Algal Research 90 (2025) 104235

Contents lists available at ScienceDirect

Algal Research

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

A new method for the online determination of mass transfer and oxygen production rates in microalgae raceway reactors☆

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ARTICLE INFO

Keywords:
Photosynthesis
Photobioreactor
Process optimization
biomass
Sustainable processes

ABSTRACT

In this study, a novel methodology for the real-time determination of mass transfer coefficient and oxygen production rates in large-scale raceway reactors for microalgae cultivation was developed. An innovative approach based on dissolved oxygen (DO) measurements and controlled air pulse injections provided accurate and real-time insights into the photosynthetic performance of microalgae cultures and the efficiency of gas exchange mechanisms within the reactor. Experiments were performed in 80 m^2 raceway reactors under continuous operation. A set of controlled aeration pulses at different flow rates (125, 250, 375, and 500 L/min) is applied throughout the day, enabling the evaluation of oxygen production under varying light intensities. The method is designed to operate without disrupting culture stability and ensures continuous monitoring of mass transfer dynamics in raceway reactors. The optimal conditions for the utilization of the proposed methodology are defined, and the robustness and accuracy of results are validated. Results demonstrate that oxygen production rates directly correlate with solar radiation, following a linear trend that serves as a proxy for biomass productivity. The study confirms that the sump section plays a crucial role in oxygen desorption, with mass transfer coefficients reaching up to 200 h^{-1} at maximal superficial gas velocities below 0.02 m/s, then operating in a homogeneous aeration regime. This research provides a powerful tool for optimizing large-scale microalgae production systems, enabling real-time performance monitoring and dynamic process control to enhance biomass productivity.

1. Introduction

Industrial production of microalgae is emerging worldwide. Applications of microalgae in pharmacy, cosmetics, nutraceuticals, food/feed and agriculture, as well as other wastewater treatment, CO_2 capture and the production of biofuels are under development [1]. Some of the major advantages of microalgae compared to traditional crops are their rapid growth rates and the possibility of being produced in non-arable land and using other than freshwater. Although heterotrophic and mixotrophic production is possible, the conventional production mode involves the use of solar energy as a driver of the process. In this sense, microalgae are produced in photobioreactors, the adequate design and operation of the photobioreactors being essential to optimize productivity [2].

Among different types of photobioreactors proposed the use of raceway reactors is the most extended due to its lower cost and easy scaleup [3]. Mass transfer of oxygen can limit the upscaling of raceway reactors. During the photosynthesis process, microalgae consume CO_2 and release O_2, therefore the adequate supply of CO_2 and removal of O_2 determine the overall performance of the photosynthetic process. The challenge is to avoid carbon limitation and, at the same time, avoid inhibition by excess oxygen accumulation [4,5]. These drawbacks are directly related to the mass transfer capacity in the sump of the reactor. In raceway reactors, it was demonstrated that although oxygen is desorbed to the atmosphere in the channel and the paddlewheel, the sump is the reactor section that mostly contributes to oxygen desorption [6,7]. It has been also confirmed that mass transfer into the sump is a function of the design of the sump and the air flow rate provided [8]. To evaluate the mass transfer capacity in raceway reactors experimental measurements were performed without microalgae culture, using conventional

☆ This article is part of a Special issue entitled: 'DigitAlgaesation' published in Algal Research.

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https://doi.org/10.1016/j.algal.2025.104235
Received 16 April 2025; Received in revised form 20 July 2025; Accepted 20 July 2025
Available online 21 July 2025

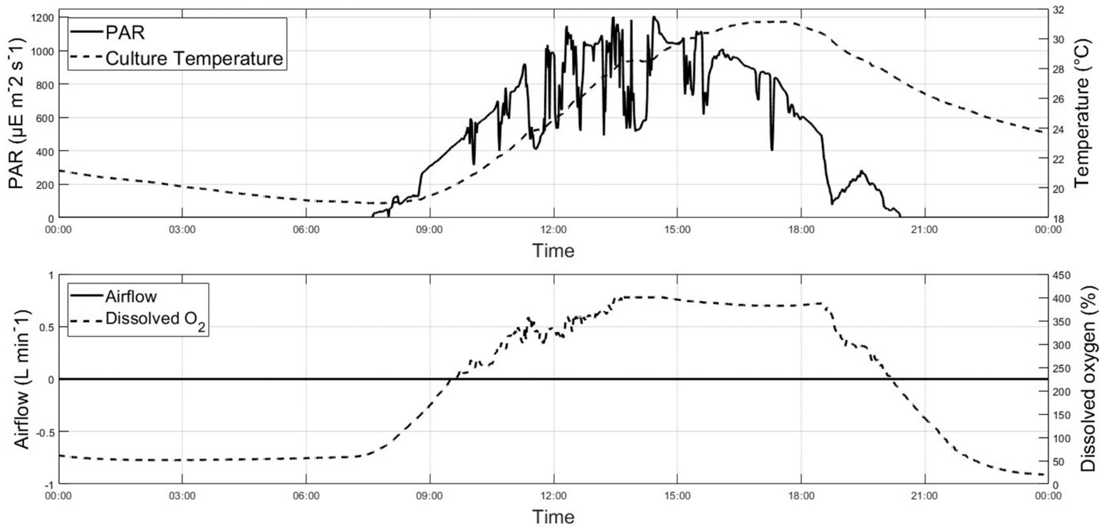

Fig. 1. Daily variation of solar radiation, temperature and dissolved oxygen concentration in the 80 m^2 raceway reactor when no air is provided to the sump.

