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Hollow glass microspheres for temperature and irradiance control in photobioreactors



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HIGHLIGHTS

- Photobioreactors made of a HGM polymer are cheaper to run.
- The broth temperature can be reduced 7 °C using 0.6 vol.% HGM in the reactor wall.
- HGM composites have mechanical properties suitable for bioreactor manufacture.
- Growth rate is improved by up to 33% using 0.6 vol.% HGM comp. in the reactor wall.

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ABSTRACT

The addition of hollow glass microspheres (HGM) to polymers to change thermal insulation and mechanical properties is widely used. In this study HGM were tested as a new construction material for photobioreactors to control irradiance and broth temperature in microalgae cultivation. The heat isolation properties of HGMs of three different densities were tested in a polymer matrix. The transmittance (5–50%) and the thermal conductivity (182.05–190.73 W/mK) of the HGM composite material were analyzed. The results were tested in a model to predict the broth temperature and the growth rate as a function of temperature and irradiance. The addition of 1.3 and 0.6 vol.% of HGM lead to an increase in the growth rate of up to 37% and a reduction in the broth temperature up to 9 °C. The mechanical resistance of the composites tested is similar to the polymer matrix.

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1. Introduction

Since early studies in biotechnology the technical focus of the outdoor mass cultivation of microalgae has been the photobioreactor. The design of the photobioreactors should maximize the amount of irradiation received (Chen et al., 2011; Mohsenpour and Willoughby, 2013; Pegallapati et al., 2012) therefore the main concern in biomass productivity is to make effective use of light irradiance in the microalgae culture (Kumar et al., 2013). Besides the effect of direct and reflected solar irradiation, the broth temperature in the reactor also has an impact on microalgae growth

rates (Franz et al., 2012; Gomez and Gonzalez, 2005; Pereira et al., 2013; Sheng et al., 2011).

According to Franz et al. (2012) the irradiation rate supplied to the photobioreactor throughout the day can be described as a function of prevailing geographical and climatic conditions. Furthermore, maximum annual yields were achieved in regions with high irradiation and temperature patterns in or near the optimum range of the specific algal strain. The limitations of outdoor full scale production of microalgae imposed by extreme irradiation and high temperatures are generally controlled by shading the reactor surface, using external cooling systems such as water-spray on the reactor or internal heat exchangers (Gutiérrez et al., 2008; Quinn et al., 2012; Sierra et al., 2008). Other solutions have focused on the geometry of the photobioreactors for spatial dilution of light, temperature-controlled greenhouses or installation facilities such as an artificial body of water to moderate the day-night temperature cycles (Carlozzi and Sacchi, 2001; Chen et al., 2011; Hulatt and Thomas, 2011; Masojídek et al., 2003; Oncel and

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Sabankay, 2012). However, most of these solutions are costly and consume large amounts of water and energy.

In fact most research has focused on the modification of the design and geometry of the photobioreactor to enhance biomass productivity. However, most of the novel photobioreactors are not suitable for large scale production of microalgae culture because of the high cost of manufacture and operation. An alternative is to evaluate the use of new materials to build photobioreactors in order to control irradiance and temperature. For example, the development of thermal insulate composite materials for the construction of photobioreactors can be tested. Irradiance can be controlled by changing the construction material of the photobioreactor that will affect the heat exchanged to the environment. The transparent material used in photobioreactors is usually polyethylene (PE), polycarbonate (PC), polyvinyl chloride (PVC), polymethyl methacrylate (PMM), polypropylene (PP), glass and silicate (Richmond, 2004). These are low cost materials and their manufacturing/shaping and transport low cost too. Yet for environmental reasons the photobioreactor should be recyclable and therefore polyethylene terephthalate (PET) should also been tested.

