}, meaning that every day 20% of the culture was harvested and replaced with fresh medium. Freshwater plus fertilizer was used in one system, while urban wastewater was used as the culture medium in the other raceway. The fertilizer contained 0.45 g L^{-1} of $NaNO_3$, 0.09 g L^{-1} of $MgSO_4$, 0.07 g L^{-1} of KH_2PO_4 and 0.0015 g L^{-1} of Kerantol (a mixture of micronutrients such as boron, copper, iron, manganese, molybdenum and zinc; Kenogard, Spain). The wastewater was collected from the University of Almería and was pre-treated simply by removing the main coarse body and then filtering the solid through an industrial filter (Azud Helix, 200 μm). The culture was continuously monitored using a SCADA system and a series of sensors connected to a PLC. The sensors used were for pH (1–12), dissolved oxygen (0–400%

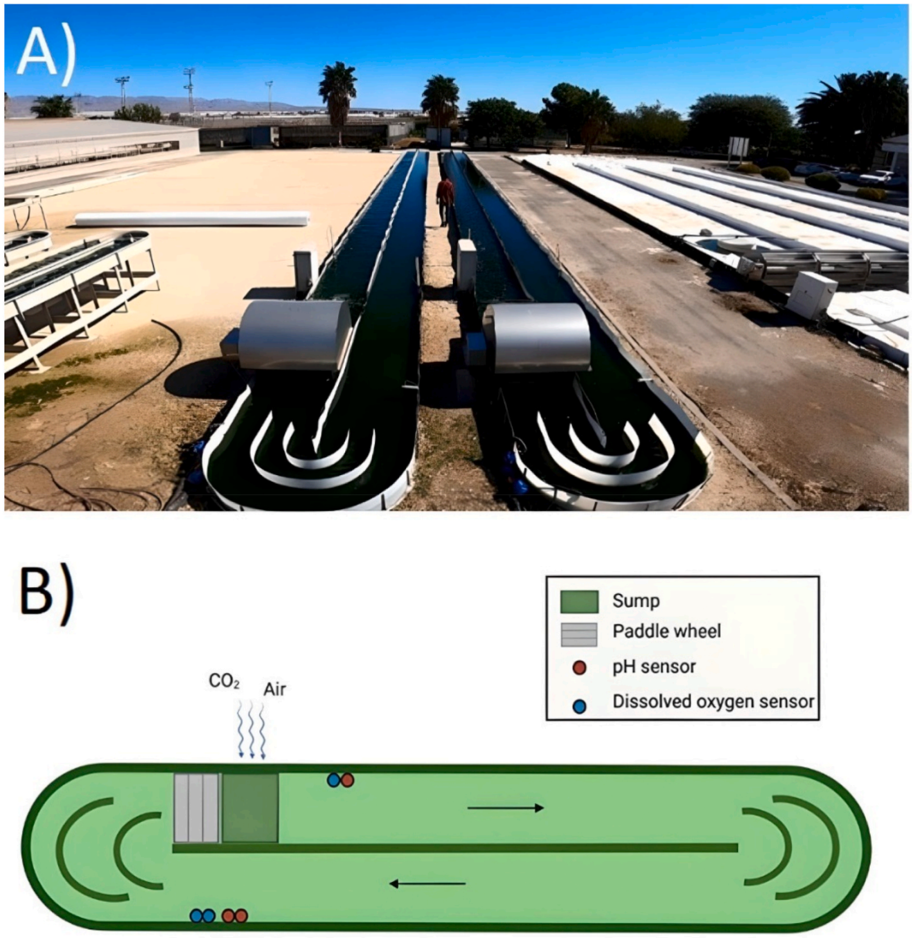

Fig. 1. Pilot-scale raceway located in the SABANA demo plant, Almeria, Spain. (A) Picture of the two pilot systems; (B) Schematic diagram of the raceway including the sump, paddlewheel, and set of sensors.

Sat.), temperature (0–80 °C), and culture depth (4–40 cm). One pH sensor and one dissolved oxygen sensor were installed after the reactor sump to provide the initial monitoring of the culture. Conversely, a second set of four sensors was installed at the end of the second channel and used to develop the culture control strategies, as this was the furthest point from the CO_2 and air injections (Fig. 1B). During the study period, all the sensors were calibrated once a week following the instructions provided by the probe manufacturers.

2.2. Strain selected and biomass concentration

The strain selected was *Scenedesmus* sp., a native microalga suitable for growing in wastewater and under the environmental conditions found in the south of Spain. It is a fast-growing strain (0.8 day^{-1}) and can survive under a wide range of temperatures (10–40 °C) and pH conditions (7.0–9.5) (Fernández Sevilla et al., 2006). The inoculum was firstly grown in a 3 m^3 tubular photobioreactor in batch mode until it reached a concentration of 0.8 g L^{-1}. It was then divided between the two raceways. One culture was slowly diluted with wastewater while the other was kept in freshwater plus fertilizer and left for a week in batch mode, allowing the culture to adapt to the new outdoor conditions. Next, the semi-continuous mode begins. The study started when steady-state conditions were reached (i.e., a stable biomass concentration was achieved).

The biomass concentration was determined offline by dry weight measurements. Samples from both reactors were collected daily at the same sampling time (9 a.m.) and in the same reactor spot (after the sump). A total of 100 mL of culture was filtered through 0.5 μm of glass fibre and dried for 24 h in an oven at 80 °C. Biomass productivity is expressed in $g{\cdot}m^{-2}{\cdot}day^{-1}$, which is an evaluation of the biomass concentration multiplied by the dilution rate and volume-to-surface ratio in the reactor. The filtrated water was stored at −20 °C and later analyzed for nutrients and carbon concentration.

2.3. Analytical measurements

The water filtrated during the dry weight procedure was analyzed for nutrients (NO_3^-, PO_4^{3-}, NH_4^+, NO_2^-, chemical oxygen demand (COD)) and carbonates (CO_3^{2-}, HCO_3^-) using spectrophotometric methodologies following the standard protocols approved by the Spanish Ministry (*MAPA. Métodos oficiales de análisis, 1986*). Nitrates were evaluated at 220 nm and 275 nm according to Standard IC 74246, while Standard IC 38364 was followed to measure the PO_4^{3-} concentration in the phospho-vanado-molybdate complex. Ammonium was measured using Nessler reactive (Standard 59755). COD and N–NO_2 were measured using Hanch-Lange kits (LCI-400 and LCK-342, respectively). Finally, the concentration of carbonates was evaluated through a volumetric method to determine the total alkalinity of the samples, using phenolphthalein and methyl orange as indicators of alkali turn and acid turn, respectively (pH Alkalinity of Water Based on ISO standard 9963–1:1994 pH-metric Titration 0.4–20 mmol/L of Total Alkalinity, 2015).

2.4. Environmental conditions

The raceways were operated outdoors, so the environmental conditions greatly influence their performance. A meteorological weather station installed next to the ponds allowed us to record environmental parameters such as solar radiation and ambient temperature. All the trials were conducted over a short period (May–June), while each control strategy was implemented for one week. The prevailing solar radiation and environmental temperatures over the experimental period are shown in Fig. 2. The average solar radiation recorded in the daylight period was 1400 μmol $m^{-2}\cdot s^{-1}$ while the average temperature was 27 °C, with maximum peaks of 30 °C and a minimum of 20 °C. Hence, the environmental conditions were constant during all the trials and did not influence any changes in either biomass productivity or culture parameters; this allowed an in-depth analysis of the influence of the different control strategies implemented.