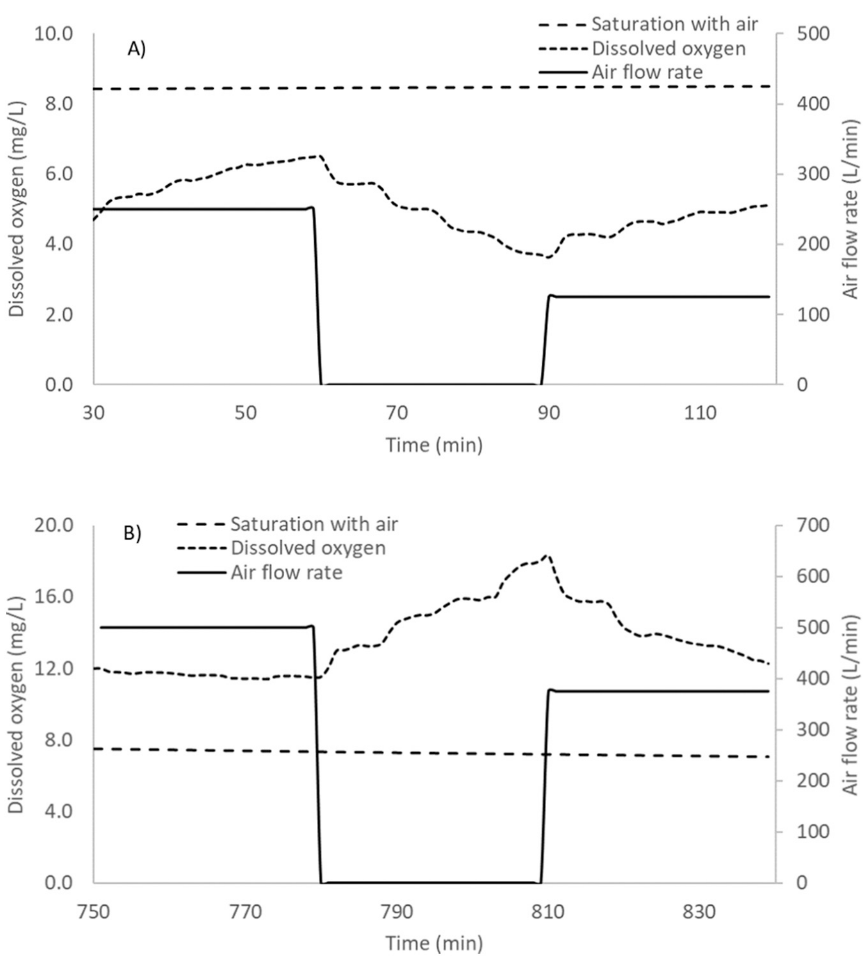

Fig. 2. Variation of dissolved oxygen concentration and saturation level at equilibrium with atmospheric air ($[O_2^*]$) values as a function of time at different aeration rates during the night (A) and daylight period (B).

dynamic or steady-state methods that require long times and the consumption of N_2 or chemicals (sulphite) [6,7]. Alternatively, some approaches have been proposed using numerical methods and Computational Fluid-dynamic tools [9,10]. The first methodology is not practicable at a large scale, whereas the second approach requires validation steps that are usually not performed. Therefore, new methods are needed to evaluate and calibrate the mass transfer capacity in existing large-scale reactors.

Otherwise, the performance of outdoor microalgae cultures is not constant, mainly because the cultures are exposed to changing

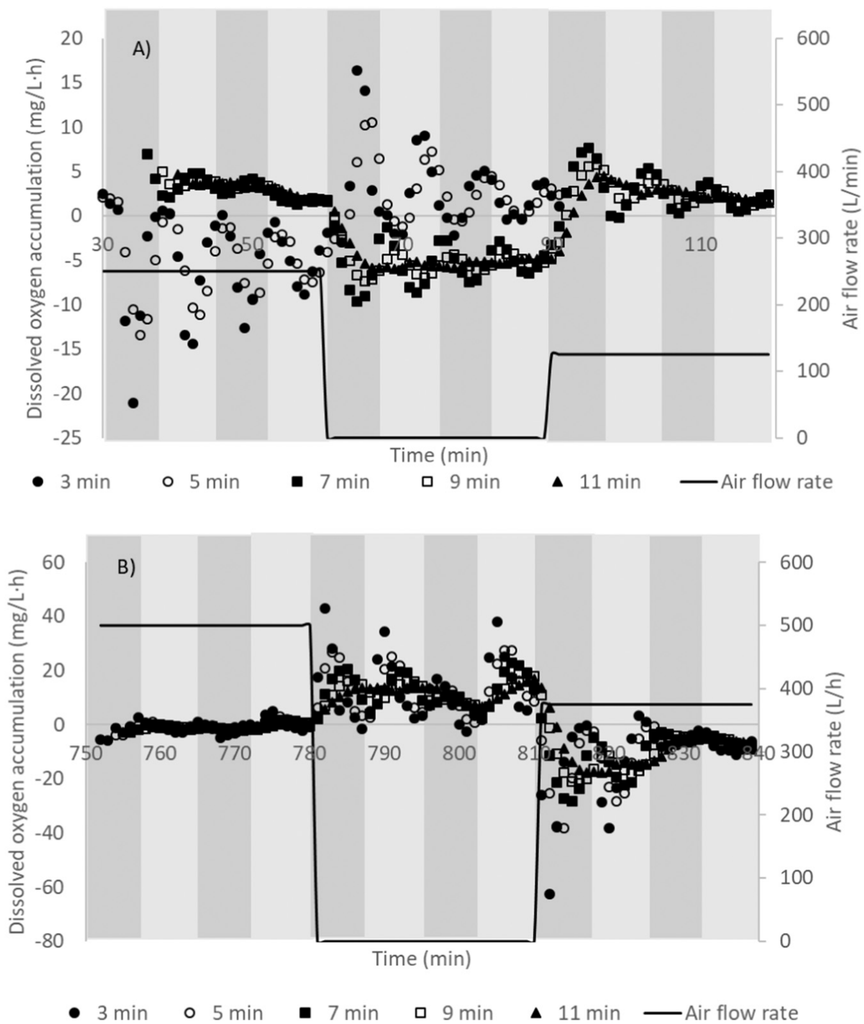

Fig. 3. Variation of dissolved oxygen accumulation as a function of time at different aeration rates during the night (A) and daylight period (B), calculated at different frequencies along each cycle.

environmental conditions during the day and along the different seasons, but also because other perturbations such as inadequate culture conditions or the presence of pathogens can also reduce it [11,12]. Daily variation of solar radiation is the major affecting the performance of microalgae cells, in addition, solar radiation and ambient temperature determine the temperature of the culture and its performance [13,14]. Concerning culture conditions, if appropriate control systems are implemented both the pH and dissolved oxygen can be maintained at their optimal setpoint otherwise large devitations take place that also reduce the performance of the microalgae cells [15,16]. Finally, the presence of predators or parasites such as chytrids can dramatically reduce the oxygen production and CO_2 demand of whatever microalgae culture [11]. To evaluate the performance of microalgae culture produced in large-scale reactors, only offline measurements such as dry weight or photorespirometric methodologies are applied [17]. However, this is a critical parameter for the optimal management of the reactor and online measurements are necessary for the continuous optimization of these production systems.