A considerable number of additives have been tested to develop polymer composite materials for different industrial application with low thermal conductivity such as hollow glass microspheres (HGM). However, HGM composite to be used as a construction material for photobioreactors to microalgae cultivation is a new issue. HGM is an inorganic, finely dispersed spherical powder material and the hollow core gives HGM a thermal insulation property. Li et al. (2011) evaluate the mechanism of heat transfer of HGM showing the low thermal conductivity of this material. Low thermal high density polyethylene (HDPE) HGM composite was tested by Patankar and Kranov (2010). Based on their findings and others regarding the insulation property of HGM (Dombrovsky et al., 2007; Gao et al., 2013; Hu et al., 2013; Park et al., 2005) this study focuses on the use of hollow glass microspheres to enhance the thermal insulation of a flat plate photobioreactor controlling light irradiance and broth temperature due to the reduction of the wall transmittance and the insulation property of the material. The results of the characterization of the polymer HGM composite are used as input parameters in the model developed by Béchet et al. (2010) to predict the broth temperature. The microalgae growth rate is estimated according to the model developed by Bernard and Rémond (2012). Thus, the overall aim of this study was to find the ideal concentration of HGM added to photobioreactor construction material in order to increase specific growth rate by controlling broth temperature and wall transmittance.

2. Methods

The materials used in this study included commercially available isophthalic polyester resin (PR) and three different sodium borosilicate hollow glass types marketed by 3 M. The features of the three different HGM tested are presented in Table 1. Other physical parameters provided by the supplier are the density of the micro-spherical shell (2.23 g/cm³), the thermal conductivity (0.023 W/mK) and the density of the gas phase inside of the HGM (7.50.10⁻⁵ g/cm³). The volume fraction of the microspheres in the PR HGM composite tested are 5.0, 2.5, 1.3 and 0.6 vol.% for

each type of microsphere. The methods for the characterization and evaluation of the composites are described as follows.

2.1. Synthesis of the polymeric matrix

The synthesis of the polymeric matrix was carried out using polyester resin with the addition of 2 vol.% of methyl ethyl ketone peroxide catalyst (matrix). The matrix was molded by removing it before the formation of polyester and then it was placed into silicone molds.

2.2. Preparation of the composites

The preparation of the composites followed the same method described above. Before the polymer reached the sol–gel state, the HGM was added to the reaction with fixed stirring for 2 min and placing it into the silicone molds.

2.3. Techniques used in the characterization of the materials

2.3.1. Tensile test and flexure test

Tensile and flexure tests were performed using universal testing equipment (EMIC, DL2000) according to ISO 527 and ISO 178 respectively. The displacement speed for the tensile test were 2 mm/min and for flexure test were 3 mm/min, both test used 2000 N load cell and distance between grips equal to 100 mm. In addition for tensile and flexure tests eight and six, respectively, tests were done.

2.3.2. Transmittance

The transmittance of the matrix and composites were carried out on a Cary 60 UV–Vis spectrophotometer. The samples were loaded and polymerized in a plastic flow cell and were measured in the region of 400–1100 nm at room temperature. The mean values obtained by the *t*-Student test are the results from three readings of the matrix and the PR HGM composites C1, V5 and H6 each at four different concentrations.

2.3.3. Thermal conductivity

The thermal conductivity of the PR matrix and PR HGM composites was evaluated according the model presented by Liang and Li (2007):

$$k_{eff} = (1/k_p(1 - 6\phi_f/\pi)^{1/3} + 2(k_p(4\pi/3\phi_f)^{1/3} + \pi(2\phi_f/9\pi)^{1/3}(k_g((\rho_s - \rho_a)/(\rho_g - \rho_a)) + k_a((\rho_g - \rho_s)/(\rho_g - \rho_a)) - k_p))^{-1} \quad (1)$$

where k_{eff} is the specific equivalent thermal conductivity (W/mK); k_p , k_g and k_a are the thermal conductivities of the polyester resin, micro-spherical shell and gas phase of the HGM, respectively (W/mK); ϕ_f is the volume fraction of the HGM in the composite (vol.%); and ρ_s , ρ_g and ρ_a are the densities of the HGM, micro-spherical shell of the HGM and the gas phase inside the HGM, respectively (kg/m³).