2.5. Control strategies

The reactors were operated with different pH (On-Off, PI, Event-based and No control) and dissolved oxygen control approaches. Every strategy was employed for an entire week, with the mean values of the dependent variables calculated for the same period. Set points were established considering the optimal process parameter values for the selected strain. These parameters have been reported in previous studies and evaluated using a photo-respirometry method, as described by Barceló-Villalobos et al., 2019. Specifically, the optimal temperature was fixed at 30 °C, while the optimal pH and dissolved oxygen values were set at 8 and 12 mg L^{-1}, respectively.

- ***On-Off control***

On-Off control is the simplest and the most used control strategy in microalgae-related processes. In this case, the controller switches between two control states (On-Off) depending on the error compared to the control variable. The control law is described in (1) (Astrom and Hagglund, 1995):

$$p(t) = \begin{cases} p_{max}, if \quad e(t) > 0 \\ p_{min}, if \quad e(t) < 0 \end{cases} \tag{1}$$

where p(t) is the control signal and e(t) is the error, defined as in (2):

$$e(t) = y_{setpoint} - y_{measured} \tag{2}$$

In other words, when the pH exceeds the established setpoint value, the CO_2 injection valve opens allowing the gas to flow, until the pH drops below the reference.

- ***PI control***

Proportional-integral control (PI) is an integral action coupled with a proportional; the control law is described in (3) (Astrom and Hagglund, 1995):

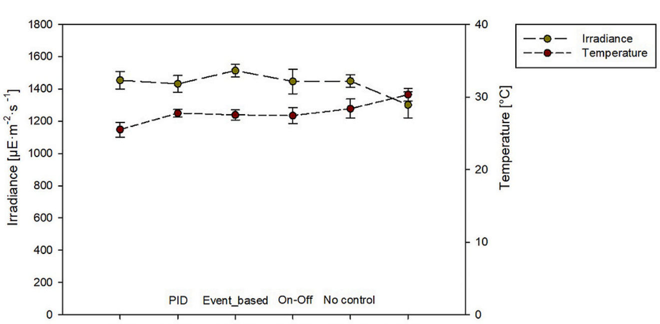

Fig. 2. Average solar radiation during the daylight period and temperature recorded during the study period.

$$C(s) = K_p \bullet \left(1 + \frac{1}{T_i \bullet s}\right) \tag{3}$$

where Kp is the proportional gain and T_i is the integral time. This strategy has the main advantage of removing off-sets and speeding up the system response compared to the open-loop, taking into account the pH evolution dynamic in the system. The pH evolution, in this case, can be modelled as a first-order plus dead time system (FOPDT), as already tested in previous studies (Rodríguez-Miranda et al., 2020). This model can be considered a reasonable approximation of the process behaviour, guaranteeing simple controller tuning at the same time. The tuning parameters have been identified from experimental data and identification tools.

$$G(s) = \frac{K}{\tau_p \bullet s + 1} \bullet e^{-\theta_p \bullet s} \tag{4}$$

where K is the process static gain, τ_p the constant time and θ_p the time delay, estimated as equal to −2.4, 1500 s and 300 s, respectively.

- ***Event-based control***

With this control strategy, the selective control of dissolved oxygen and pH was applied with an Event-based control algorithm. Specifically, a PI controller with hysteresis was applied for the pH, while the DO was controlled through an On-Off controller, assuming a constant Kl_a. In this case, the control signal was activated when the pH increased up to 8.3, at which point the CO_2 injection was stopped and air flowed into the culture. A similar approach was already tested by A. Pawlowski et al. reporting good control results (Pawlowski et al., 2015).

- ***No control***

The pH control was interrupted for the entire week by completely closing the CO_2 valve and letting the pH evolve freely.

- ***Dissolved oxygen control***

The dissolved oxygen levels in the culture can be reduced by injecting air into the reactor sump. Previous studies have reported the importance of DO concentration in the microalgal culture due to its strong influence on productivity (Barceló-Villalobos et al., 2022). In this study, a constant K_{la} was considered and the air was injected on demand according to an On-Off control strategy. As a result, the air valve was opened when the DO levels passed the set point. It is important to point out that, because of the reactor sump's initial design, all the gases (CO_2, air) flowed from a single gas diffusor, so the pH and DO strategies were implemented according to a selective strategy, such as that described above. As the pH was prioritized over the DO concentration, the air control was activated only after the pH went below the set point. In a second set of experiments, the configuration of the sump was modified to allow the simultaneous injection of CO_2 and air without an increase in carbon losses. In this case, a simultaneous strategy for pH and DO control was tested using On-Off control for both variables.

2.6. Impact of the culture conditions on the growth rate

As already mentioned, it is possible to consider a constant influence of light and temperature on the growth rate of microalgae cells because both variables remained constant during the experiments. However, productivity is influenced by other parameters such as pH and dissolved oxygen concentration, as well as the total inorganic carbon and nutrients concentration, even if some of them vary as a function of the control strategies and the type of culture medium used. A comprehensive understanding of the influence of these parameters on the growth rate of microalgae cells can be tricky and challenging. For this reason, the

ABACO model was applied to evaluate the impact of these parameters on the biological system performance as a normalized value between 0 and 1 (Sánchez-zurano et al., 2021). The ABACO model describes the behaviour of the microalgae-bacteria consortia when using wastewater as the culture medium. Specifically, the microalgal growth rate (μ, day^{-1}) is represented as a function of the average irradiance on the culture (μ(Iav)) and the normalized influence of temperature (μ(T)), along with the dissolved oxygen and pH (μ(DO)), (μ(pH)), inorganic carbon concentration (IC) (μ(IC)), and nutrient concentrations (μ($N–NH_4^+$), μ($P-PO_4^{3-}$)), as described in (5):

$$\mu = \mu(I_{av}) \bullet \overline{\mu(T)} \bullet \overline{\mu(DO)} \bullet \overline{\mu(pH)} \bullet \overline{\mu(IC)} \bullet \overline{\mu(N-NH_4^+)} \bullet \overline{\mu(P-PO_4^{3-})} \tag{5}$$

When the culture conditions (T, pH, DO, IC, $N–NH_4^+$, $P-PO_4^{3-}$) are optimal, μ functions approach a normalized value of 1; on the contrary, as they disclose from the most favourable conditions for microalgal growth, μ assume values closer to zero. The influence of the temperature and the pH can be described through a cardinal equation with inflexion as proposed by Bernard and Rémond (2012); Ippoliti et al. (2016), respectively, as in equations (6) and (7).

$$(T) = \frac{(T - T_{max})(T - T_{min})^2}{(T_{opt} - T_{min})\left[(T_{opt} - T_{min})(T - T_{opt}) - (T_{opt} - T_{max})(T_{opt} + T_{min} - 2T)\right]} \tag{6}$$

$$\mu(pH) = \frac{(pH - pH_{min})(pH - pH_{max})^2}{(pH_{opt} - pH_{min})\left[(pH_{opt} - pH_{min})(pH - pH_{opt}) - (pH_{opt} - pH_{max})(pH_{opt} + pH_{min} - 2pH)\right]} \tag{7}$$