In recent years, extensive efforts have been made to optimize the design and operation of open photobioreactors for large-scale microalgae cultivation. However, accurate and continuous monitoring of key parameters such as oxygen production and mass transfer remains a technical challenge, especially under dynamic outdoor conditions. Traditional methods for determining the volumetric oxygen mass transfer coefficient (k_La) often involve invasive or non-continuous techniques such as gas stripping, oxygen enrichment pulses, or dark/light cycling, which are difficult to apply at large scale and may disturb the system's natural dynamics [18]. In contrast, the methodology proposed in this work enables the online and in situ determination of both the oxygen production rate and mass transfer capacity using only routinely monitored operational data (e.g., temperature, flow rates, solar irradiance, and dissolved oxygen). This represents a significant advancement, as it allows for continuous assessment under steady-state, real-world conditions, without the need for tracer gases or disruptive interventions. The application of this approach in raceway reactors offers a novel solution to quantify photosynthetic activity and gas exchange efficiency at scale, contributing to more robust process control and optimization in microalgae-based production systems.

The implementation of adequate control strategies is crucial to ensure the optimal performance of whatever bioprocess, including microalgae-related ones. Advanced control strategies are developed based on the knowledge of the behaviour of the systems. If this behaviour is modified as a consequence of changes in the state of the microalgae cultures, the control strategies must be also readjusted. It is possible to develop automatic routines for the online adjustment of control parameters but all of them require online measurements of the behaviour of the system [19]. Moreover, the availability of adequate models predicting the behaviour of the cultures allows the development of model predictive control systems that anticipate these changes for optimal performance of the systems [20,21]. For this purpose, the online

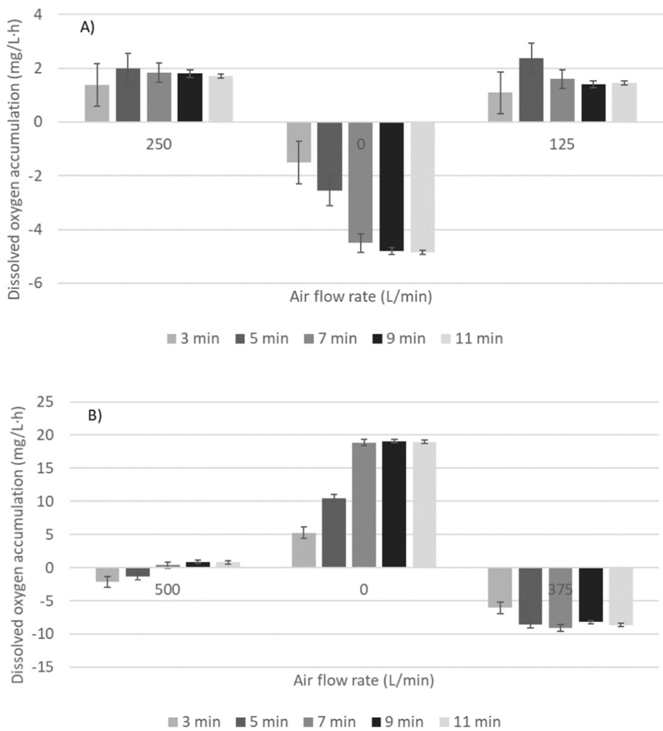

Fig. 4. Variation of dissolved oxygen accumulation at the end of each cycle as a function of aeration rate during the night (A) and daylight period (B), calculated at different frequencies.

measurement of the mass transfer capacity of the reactor and the performance of the microalgae cells is highly valuable.

This work aims to develop a methodology for the development of online sensors to evaluate both the mass transfer capacity and oxygen production rate in large-scale microalgae reactors. The methodology has been developed for raceway reactors, but it can be also applied to other types of photobioreactors. Moreover, the methodology is useful for developing robust and reliable models about the performance of microalgae cultures to be used for the development of advanced control systems that maximize the performance of large-scale microalgae production systems. In summary, the proposed methodology is a powerful tool in the scale-up and optimization of large-scale raceways for microalgae production.

2. Materials and methods

2.1. Microorganism and culture medium

The microalgae strain *Scenedesmus almeriensis* (CCAP 276/24) was used to inoculate the raceway. This strain was selected due to its tolerance to variable growth conditions and robustness versus contaminations and grazers. Assays were performed using Mann & Myers medium prepared using fresh water with fertilizers (FW) (0.9 g·L^{-1} $NaNO_3$, 0.14 g·L^{-1} $KH_2(PO_4)$, 0.18 g·L^{-1} $Mg(SO_4)_2$ and 0.02 g·L^{-1} Karentol (Konegard, Spain)).

2.2. Raceway reactors

Experiments were performed in an 80 m^2 raceway reactor located at the "IFAPA" Research Centre, 36° 48′N–2° 43′W, (Almería, Spain). The reactor consists of two 50 m long channels (0.46 m high × 1 m wide), both connected by 180° bends at each end, with a 0.59 m^3 sump (0.65 m long × 0.90 m wide × 1 m deep) located 1 m along one of the channels. The total volume of the liquid culture is 13.4 m^3, of which 12.8 m^3 is in the channel and 0.6 m^3 is in the sump. The pH, temperature and DO in the culture were measured using appropriate probes (5083 for pH/T and 5120 for DO/T, Crison, Barcelona, Spain), connected to an MM44 control-transmitter unit (Crison Instruments, Barcelona, Spain), and data acquisition software (DAQFactory, Anchorage, USA) providing complete monitoring and control of the installation.

The optimal position to locate dissolved oxygen probes in raceway reactors is at the end of the channel, before the paddlewheel, as it is where the major variations in dissolved oxygen can be determined throughout the whole day. The gas flow rate entering the reactor was measured by a mass flow meter (PFM 725SF01-F, SMC, Tokyo, Japan). The pH of the culture was controlled at 8.0 by on-demand injection of CO_2. Temperature was not controlled, it ranged ±5 °C from the daily mean air temperature, which varied from 20 °C in April to 25 °C in July. Air was supplied to the reactor from a blower providing 350 mbar overpressure, through a fine bubble diffuser AFT2100 (ECOTEC, Sallent, Spain) providing bubbles with a diameter smaller than 2 mm at the minimum pressure drop; the estimated residence time of the bubbles in the sump ranged from 5 to 10 s.

A SCADA (Supervisory Control and Data Acquisition) system was implemented to enable real-time monitoring and control of the raceway reactor, ensuring the continuous collection of key process and environmental variables. The system is based on a distributed architecture integrating sensors such as for pH, dissolved oxygen, temperature, flowmeters and water level within the reactor, alongside meteorological inputs such as solar radiation, air temperature, and wind speed. All sensors operate with a sampling frequency of 10 s, enabling the detection of rapid process fluctuations and supporting adaptive control strategies. Data are acquired and managed using a central control unit connected to the instrumentation via industrial communication protocols, and visualized through a human-machine interface (HMI) that allows remote supervision and alarm management [22].