The inhibition provoked by high oxygen concentration in the culture can be described by the equation proposed by Costache et al. (2013) and reported in equation (8)

$$\mu(DO) = 1 - \left(\frac{S_{O_2}}{S_{O_2,max}}\right)^z \tag{8}$$

Finally, the Monod function (Monod, 1949) is used for growth rate as a function of the nutrient availability (IC, $N–NH_4^+$, $P-PO_4^{3-}$), according to equations (9)–(11). Notably, it included the inhibitory effect that can be provoked by high concentrations of ammonium in the culture, as suggested in previous modelling studies (Sánchez-zurano et al., 2021).

$$\mu(IC) = \frac{S_{IC}}{S_{IC} + K_{s,IC}} \tag{9}$$

$$\mu(N - NH_4^+) = \frac{S_{N-NH_4}}{S_{N-NH_4} + K_{s,N-NH_4} + \frac{(S_{N-NH_4})^{n_{N-NH_4}}}{K_{I,N-NH_4}}} \tag{10}$$

$$\mu(P - PO_4^{3-}) = \frac{S_{P-PO_4}}{S_{P-PO_4} + K_{s,PO_4}} \tag{11}$$

Parameters such as minimum, maximum, optimum pH and temperature or the nutrient saturation half constants (Ks) have been taken from previous modelling studies such as the one reported by (Sánchez Zurano et al., 2021; Zurano et al., 2021).

Finally, the overall normalized growth rate that only includes the contribution of the culture conditions (CC), without considering the light intensity, is called μ_{cc} [-] (12), thus allowing the influence of this variable to be evaluated separately:

$$\mu_{CC} = \overline{\mu(T)} \bullet \overline{\mu(DO)} \bullet \overline{\mu(pH)} \bullet \overline{\mu(IC)} \bullet \overline{\mu(N-NH_4^+)} \bullet \overline{\mu(P-PO_4^{3-})} \tag{12}$$

in the case of the system operated with fertilizer, it is assumed that the culture has an excess of nutrients, so the values of μ($N–NH_4^+$) and μ($P-PO_4^{3-}$) are always equal to 1.

3. Results and discussion

The production of microalgae presents numerous environmental benefits, especially when integrated with wastewater treatment and nutrient recycling. Additionally, it offers alternatives for more sustainable feed or fertilizers. It is worth noting that algae-based wastewater treatment processes are an alternative to conventional methods, offering both economic and environmental benefit by merging water remediation with biomass production. Advancing our understanding of microalgae systems has the potential to amplify their efficiency and sustainability. Improved process controls and knowledge can help overcome significant drawbacks associated with microalgae production. These challenges encompass the reduction of emissions of greenhouse gases, the potential for toxins accumulation in the biomass, and relatively low energy efficiency (Usher et al., 2014). Addressing these issues necessitates further research and optimization of microalgae-based systems, focusing on comprehending how operational factors impact their performance. To aid in the optimization of raceway systems, this section discusses the experimental outcomes derived from the control approaches described earlier. The testing occurred from May to June, and each control solution was implemented for one week in two parallel reactors.

3.1. Freshwater plus fertilizer system

Large-scale microalgae production systems mainly use freshwater enriched with fertilizer as the culture medium. In this case, the pH and dissolved oxygen variations are a function of the photosynthesis rate of the microalgae cells. Hence, it is possible to neglect the influence of the existing organic matter and bacteria that, on the contrary, are relevant when using wastewater as a culture medium. Fig. 3 shows the most relevant results when evaluating different pH control strategies under such conditions. Specifically, Fig. 3A illustrates the weekly average evolution of temperature, pH, dissolved oxygen and inorganic carbon concentrations, whereas Fig. 3B represents the average impact of each parameter (T, pH, DO, IC) on the normalized growth rate. Fig. 3C shows the overall normalized growth rate (μ_{CC}) as a function of the prevailing culture conditions when using each type of pH control strategy. As already mentioned, when using freshwater plus chemical fertilizer, it is assumed there is no nutrient limitation (Nordio et al., 2023); for this reason, their contribution was not included.

The results confirm that all the control strategies tested allow the culture parameters, such as pH and dissolved oxygen, to remain constant. Furthermore, the inorganic carbon concentration stays constant, thus demonstrating that the CO_2 supply provides sufficient inorganic carbon to be consumed by the algae cells during photosynthesis. Similarly, the temperature of the cultures remained constant during the experiments. Only when testing no pH control did all these variables become uncontrolled, with the pH rising while the dissolved oxygen and inorganic carbon concentrations decreased (Fig. 3A). The normalized growth rate as a function of each of the culture parameters shows that the temperature was adequate over all the trials, whereas relevant deviations in the pH and inorganic carbon concentrations were observed when no pH control was tested, thus diminishing the culture

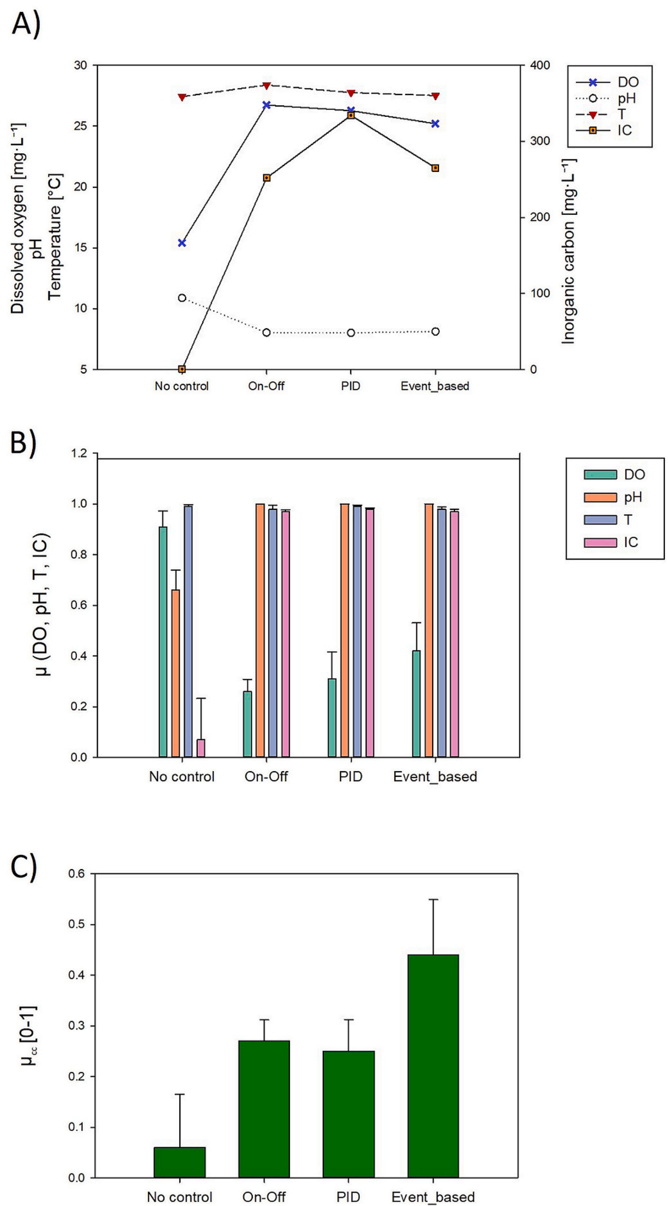

Fig. 3. Average influence of the pH control strategy on the variation in culture conditions such as pH, dissolved oxygen and inorganic carbon (A), the normalized growth rate for each culture parameter (B), and the overall normalized growth rate as a function of the culture conditions (C) for experiments performed using freshwater plus fertilizer.