The raceway reactor was operated at a constant culture depth of 0.15 m, in semi-continuous mode, at a fixed dilution rate of 0.2 day^{-1}, to achieve a biomass concentration of 0.8 g/L Biomass was harvested and fresh medium was added daily to avoid the accumulation of biomass thus operating in a steady state, although variations of the biomass concentration take place due to perturbations (environmental and operational). Evaporation inside the reactor was compensated by the daily addition of fresh water. The biomass concentration was evaluated on a daily base by measuring the dry weight of a sample taken in the morning before harvesting/dilution of the culture. For that 40 mL aliquots of the culture were filtered through a pre-dried 0.45 μm filter (Macherey-Nagel GmbH & Co. KG, Düren, Germany). Then, the filters were dried in an oven at 80 °C for 24 h. The nutrient content of the supernatant was also daily evaluated to confirm that no nutrient limitation took place.

3. Proposed methodology

The methodology is based on providing air pulses into the sump during short intervals. From the variation of dissolved oxygen concentration in time, the oxygen production (photosynthesis rate) and exchange (mass transfer) into the reactor could be determined. The pulses are performed throughout the entire day to evaluate the variation of photosynthetic performance along the solar cycle, whereas the mass transfer is only a function of the gas flow rate provided to the sump because the other variables such as the type of diffusor and liquid velocity remained constant.

The proposed methodology is based on the oxygen mass balance of the entire reactor [23]. When oxygen is either produced (day) or consumed (night) by the microalgae cells (PO_2), the oxygen is transferred from/to the atmosphere at the different sections of the raceway reactor such as the channel ($N_{O2,channel}$), the paddlewheel ($N_{O2,paddlewheel}$) or the sump ($N_{O2,sump}$), or it is accumulated into the culture (Eq. (1)).

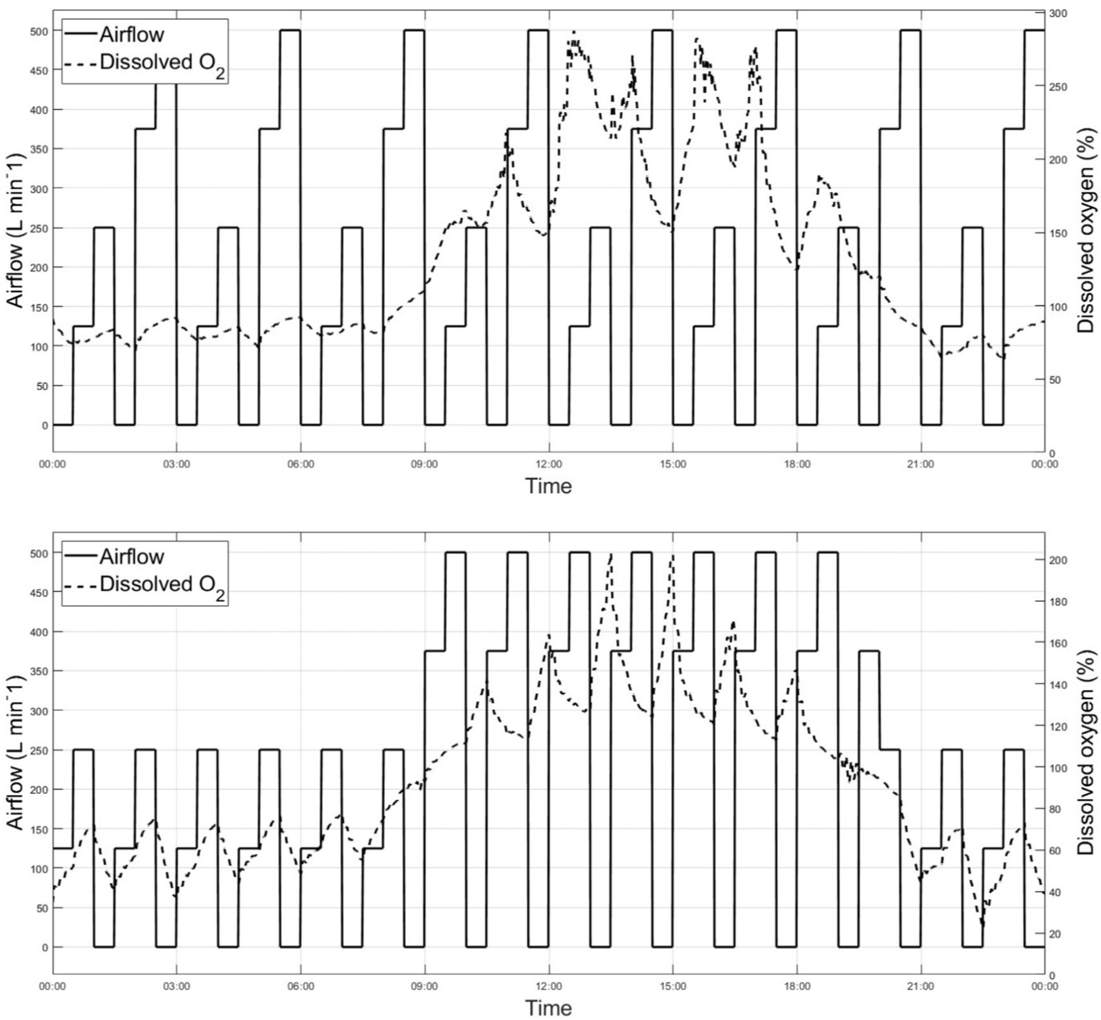

Fig. 5. Daily variation of dissolved oxygen concentration in the 80 m^2 raceway reactor when providing different series of pulses. A) An homogeneous series of pulses with no aeration into the sump every two aerated pulses, and sequential airflow rates of 125, 250, 375 and 500 L/min, B) a non-homogeneous series of pulses with airflow rates of 125 and 250 L/min during the night period and 375 and 500 L/min during the daylight period.

$$V_{total}\frac{d[O_2]}{dt} = P_{O_2} + N_{O_2,channel} + N_{O_2,paddlewheel} + N_{O_2,sump} \quad (1)$$