performance (Fig. 3B). When pH was not controlled, the normalized growth rate for pH decreased ($\mu(pH) = 0.7$), while the normalized growth rate for dissolved oxygen was kept close to optimal values ($\mu(DO) = 0.9$). Indeed, in this specific case, the pH reached values of 11.5 during the hours of maximum irradiance, then dropped to 9 during the night due to respiration. Moreover, since the CO_2 injection was interrupted, it was possible to continuously provide air and reduce the dissolved oxygen to values below 15 mg L^{-1}. Conversely, all the pH control strategies allowed the pH to remain at the optimal value, and the normalized growth rate was maintained close to one. However, under these conditions, the dissolved oxygen concentration exceeded 26 mg L^{-1}, so the normalized growth rate for DO was reduced ($\mu(DO) =$ 0.2–0.4). Regarding the influence of the IC concentration, only in the no pH control scenario did the normalized growth rate for CO_2 decrease due to limitation by this factor ($\mu(CO_2) = 0.1$). For the other cases, this factor is close to 1, thanks to the CO_2 injected for the pH control.

Considering all these parameters, the results reveal that the least favourable conditions for biomass production were achieved when the pH was uncontrolled since the μ_{CC} approached a value close to zero (Fig. 3C). The PI and On-Off strategies had similar results to each other, with a μ_{CC} equal to 0.25 and 0.27, respectively. In the literature, these two strategies have already been tested for microalgae production in outdoor facilities. A comparison between these two control methods in raceway reactors revealed that PI is an improved control strategy compared to the On-Off as it permits enhanced pH control, thus reducing the CO_2 consumption and the valve effort (Rodríguez-Torres et al., 2021). However, in this trial, the overall normalized growth rates calculated were similar, as both strategies allowed the pH and the carbon source concentration to be maintained at the optimal value. Moreover, the low μ_{CC} value might be related to high dissolved oxygen levels in the culture during the daytime. It is known that the oxygen concentration increases when the sun rises due to microalgal photosynthesis, and follows a similar trend as the solar radiation curve, achieving inhibitory values for the photosynthesis process. To reduce this adverse effect, it is necessary to inject air into the reactor sump. Even so, with the On-Off strategy the CO_2 demand for pH control was extremely high, and air injection was allowed only when the pH value fell below the set point values. Similarly, the PI controller sent a signal to the CO_2 valve proportional to the pH dynamics so that it could be precisely controlled without significant variations. In this way, the CO_2 injection was kept continous throughout the day with no air injection allowed at any time. Consequently, in both cases, the dissolved oxygen reached values above 27 mg L^{-1}, reducing the normalized growth rate. The Event-based strategy was the most suitable control approach as it allowed an $\mu_{CC} = 0.44$ to be reached (Fig. 3C). Indeed, this methodology enables improved DO removal because of the selective injection of CO_2 and air while maintaining the pH value in the optimal range throughout the day. These results are in line with those previously reported, which describe the great potentiality of this control approach when applied to microalgae production, leading to reduced energy requirements and improved biomass productivity (Rodríguez-Miranda et al., 2019). However, it is important to point out that, although this strategy produced the best results, the high DO levels still limit microalgal growth.

Concerning the influence of the pH control strategy on biomass productivity, as expected, the results show a similar trend to that observed for the overall normalized growth rate (Fig. 4). As was foreseen when evaluating the individual process parameters and the overall normalized growth model for each control strategy, the minimum biomass productivity (up to 15 g m^{-2}·day^{-1}) was observed when no control of pH was performed, mainly due to inorganic carbon limitation under these conditions. When using either the PI or On-Off pH control strategies, similar biomass productivity values were obtained (up to 20 g m^{-2}·day^{-1}) despite inadequate dissolved oxygen concentrations in

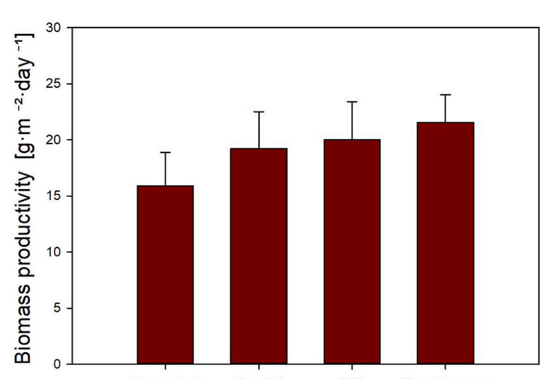

Fig. 4. Influence of the control strategy on the biomass productivity of microalgae cultures performed in raceway reactors using freshwater plus fertilizer.

both cases. Maximal performance was observed using the Event-based control (up to 22 g m^{-2}·day^{-1}), confirming that this strategy is the most recommendable. In relative terms, the use of the PI or On-Off control strategies increases the biomass productivity by up to 25% compared to no control of pH, whereas the Event-based strategy increases the biomass productivity by up to 36% compared to the same base case where there is no control of pH. In conclusion, the results reveal that the pH and the dissolved oxygen are equally relevant in terms of biomass productivity and must both be controlled to maintain a stable and productive culture.

3.2. *Wastewater system*

When using wastewater as the nutrient source in microalgal cultures, one must consider certain additional phenomena that occur in the system compared to when freshwater is used. First, in wastewater, the nutrient concentration can vary significantly (Mohsenpour et al., 2021). Analysis of the water inlet recorded ammonium and phosphorus concentrations ranging from 100 to 160 mg L^{-1} and from 30 to 50 mg L^{-1}, respectively. These concentrations did not limit the microalgal growth; consequently, the normalized growth rate for phosphorus $\mu(PO_4^{3-})$ was equal to 1 during the cultivation period, while the $\mu(NH_4^+)$ varied from 0.8 to 0.9. Moreover, organic carbon enters the system with the wastewater so bacteria live together with microalgae, thus influencing the gas fluxes in the CO_2 and O_2 consumption/production rates.

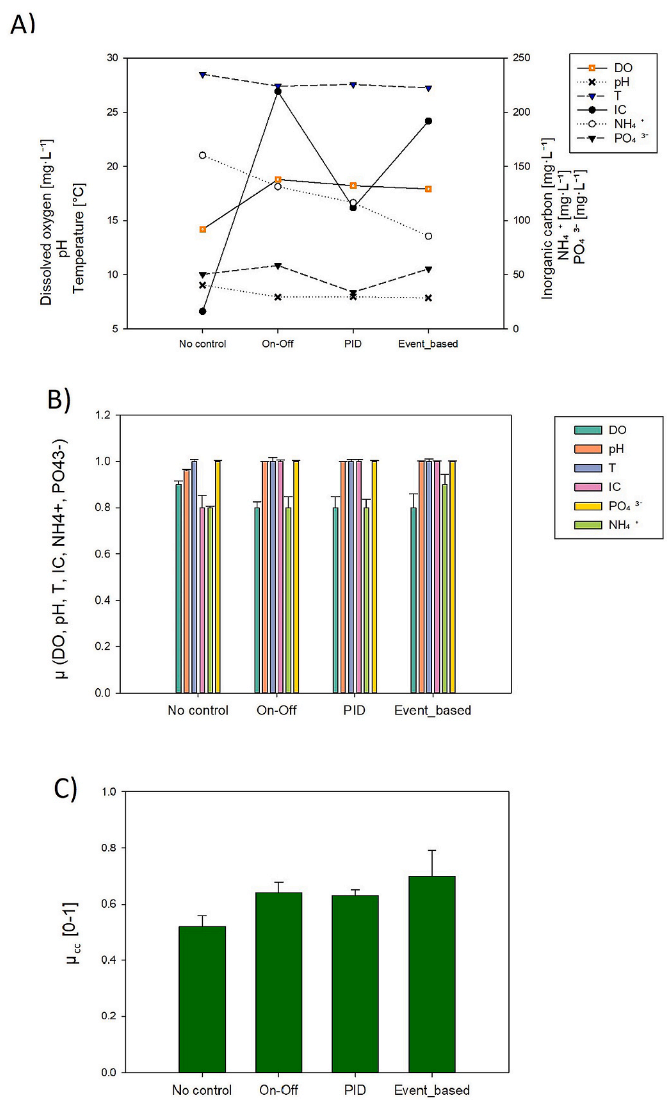

Fig. 5. Average influence of the pH control strategy on the variation in culture conditions such as pH, dissolved oxygen and inorganic carbon (A), the normalized growth rate for each culture parameter (B), and the overall normalized growth rate as a function of the culture conditions (C) for experiments performed using wastewater.

The most relevant results when evaluating the different control approaches in wastewater-related microalgae cultures are shown in Fig. 5. Specifically, Fig. 5A presents the average variation in culture conditions (T, pH, DO, IC, N–NH_4^+, P-PO_4^{3-}) throughout the experiments, while Fig. 5B presents the impact of each parameter (T, pH, DO, IC, N–NH_4^+, P-PO_4^{3-}) on the normalized growth rate for each variable. Finally, Fig. 5C shows the overall normalized growth rate (μ_{CC}) as a function of the prevailing culture conditions when using each type of pH control. Whichever control strategy was implemented, the pH and the dissolved oxygen concentration were kept constant: the pH was always at the optimal value of 8.0 while the DO remained at a concentration equal to 18 mg L^{-1}. Moreover, an increase in the inorganic carbon concentration was recorded when implementing the On-Off and the Event-based strategies (200–220 mg L^{-1}), while it reduced to 100 mg L^{-1} when using the PI control. As already mentioned, this latter type of controller allowed the pH to stay at the optimal value and reduced the CO_2 injection effort. In contrast, when no control of pH was implemented, the pH increased to values up to 10 while the IC and DO concentrations decreased (equal to 16 mg L^{-1} and 14 mg L^{-1}, respectively) (Fig. 5A); in a similar way as the previous results obtained using freshwater plus fertilizer. Regarding the temperature and the nutrient availability (N–NH_4^+, P-PO_4^{3-}), the data confirm that all remained constant, regardless of the pH control strategy tested.

The variation in the normalized growth rate as a function of the process parameters shows that, as in the previous case, the culture temperature was always optimal; moreover, the nutrient availability was also higher than the limiting conditions. One can observe that, despite the changes already described in Fig. 5A, the pH was always maintained at the optimal value, regardless of which pH control strategy was implemented, with only a slight decrease when the system had no pH control ($\mu(pH) = 0.95$) (Fig. 5B). This fact demonstrates the auto-regulating capacity of the microalgae-bacteria culture when using wastewater as a culture medium, with pH mantained below 10 including when no CO_2 is provided. As already mentioned, when using wastewater, consortia of microalgae and bacteria always exists, mainly composed by heterotrophic and nitrifying (Sánchez-Zurano et al., 2020). Bacteria use the dissolved oxygen produced by microalgae during photosynthesis to oxidize organic matter, thus releasing CO_2, which is then consumed by the microalgae to perform photosynthesis. However, the dynamics are much more complex, with multiple metabolic processes occurring simultaneously. These processes include nitrification, phototrophic growth, and nitrate assimilation, among others. The variation of pH is a function of these processes: it increases due to photosynthesis and concomitant uptake of CO_2 and NO_3^- by the microalgae cells while it decreases due to respiration and the release of CO_2 by heterotrophic bacteria (Acién et al., 2016b). The culture pH is determined by the relative significance of these two phenomena, and explains why no significant differences were observed when implemented different control strategies into system where wastewater was the culture medium. Moreover, this also explain why no major differences were observed when the pH was not controlled. However, it is possible to note that when no additional CO_2 was supplied to the system, the $\mu(CO_2)$ dropped to 0.8, meaning that there was not enough carbon supplied by the bacteria to avoid carbon limitation for the microalgae growth. Furthermore, with no pH control, the dissolved oxygen level dropped below 14 mg L^{-1}, so the μ(OD) approached the optimal value (0.9).

Regarding the overall effect of the control strategies on the normalized growth rate, the results show that the μ_{CC} was optimized regardless of the control strategy implemented. A slight improvement was observed when implementing the Event-based approach (0.70), similar to that demonstrated when using freshwater plus fertilizer

(Fig. 5C). Despite the pH remaining below hazardous levels when not controlled, the reduced value of $\mu_{CC} = 0.5$ was due to the reduced amount of IC that was limiting microalgal growth under such conditions. These results confirm the need for an external CO_2 supply to avoid carbon limitation and increase productivity even when using wastewater as the nutrient source. Similar results have already been reported at the laboratory scale: some authors have demonstrated that the external CO_2 supply in microalgae cultures grown in wastewater increases microalgal productivity (Sutherland et al., 2015). Conversely, other authors have indicated that no CO_2 is required for pH control when producing microalgae in wastewater although, in this case, the experiments were performed under unfavourable conditions where the CO_2 demanded by the microalgae cells was low. Nevertheless, these same authors demonstrated that, in this type of application, using CO_2 supplied from industrial flue gases was a more eco-friendly option (Posadas et al., 2015a).

About the influence of the pH control strategy on the variation in biomass productivity when using wastewater (Fig. 6), this was equal to 23 g m^{-2}·day^{-1} when the pH was not controlled, while it it slightly increased to 25 g m^{-2}·day^{-1} when using the On-Off or PI control strategies, and up to 26 g m^{-2}·day^{-1} when using the Event-based control strategy. The self-regulating capacity of cultures grown in wastewater demonstrates that microalgae biomass production is favoured by minimizing any deviation in culture parameters, thus diminishing the relevance of using improved control strategies. This has already been reported in both laboratory and pilot-scale reactors (Arbib et al., 2013; Posadas et al., 2015b) and this strategy option is used in large-scale facilities (Mennaa et al., 2019). However, leaving the production systems with no control strategy is not recommended because disturbances or an inadequate composition of inlet flow may provoke system collapse and microalgal cellular death.