In general, the mass transfer rate is calculated as a function of the mass transfer coefficient (k_La_L) multiplied by the driving force ($[O_2] - [O_2^*]$) and the volume of the liquid in each section (Eq. (2)). The dissolved oxygen concentration in the liquid ($[O_2]$) is measured using adequate dissolved oxygen probes. The dissolved oxygen concentration in equilibrium with the gas phase (air) ($[O_2^*]$) is calculated by Henry's law. It is a function of temperature affecting the solubility of dissolved oxygen according to Eq. (3).

$$NO_2 = k_La_L\ ([O_2] - [O_2^{\ *}])\ V \quad (2)$$

$$[O_2^{\ *}] = 12.408 - 0.1658{*}T \quad (3)$$

The mass transfer coefficient into the channel and paddlewheel remained constant because both the liquid velocity and the rotation speed of the paddlewheel were kept constant. The mass transfer coefficient in the channel ($N_{O_2,channel}$) was reported to be 0.9 h^{-1} and the mass transfer coefficient in the paddlewheel ($N_{O_2,paddlewheel}$) was reported to be 164 h^{-1} [6]. When no air is provided to the sump, the mass transfer coefficient in this section is zero. Then, the oxygen production rate can be calculated as a function of mass transfer into the channel ($N_{O_2,channel}$) and paddlewheel ($N_{O_2,paddlewheel}$) plus the dissolved oxygen accumulation (P_{O_2}) (Eq. (4)). Knowing the oxygen production rate and providing different air flow rates to the sump, the mass transfer rate into the sump ($N_{O_2,sump}$) can be calculated considering the oxygen production rate previously determined in addition to the mass transfer rate into the channel and paddlewheel (Eq. (5)).

$$P_{O_2} = N_{O_2,channel} + N_{O_2,paddlewheel} - V_{total}\frac{d[O_2]}{dt} \quad (4)$$

$$N_{O_2,sump} = P_{O_2} + N_{O_2,channel} + N_{O_2,paddlewheel} - V_{total}\frac{d[O_2]}{dt} \quad (5)$$

The challenge is to determine the optimal distribution of pulses along the day and the intensity of these pulses to firstly not negatively affect the performance of the cultures, and secondly to obtain accurate data about both mass transfer capacity and oxygen production rates.

4. Results and discussion

In large-scale raceway reactors, the microalgae cultures are exposed to environmental and operational conditions that determine the overall performance of the microalgae cells. Fig. 1 shows the typical variation of solar radiation and temperature in outdoor raceway reactors, including the variation of dissolved oxygen concentration, when not providing aeration into the sump. During the night, there is no light and the

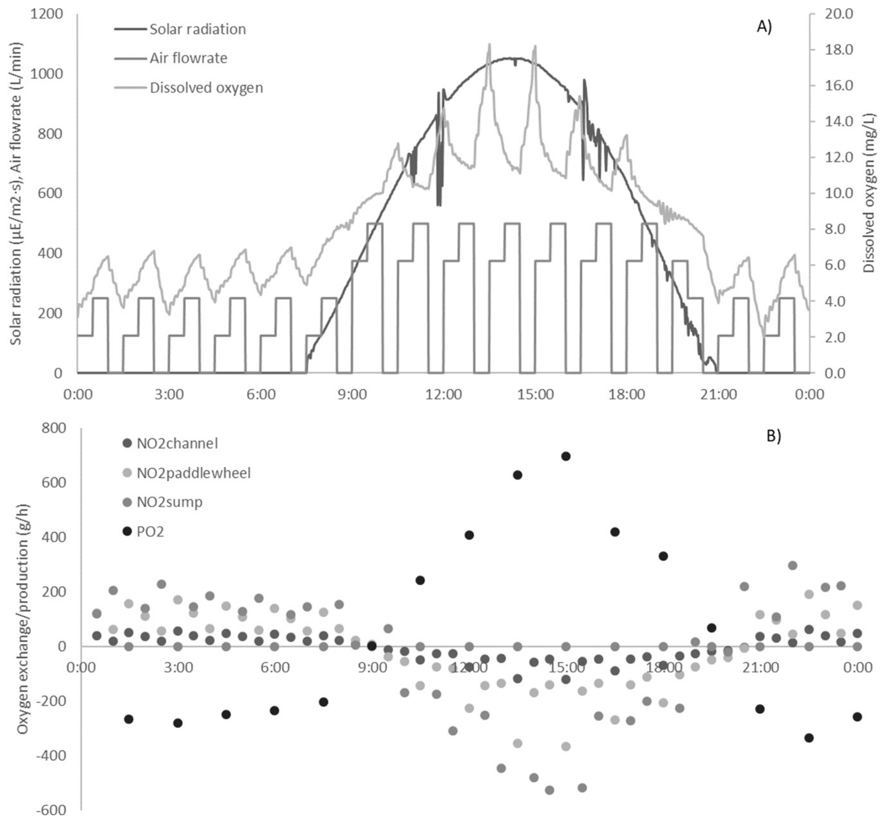

Fig. 6. Daily variation of solar radiation, dissolved oxygen and air flow rate provided to the sump (A), in addition to parameters determined using the proposed methodology of driving force and mass transfer coefficient into the sump along the day (B).

microalgae cells perform respiration, then the dissolved oxygen concentration reduces below 100 %Sat. Minimum values of dissolved oxygen concentration of 30 %Sat indicate that the respiration rate is not enough to achieve anaerobic conditions in the system. Anaerobic conditions in microalgae cultures can cause severe damage, affecting growth, biomass production, and cellular stability. Oxygen deprivation disrupts cellular respiration, reducing ATP synthesis and leading to the accumulation of toxic metabolites [24]. Additionally, it promotes the proliferation of undesirable microorganisms and the degradation of photosynthetic pigments, decreasing photosynthetic efficiency and potentially causing culture collapse. To prevent these effects, proper aeration, agitation, and monitoring of dissolved oxygen are essential [25]. During the daylight period, the cells perform photosynthesis then the dissolved oxygen concentration increases by more than 100 %Sat. Maximum values up to 400 %Sat can be measured although it is reported that values higher than 200 %Sat strongly reduce the performance of the photosynthesis process [26]. One primary adverse effect of elevated dissolved oxygen concentration is the enhancement of photorespiration. Under high oxygen conditions, the enzyme RuBisCO favours oxygenation over carboxylation, leading to increased photorespiration rates and decreased photosynthetic efficiency. This shift results in reduced carbon fixation and, consequently, lower biomass yields [27].

Whatever the daily period mass transfer between the culture and the air ambient takes place, thus oxygen is exchanged through the surface of the culture into the channel and paddlewheel. Because the characteristics of both paddlewheel and channel don't modify during the regular operation of the raceway the mass transfer phenomena in these sections are solely a function of changes in the driving force into the system, which are determined as the difference between the dissolved oxygen concentration in the liquid and dissolved oxygen concentration in equilibrium with air (saturation). Because the respiration rate is much lower than the photosynthesis rate (ten times on average) the oxygen adsorption during the night prevents the achievement of anaerobic conditions in the reactor. However, oxygen desorption during the daylight period is not enough to prevent over-accumulation of dissolved oxygen, especially in large-scale reactors. To solve this problem the use of sumps on which aeration is provided has been proposed [6]. This strategy is now being used in most industrial raceways. If adequately designed and operated this sump allows to keep the dissolved oxygen concentration in the culture controlled to maximize the performance of the cultures [28]. In this sense, to be able to determine and optimize the mass transfer capacity into the sump as a function of the airflow rate provided is highly relevant.

The objective of this work was to develop and optimize a methodology for the online characterization of mass transfer and photosynthetic performance of microalgae cultures in raceway reactors. The proposed methodology was defined based on knowledge of the boundary conditions of the system. Thus, the cycling time into the channel was 7.3 min as determined following the trace-response pulse methodology [29]. Based on this value a minimum time for pulses equal to three times the cycling time was defined, up to 30 min. Regarding the airflow rate, the maximum value achievable was 500 L/min, then pulse values of 125, 250, 375 and 500 L/min were defined, in addition to airflow equal to zero to evaluate the photosynthetic performance without mass transfer into the sump. An example of the results obtained in a single day under these conditions is shown in Fig. 2. During the night, the DO concentration in the culture was reduced up to 4 mg/L, lower than the saturation level at equilibrium with atmospheric air, due to respiration by the cells. When providing aeration, the DO concentration increases up to 6 mg/L, due to oxygen absorption into the sump, whereas DO concentration reduces when no aeration is provided due to respiration (Fig. 2A). The DO concentration also modifies with the time at which the aeration rate is provided. Initially, it increases very fast to achieve a constant slope when approaching the pulse maximal time of 30 min. Because aeration provokes a change in the behaviour of the system a certain time is required to achieve the new pseudo-steady state. A

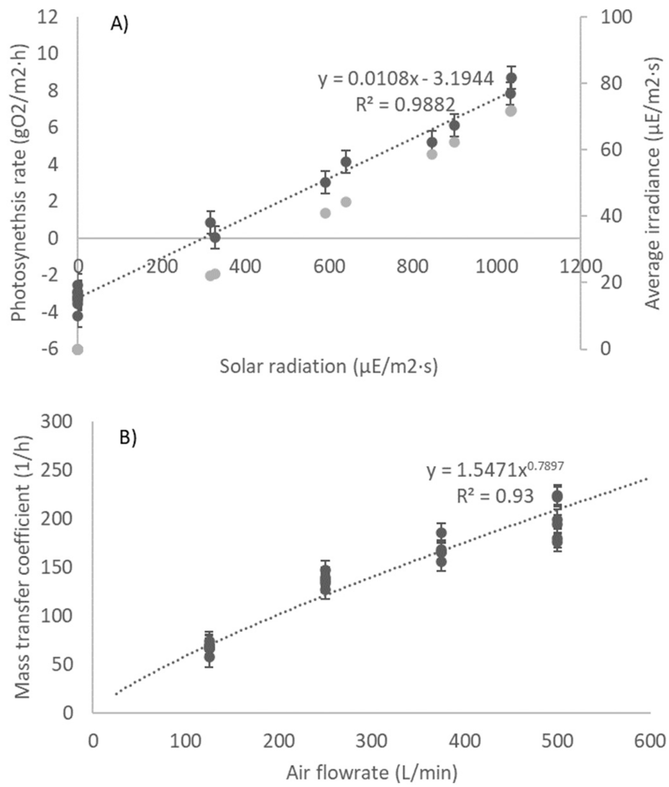

Fig. 7. Modelling of photosynthesis rate (A) and mass transfer coefficient into the sump (B) as a function of solar radiation and air flow rate provided to the sump. Values were obtained from the daily characterization of the performance of the raceway reactor using the proposed methodology.

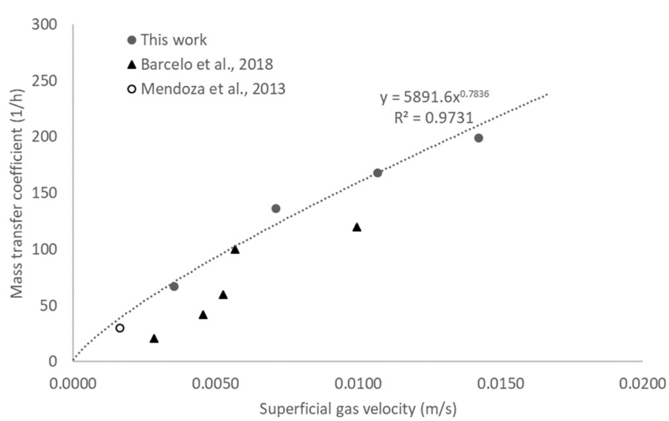

Fig. 8. Variation of mass transfer coefficient as a function of superficial gas velocity into the sump from different sources. The line corresponds to the correlation of experimental data from this work to a potential function.

similar trend is observed during the daylight period but in this case, the DO concentration is over the saturation level at equilibrium with atmospheric air, values ranging from 12 to 18 mg/L (Fig. 2B). DO concentration decreases when providing air due to oxygen desorption into the sump, and increases when no aeration is provided due to oxygen production and accumulation into the reactor, despite oxygen desorption taking place into the paddlewheel and channel.