3.3. Simultaneous control of pH and dissolved oxygen

To solve the issue of sequential CO_2/air injection to control the pH/DO, a new design for the raceway gas injection system was developed. Typically, raceways consist of a paddlewheel for culture recirculation and a sump for gas injection. These two zones were shown to be where the mass transfer mainly occurs (J. L. Mendoza et al., 2013b). Moreover, it was demonstrated that using adequate gas diffusers improves gas-liquid transfer (J. L. Mendoza et al., 2013a). Despite having already fully demonstrated the importance of removing the DO generated (Weissman et al., 1988), in classical raceway configurations, the sumps are mainly used to supply inorganic carbon in the form of CO_2 and to control the pH, while it is assumed that the DO is removed in the reactor channels. Nevertheless, the importance of dissolved oxygen accumulation in raceway reactors has been extensively shown (Barceló-Villalobos et al., 2018; J.L. Mendoza et al., 2013). By evaluating the influence of

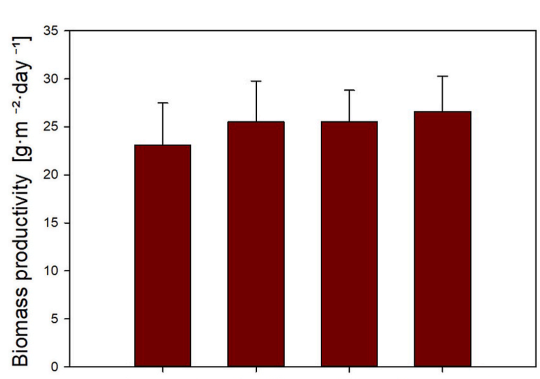

Fig. 6. Average influence of the control strategy on the biomass productivity of microalgae cultures grown in raceway reactors using wastewater.

the process parameters on microalgal growth, we have demonstrated that the pH and the DO play an equally important role in process performance, with DO accumulation markedly reducing the normalized growth rate of the microalgae cells. In addition, despite the good results achieved, the selective strategy using Event-based control was still unable to tackle the challenge related to high dissolved oxygen levels, especially when using freshwater plus fertilizer. For this reason, we propose a modification to the gas injection system, specifically that the gas injection (CO_2 and air) should be separated into two different gas diffusers; a smaller one comprising a small porous tube for the CO_2 and a larger one consisting of three disk gas diffusers for the air.

The new sump configuration permits the simultaneous gas injection of CO_2 and air, thus making it possible to have independent control strategies for the pH and dissolved oxygen. In particular, a new control approach was tested in which the pH and DO were independently controlled using an On-Off strategy. For these experiments, only the On-Off controllers were tested, since the objective was to evaluate the performance of the new diffuser configuration. The results obtained with this new solution are shown in Fig. 7. Using simultaneous gas injections, adequate pH control was ensured. At the same time, it was possible to reduce DO accumulation from 27 mg L^{-1} to 20 mg L^{-1} when using freshwater plus fertilizer, and from 19 mg L^{-1} to 14 mg L^{-1} when using wastewater as the culture medium. Furthermore, an adequate inorganic carbon concentration was ensured in both cases, although there was a slight decrease in the sequential control of pH/DO (Fig. 7A and B). From the separate analysis of the normalized microalgal growth, one can see that, when separately controlling the pH and the DO, it was possible to maintain the main parameters (pH, IC) close to the optimal values. Moreover, the increase in μ(DO), from 0.8 to 0.9 and from 0.3 to 0.7, when using wastewater and freshwater plus fertilizer, respectively (Fig. 7C and D), resulted in a substantial improvement in process performance. Concerning the overall normalized growth rate, μ_{CC}, the values increased from 0.27 to 0.66 when using freshwater plus fertilizer, and from 0.64 to 0.78 when using wastewater (Fig. 7E).

The influence of sequential/simultaneous pH/DO control on biomass productivity is presented in Fig. 8. The results show that for the freshwater plus fertilizer culture medium, the biomass productivity increased from 19 to 23 g m^{-2}·day^{-1} (a 21% increase) when using separated or simultaneous pH control, respectively. Similarly, for the wastewater culture medium, the biomass productivity increased from 25 to 30 g m^{-2}·day^{-1} (a 20% increase) when using separated or simultaneous pH control, respectively. Therefore, the productivity was increased by 47% for the fertilizer and 31% for the wastewater compared to the case of no pH control. These results confirm that by modifying the reactor design and control approach, biomass productivity is significantly increased. As previously indicated, the increase in productivity is related to the better performance of the new control system allowing the simultaneous improvement of pH and dissolved oxygen (DO) control, which was not possible if using the previous CO_2/air injection system. In future works, it will be necessary to perform more studies and apply different control strategies for pH and DO, such as PI and Event-based, to evaluate their benefit.

3.4. Energy consumption

The different pH/DO control strategies have demonstrated the possibility of adequately modifying the culture conditions (pH, DO, IC), providing important improvements in the growth rate and biomass productivity. However, it is also necessary to analyze the control effort invested to do so, that is, the required resources and costs in each case. For that, we evaluated the CO_2 and air consumption when using the different pH/DO control strategies (Fig. 9), by measuring the consumption of CO_2 (Fig. 9A) and air (Fig. 9B) for each strategy implemented. As already discussed, when using wastewater, the CO_2 demand was always lower than when using freshwater plus fertilizer, as the bacteria contribute to the production of inorganic carbon. Furthermore,