The proposed methodology is based on the determination of the DO accumulation rate in the system at different conditions, then closing the oxygen mass balances to estimate both the oxygen transfer and production rates. In each pulse, after changing the air flow rate, it takes time for the system to accumulate steadily oxygen. Then it is necessary to determine the most adequate method to calculate it from experimental data. The variation of dissolved oxygen accumulation as a function of time at different aeration rates during the night and daylight period, calculated at different frequencies along each cycle is shown in Fig. 3. An oscillating behaviour is visible in the oxygen accumulation. The period of the oscillations corresponds to the time for the liquid to circulate through the raceway reactor. Results show that the accumulation of dissolved oxygen, calculated as the slope of DO with the time at different frequencies, from 3 to 11 min, is unstable. These frequencies were defined considering the cycling time of 7 min experimentally determined as the central point, and two values higher and lower than this value to analyze the influence of this variable in the results. Values of DO accumulation at frequencies of 3, 5 and 7 min show a large variability whereas the values calculated at frequencies of 9 and 11 min show a much more stable behaviour (Fig. 3). Moreover, data calculated at the beginning of each pulse are largely affected by the previous state of the system or the perturbation created by the change in the airflow rate. However, the values become stable at the end of the pulse, remaining stable during the last minutes of each cycle, especially at frequencies of 9 and 11 min (Fig. 3). During the night only the slopes calculated at frequencies of 7 to 11 min are negative when not providing aeration as expected for respiration rate, whereas the slope becomes positive at the same frequencies when providing aeration due to absorption of oxygen into the sump (Fig. 3A). Opposite, during the daylight period the slope of DO concentration with time is positive when no aeration due to the accumulation of oxygen produced by photosynthesis, whereas the slope is close to zero or negative when providing aeration due to the desorption of oxygen into the sump (Fig. 3B).

To determine the precise values of frequency and times to be used to calculate the accumulation of DO into the system an analysis of results obtained in these first tests was performed. The mean values of DO accumulation into the reactor at the end of each cycle as a function of airflow during the night and daylight period are shown in Fig. 4. Results show that the mean values and standard deviation of measurements performed at frequencies of 3 and 5 min were not adequate for all measurements. However, the accuracy of the method increases when using frequencies of 7 to 11 min to calculate the DO accumulation in the reactor, with no statistical difference being observed in this range of frequencies. From these data, it is concluded that pulses longer than three times the cycling time on the reactor must be utilized, with a final duration of each pulse of 30 min being defined. The accumulation of DO in the reactor must be calculated at the end of each pulse, the slope of DO concentration with time during a minimum of 7 min must be used for that. However, to increase the accuracy of the methodology it is recommended to determine the accumulation of DO as the slope of DO concentration in time during the last 9 min.

Because the duration of each pulse is 30 min a total number of 48 pulses per day can be performed. To enhance the monitoring of the performance of the culture, a pulse without aeration was performed for each two pulses with aeration. In this way, up to 16 pulses without aeration can be performed to monitor the daily variation of oxygen production rate along the solar cycle. For pulses with aeration, a sequential series of pulses at aeration rates of 125, 250, 375 and 500 L/min were defined during day and night. However, DO concentration during the night was close to the equilibrium concentrations. Therefore, the oxygen driving force was insufficient to calculate the mass transfer rate. On the other hand, the DO concentration during the daylight period increased up to values of 300 %Sat (Fig. 5A). The excess DO concentration damages the performance of the microalgae cells [6]. Therefore, the photosynthesis rate measured at these conditions is not representative of the real performance of the cells under appropriate culture conditions.

To overcome these problems a new series of pulses was designed. The frequency of non-aerated pulses remained constant but during the night only pulses at lower aeration rates (125 and 250 L/min) were performed to minimize oxygen adsorption and to maximize the driving force.

During the daylight period, higher air flow rates are provided (375 and 500 L/min) to maximize oxygen desorption and avoid excess DO concentration in the culture. Results obtained when operating in this way confirm that at these conditions the DO concentration during the night was lower than saturation whereas the DO concentration during the daylight period remains below 200 %Sat avoiding excess dissolved oxygen concentration that could damage the performance of microalgae cells (Fig. 5B). Based on the results this configuration was selected as optimal.

Once the methodology was validated, it was applied to determine the oxygen exchange and production rate in the reactor. The methodology requires knowing the mass transfer capacity into the channel and paddlewheel to determine the oxygen production rate from the measurements performed without aeration into the sump, and to calculate the mass transfer into the sump when aeration is provided at different flow rates. The mass transfer coefficient into the channel and paddlewheel was previously reported to be 0.9 and 164 h^{-1}, respectively [6]. The mass transfer coefficient into the channel is low due to the poor contact between the culture and the air in the channel. Because the recommended liquid velocity in raceway reactors is 0.2 m/s to avoid settling of the cells and excess energy consumption, the mass transfer coefficient into the channel is constant for whatever raceway reactor and similar values have been reported in other studies [7]. Concerning the paddlewheel, the mass transfer coefficient in this section is much higher due to the intense mixing of air and culture induced by the rotation of the paddlewheel. It has been reported that modifications of paddlewheel rotation speed increase the mixing and mass transfer into raceway reactors [30,31]. However, the total volume of this section is low in comparison with the total volume of the reactor. Moreover, the rotation speed of the paddlewheel is related to the liquid velocity, it also remains constant for most of the raceway reactors. Thus, the total contribution of the paddlewheel to mass transfer in large-scale raceway reactors is limited and cannot be enlarged. By using these values the oxygen exchange and production rates were experimentally determined (Fig. 6).

Results show that the daily variation of solar radiation provokes a variation of DO concentration that is perturbed by the supply of air at different flow rates, but always keeping the DO in the range of 3 to 18 mg/L to prevent anaerobic conditions and excess of DO concentration that could damage the microalgae cells (Fig. 6A). Applying the proposed methodology the oxygen exchange in each zone of the reactor ($NO_{2,channel}$, $NO_{2,paddlewheel}$, $NO_{2,sump}$) and the overall oxygen production rate (PO_2) are calculated. Regarding the oxygen production rate, a clear variation with the solar radiation is observed, with values ranging from −335 g/h during the night to +700 g/h during the daylight period (Fig. 6B). Regarding oxygen exchange, values modify along the solar cycle due to variations in the driving force. It is confirmed that oxygen exchange into the channel represents a minor contribution, with values ranging from −120 to 63 g/h, whereas into the paddlewheel the values ranged from −360 to 192 g/h. The oxygen exchange into the sump is the only one affected by the modifications of the aeration rate provided, with values ranging from −530 to 300 g/h, with maximum values being measured at maximal aeration rates of 500 L/min (Fig. 6B). These values are highly relevant to elucidate the major phenomena taking place in the system but moreover, they are useful for the characterization and modelling of both the photosynthetic performance of microalgae cultures and the mass transfer capacity of the raceway reactor.