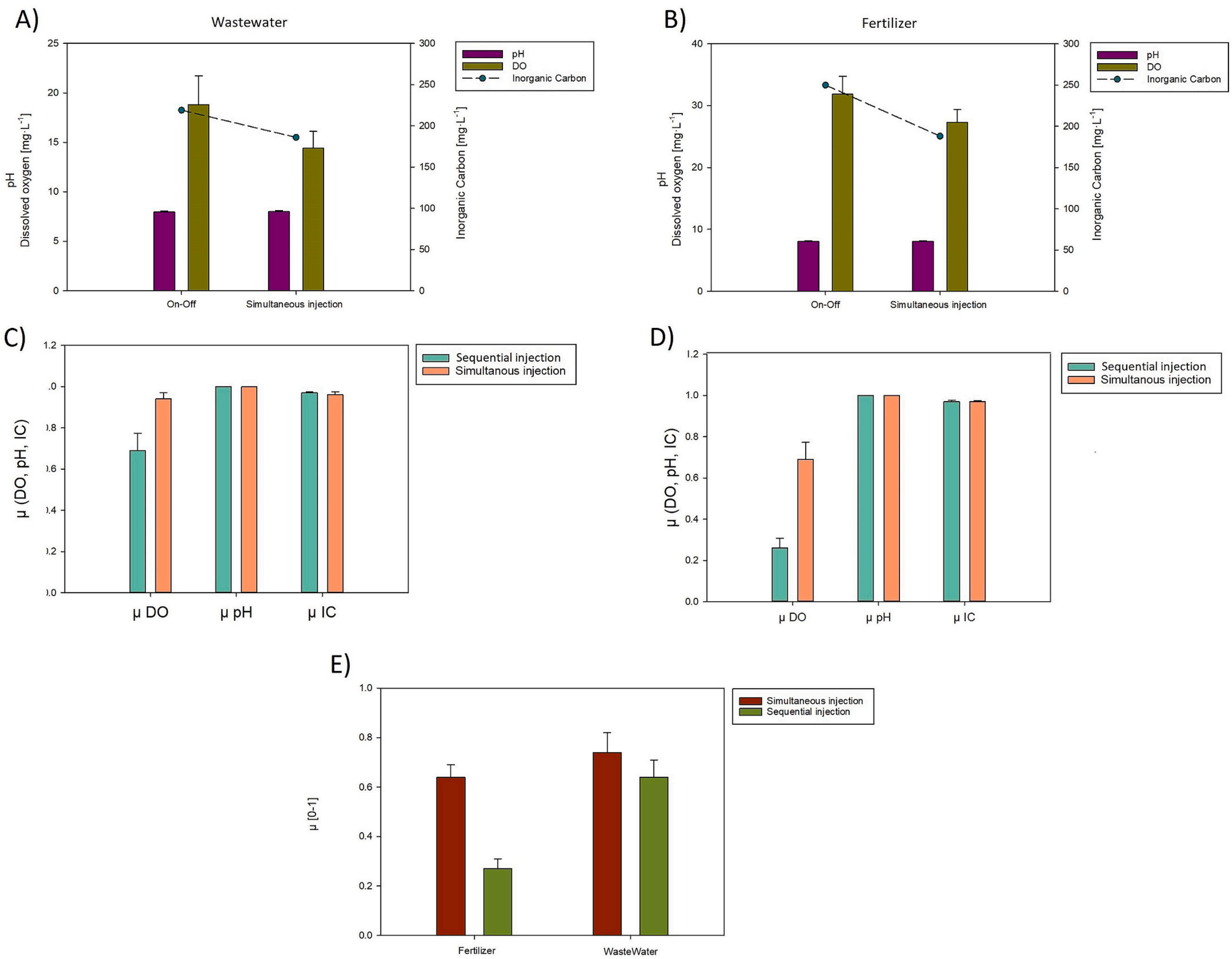

Fig. 7. Influence of sequential and simultaneous pH and DO control on the culture parameter values (pH, DO and IC) for experiments performed using both wastewater (A) and fertiliser (B), on the normalized growth rate for each culture parameter (pH, DO and IC) for experiments performed using both freshwater wastewater (C) and fertiliser (D), and the overall normalized growth rate as a function of the culture conditions for experiments performed using both freshwater plus fertilizer and wastewater (E).

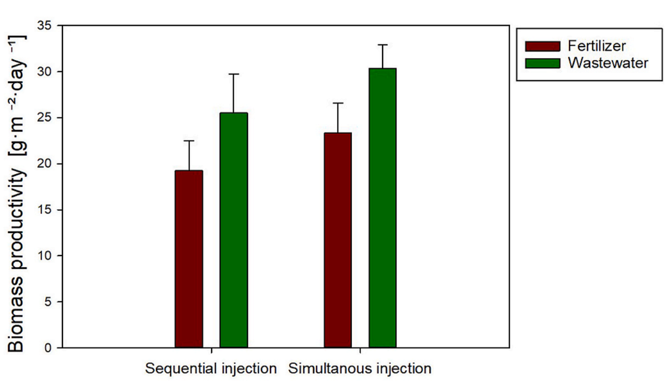

Fig. 8. Influence of sequential/simultaneous pH/DO control strategy on the biomass productivity of microalgae cultures performed when using both freshwater plus fertilizer and wastewater.

among the separated pH/DO control strategies, the PI strategy resulted in lower CO_2 consumption (up to 23% less than using the On-Off strategy) when using the freshwater plus fertilizer medium, from 34 to 26 L $m^{-2}\cdot day^{-1}$; whereas for the wastewater medium, the Event-based strategy presented the best performance, with lower CO_2 consumption than with the On-Off strategy, from 20 to 16 L $m^{-2}\cdot day^{-1}$ (a 20% reduction). Regarding the simultaneous control of pH/DO, there was an increase in CO_2 demand regardless of the culture medium used. This increase was minimal when using freshwater plus fertilizer, from 34 to 35 L $m^{-2}\cdot day^{-1}$ (a 2% increase), but became more relevant when using wastewater, increasing from 20 to 28 L $m^{-2}\cdot day^{-1}$ (a 40% increase). This makes sense from the efficiency analysis performed, as discussed below.

Regarding air demand, the use of separated pH/DO control strategies limits the amount of air being provided to the reactors, thus negatively affecting biomass productivity. The airflow supplied was higher when using the On-Off strategies regardless of the culture medium, with values of 500 and 600 L $m^{-2}\cdot day^{-1}$ for freshwater plus fertilizer and wastewater, respectively. Only when using simultaneous pH/DO control was the air demand not restricted by the CO_2 supply; in this case, the air supply greatly increased up to 1700 and 1100 L $m^{-2}\cdot day^{-1}$ for freshwater plus fertilizer and wastewater, respectively. The air demand is greater when using freshwater plus fertilizer because, under these conditions, there are no relevant bacteria populations consuming part of the