The variation of photosynthesis rate per unit surface as a function of solar radiation is shown in Fig. 7A. Results show that the photosynthesis rate per unit surface increases from −3.2 to +8.7 $gO_2/m^2{\cdot}h$ when the solar radiation modifies from zero during the night to values up to 1030 $\mu E/m^2{\cdot}s$ at midday. The variation of the photosynthesis rate with the solar radiation confirms that the oxygen desorption capacity required to avoid achieving DO concentrations higher than 200 %Sat is not constant then the air flow rate required to be provided to the sump must be adjusted to the oxygen production into the reactor [7]. A linear correlation was observed between the photosynthetic oxygen production rate per unit surface area and the incident solar radiation on the reactor surface, indicating that the cultures operated primarily under light-limited conditions. Although photoinhibition has been reported in outdoor reactors during peak daylight hours, particularly under low-density cultures, this effect was not observed in our study. This is likely due to the maintenance of high biomass concentrations, which effectively shield cells in deeper layers from excessive light. As a result, the average irradiance experienced by the microalgae within the culture is significantly lower than the surface irradiance [3,32]. In our system, calculated average irradiance values within the culture reached a maximum of 70 $\mu E{\cdot}m^{-2}{\cdot}s^{-1}$, well below the saturation threshold for the produced strain (300 $\mu E{\cdot}m^{-2}{\cdot}s^{-1}$) and even lower than the half-saturation irradiance (~175 $\mu E{\cdot}m^{-2}{\cdot}s^{-1}$) [33]. This internal light environment supports the observed photolimitation and explains the linear photosynthetic response across the range of operational conditions. Furthermore, a linear relationship was also confirmed between oxygen production and the calculated internal irradiance, reinforcing the robustness of the system and its sensitivity to changes in light availability within the culture depth.

Moreover, the oxygen production rate can be used as an estimation of biomass productivity. The developed methodology is useful as a virtual sensor of oxygen and biomass production rates. A correlation between the mass transfer coefficient in the sump and the aeration flow rate was found (Fig. 7B). It was previously reported that the mass transfer coefficient in the sump increased from 22 to 118 h^{-1} when the air flow rate increased from 50 to 350 L/min for a similar raceway reactor [7]. A similar trend was also observed in this work, confirming that the most suitable method to increase the oxygen desorption capacity in raceway reactors is to increase the airflow rate provided to the sump. However, the airflow rate cannot be increased infinitely moreover the higher the airflow rate the higher the energy consumption of the entire system.

The superficial gas flow rate (U_{gr}), defined as the gas flow rate to the cross-section of the aerated section ratio, is the key parameter representing the intensity of aeration in bubble column systems. It ranges from 0.005 to 0.020 m/s for homogeneous flow regime characterized by uniform bubble distribution and minimal coalescence, to 0.02–0.10 m/s in transition to heterogeneous flow with increased bubble interactions and coalescence, and to 0.1–0.5 m/s for heterogeneous churn-turbulent flow characterized by large irregular bubbles and vigorous mixing [34]. When analyzing data from available studies it is concluded that the superficial gas velocity provided into the sump is always lower than 0.02 m/s, thus operating in a homogenous regime to maximize the efficiency of aeration by avoiding coalescence of bubbles and favouring the presence of small and efficient bubbles (Fig. 8). At these conditions, the maximum values of the mass transfer coefficient of 200 h^{-1} are achieved at maximal superficial gas velocities of 0.015 m/s. The same tendency is observed for all the datasets available, although the behaviour of each system is a function of the configuration of each system such as the number and type of diffusers, distribution of the diffusers into the sump, etc. [6,7].

In summary, the proposed methodology is highly interesting for the onsite characterization of the mass transfer capacity of large-scale raceway reactors, and to evaluate potential improvements that could be necessary for optimal control of DO concentration and microalgae cultures performance. The methodology has been developed and validated for raceway reactors but it can be also useful for other types of photobioreactors such as tubular ones in which there is no mass transfer into the pump or tubes (when not providing gases into the tubes), thus simplifying the calculations. The application to flat panels or thin-layer cascades is also possible, currently being under development. Whatever the reactor, the automatic implementation of the methodology constitutes a virtual sensor for the online monitoring of microalgae production systems, from both engineering and biological points of view.

5. Conclusions

This study successfully developed and validated a real-time methodology for measuring mass transfer coefficients (K_la_l) and oxygen production rates (PO_2) in large-scale raceway reactors using dissolved oxygen (DO) monitoring and controlled air pulse injections. The results confirm that the sump section plays a crucial role in oxygen desorption, in average up to 60 % of oxygen desorption takes place in the sump whereas 30 % is performed into the paddlewheel and only 10 % into the channel, at scales of 80 m^2. Oxygen transfer is a function of volume and driving force, but mainly of the mass transfer coefficient in each zone of the reactor. The methodology proposed allows the determination of the mass transfer coefficient (K_la_l) into the sump, with values reaching up to 200 h^{-1} at superficial gas velocities below 0.02 m/s, ensuring a homogeneous aeration regime. Compared to the sump, the channel ($K_la_l = 0.9$ h^{-1}) and paddlewheel ($K_la_l = 164$ h^{-1}) contributed minimally to mass transfer, highlighting the importance of optimizing sump aeration. Additionally, a linear correlation between oxygen production and solar radiation was found, demonstrating that oxygen evolution can serve as a reliable proxy for biomass productivity. The proposed methodology provides a powerful tool for optimizing large-scale microalgae production, allowing real-time monitoring and adaptive process control to enhance biomass productivity. These findings contribute to the development of more efficient and sustainable industrial-scale microalgae cultivation systems.

CRediT authorship contribution statement

R. Arraga: Methodology, Investigation. **N.A. Dambruin:** Resources, Project administration. **M. Barceló-Villalobos:** Methodology, Investigation, Data curation. **M. Janssen:** Supervision, Software, Resources, Investigation. **F.G. Acién:** conceptualized the study, supervised all aspects of the project, coordinated the collaboration between institutions, and led the writing and final revision of the manuscript.

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.

This study was conducted as part of collaborative research between academic and industrial partners, including the University of Almeria, Wageningen University, and CHLYDRO S.L., within the framework of publicly funded research projects supported by the Spanish Ministry of Science, the Andalusian regional government, and the European Union (REALM, NIAGARA, and COSEC projects).

All funding sources are acknowledged transparently, and none of the funding bodies had any role in the study design, data collection, interpretation, or decision to submit the manuscript for publication.

Acknowledgements

This work was funded by the Spanish Ministry of Science (PID2023-150739OB-I00), the regional government of Andalusia (DGF_PLSQ_2023_00233; PCM_00083) and the European Union (REALM, grant agreement ID: 101060991; NIAGARA, grant agreement ID: 101146861; COSEC, grant agreement ID: 101172850).

Data availability

Data will be made available on request.

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