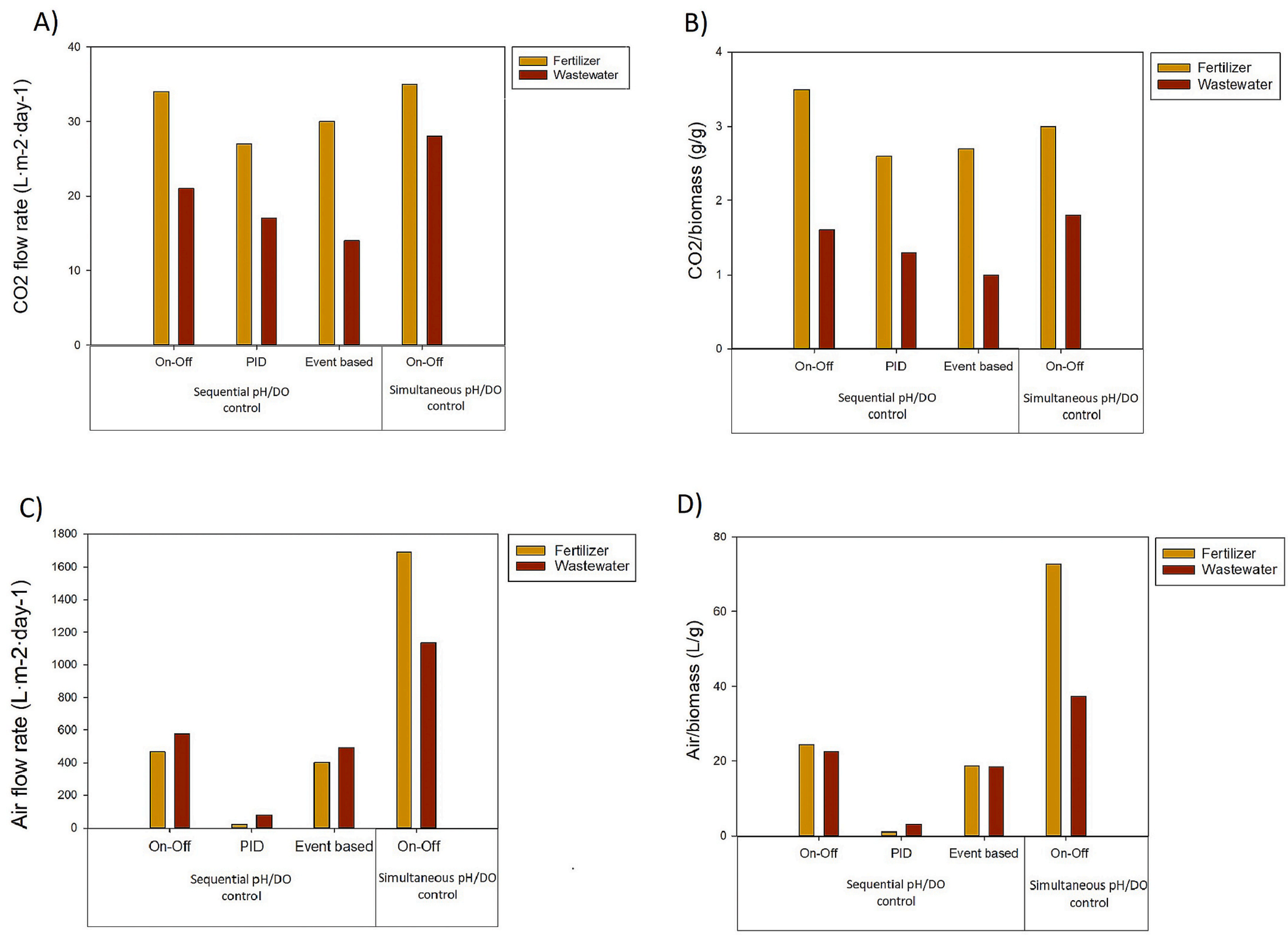

Fig. 9. Influence of pH/DO control strategy on the CO_2 (A) and air (B) consumption, and the specific CO_2 (C) and air (D) consumption of microalgae cultures grown using both freshwater plus fertilizer and using wastewater.

oxygen produced by the photosynthesizing microalgae cells.

In terms of efficiency, the gas flow to biomass ratio must be analyzed. In this case, the CO_2 to biomass ratio was lower when using wastewater than for the freshwater plus fertilizer medium regardless of the control strategy used. This confirms that the carbon from the organic matter is utilized when using wastewater as the nutrient source (Fig. 9C). When using separated pH/DO control strategies, the minimum values for the CO_2 flow to biomass ratio were obtained using the Event-based control strategy, with values up to 1.34 and 0.6 L g^{-1} (2.6 and 1.2 $g_{CO2} \cdot g_{biomass}^{-1}$) for the freshwater plus fertilizer and wastewater media, respectively. Using the simultaneous pH/DO control strategy, the CO_2 flow to biomass ratio increased to 1.5 and 0.92 L g^{-1} (2.9 and 1.8 $g_{CO2} \cdot g_{biomass}^{-1}$) for freshwater plus fertilizer and wastewater, respectively. However, these values are much lower than those obtained when using the On-Off control strategy with separated pH/DO control, achieving values up to 1.80 and 0.80 L g^{-1} (3.5 and 1.5 $g_{CO2} \cdot g_{biomass}^{-1}$) for the freshwater plus fertilizer and wastewater media, respectively. Although more research on this topic is required, the simultaneous pH/DO control system that was developed reduced the CO_2 flow to biomass ratio by up to 20% when using freshwater plus fertilizer. However, it is known that the dissolution of CO_2 from the gas to the liquid phase is limited by a low mass transfer coefficient, limiting its availability for microalgae (Wu et al., 2023), so further studies are necessary to improve the mass trasfer capacity. In the case of the airflow to biomass ratio, different behaviour was observed (Fig. 9D). In general, the airflow to biomass ratio was higher when using freshwater plus fertilizer than when using wastewater, thus confirming the lower oxygen desorption requirement for wastewater due to the bacteria consuming oxygen. Concerning the control strategies, only the simultaneous control of pH/DO allows adequate control of dissolved oxygen given the high airflow to biomass ratio required, up to 73 and 36 L g^{-1} when using freshwater plus fertilizer and wastewater, respectively. These values are much higher than those measured when using separated pH/DO control, regardless of the final control strategy selected (On-Off, PI, Event-based). In future works, it will be necessary to further optimize the CO_2/air consumption as well as the mass transfer in the sump and the control strategy.

3.5. Main insights

This study demonstrates the significance of pH and dissolved control systems in optimizing microalgae production processes. Although the experiments were carried out using *Scenedesmus* sp. as the predominant microalgae strain within the cultures, even when using wastewater as a nutrient source, the findings hold relevance for the production of any other strain. Notably, the observed phenomena and their relative significance are more influenced by bacterial presence due to wastewater usage than by the specific microalgae strain being cultivated. This comparative study between the different control strategies has provided certain insights into the best control approach to apply.

- pH control is fundamental in guaranteeing a stable culture. However, at the same time, it is necessary to control the dissolved oxygen concentration to increase biomass productivity.
- Adequate CO_2 injection is crucial to ensure the correct carbon supply to the microalgae cultures. When using wastewater, less CO_2 is required as the bacteria present also contribute to its biological release; furthermore, it can "auto-regulate," maintaining pH levels that are always below 10. However, further studies will be necessary to evaluate the impact of free pH variation on the balance between microalgae and bacteria, defining which population is favoured.
- Event-based control approaches proved the most suitable (especially when using freshwater plus fertilizer) to ensure adequate pH control, lower the dissolved oxygen concentration, and enhance productivity when separated pH/DO control strategies are employed.
- Using simultaneous pH/DO control strategies significantly improved both the culture control parameters and the biomass productivity. Moreover, the CO_2 demand was reduced compared to the classical sequential On-Off strategies. Therefore, it was demonstrated that the simultaneous pH/DO control approach is recommendable for pilot and industrial-scale microalgae cultivations.
- In future work, it is recommended to evaluate the new sump configuration by using more advanced strategies such PI and Event-Based. Also, the oxygen/CO_2 mass transfer should be optimized. Furthermore, the system performance have to be evaluated during different seasons of the year. This would allow for performing realistic techno-economic analysis enabling the assessment of productivity gains relative to control effort expenses.

4. Conclusion

It has been demonstrated that adequate pH/DO control systems are central to determining the performance of microalgae cultures grown in raceway reactors, resource use, and overall production process efficiency. Different sequential pH/DO control strategies were evaluated; the most recommended is the Event-based. However, this strategy does not adequately control the dissolved oxygen concentration in the culture. Therefore, in this study, it has been demonstrated the effectiveness of a novel control approach for raceway reactors that allow simultaneously controlling the pH and DO. These new strategies are suitable for the optimized operation of these systems permitting to reach increased productivity and maximizing the microalgae cultures' growth rate and they can be considered a valid configuration for industrial-scale productions. However, more research is required to test other controllers apart from the classical On-Off and to optimize both the CO_2 and the airflow-to-biomass ratio.

CRediT authorship contribution statement

Rebecca Nordio: Investigation, Formal analysis, Validation, Visualization, Writing – original draft. **Emanuele Viviano:** Investigation, Writing – original draft. **Ana Sánchez-Zurano:** Visualization, Writing – original draft. **José González Hernández:** Investigation, Software, Data curation. **Enrique Rodríguez-Miranda:** Formal analysis, Software, Data curation. **José Luis Guzmán:** Conceptualization, Supervision, Writing – review & editing, Funding acquisition. **Gabriel Acién:** Conceptualization, Supervision, Writing – review & editing, Funding acquisition.

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.

Acknowledgements

This research was funded by the H2020 Research and Innovation Programme under the Marie Sklodowska-Curie grant agreement (project: Digitalgaesation, 955520) and by the H2020 Research and Innovation Framework Programme (projects: PRODIGIO, 101007006; REALM, 101060991). Also, this work was supported by the Spanish Ministry of Science and Innovation (project: HYCO2BIO, PID2020-112709RB-C21). All the authors would like to thank the Institute for Agricultural and Fisheries Research and Training (IFAPA).

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