Table 1
Basic features of the HGM tested.

Sample	Density (g/cm ³)	Size (μm)	Crushing strength (psi)	Thermal conductivity (W/mK) @ 21 °C
HGM C1	0.12	120	250	0.047
HGM V5	0.38	85	5500	0.127
HGM H6	0.60	60	18,000	0.200

2.4. Broth temperature and the growth rate

The growth rate in the photobioreactor using the HGM composite as construction material was evaluated in two steps. First, the results from transmittance and thermal conductivity of the composite were used as input parameters in the model presented by B  chet et al. (2010) adapted for a flat plate photobioreactor to predict the broth temperature (Eq. (2)). Then the results of the broth temperature and the irradiation data were used as input parameters in the model presented by Bernard and R  mond (2012) to finally estimate the growth rate in an outdoor production plant (Eqs. (3)–(7)).

The equation that describes the heating balance in a photobioreactor yields (Bechet et al., 2010):

$$\rho_w V_r C_{pw} dT_r/dt = Q_A + Q_B + Q_C + Q_D + Q_E + Q_F + Q_G + Q_H + Q_J + Q_K + Q_L \quad (2)$$

where T_r is the reactor broth temperature (K); ρ_w and C_{pw} are the density (kg/m³) and the specific heat capacity (J/kg/K) of water, respectively; V_r is the volume of the broth or reactor working volume (m³); Q_A is the rate of heat transfer due the radiation from the reactor itself (W); Q_B is the rate of heat transfer due to direct solar radiation (W); Q_C is the rate of heat transfer due to diffuse solar radiation (W); Q_D is the rate of heat transfer due to solar radiation reflected from the ground (W); Q_E is the rate of heat transfer due to radiation from the air surrounding the reactor (W); Q_F is the rate of heat transfer due to air radiation reflected from the ground (W); Q_G is the rate of heat transfer due to radiation from the ground (W); Q_H is the convective flux (W); Q_J is the rate of heat transfer due to the evaporation flux inside the reactor (W); Q_K is the rate of heat transfer from the air bubbles to the broth (W); and Q_L is the conductive flux with the ground surface at the base surface of the reactor (W). In this balance, the heat capacity of the reactor wall (in J/K) was considered negligible when compared to the heat capacity of the liquid phase.

The light intensity and temperature relationships based on specific growth rate (μ) are expressed as (Bernard and R  mond, 2012):

$$\mu = \mu_{opt}(I) \cdot \varphi(T) \quad (3)$$

$$\mu_{opt}(I) = \mu_m \cdot I/[I + \mu_m/\alpha \cdot (I/I_{opt} - 1)^2] \quad (4)$$

$$\varphi(T) = (T - T_{max}) \cdot (T - T_{min})^2 / [(T_{opt} - T_{min}) \cdot (f(T) - g(T))] \quad (5)$$

$$f(T) = (T_{opt} - T_{min}) \cdot (T - T_{opt}) \quad (6)$$

$$g(T) = (T_{opt} - T_{max}) \cdot (T_{opt} + T_{min} - 2 \cdot T) \quad (7)$$

where μ (day⁻¹) is the specific growth rate; $\varphi(T)$ is the cardinal temperature model with an inflexion term representing the influence of temperature; α is the initial slope of the light response curve; I_{opt} ($\mu\text{E}/\text{m}^2 \text{ s}$) is the irradiance for which growth is maximum (with respect to light); μ_{opt} (day⁻¹) is the maximum growth rate that occurs at temperature T_{opt} ($^\circ\text{C}$); μ_m (day⁻¹) is the maximum growth rate for optimal irradiance and temperature T_{opt} ($^\circ\text{C}$); T_{min} ($^\circ\text{C}$) is the temperature below which growth is assumed to be zero; T_{max} ($^\circ\text{C}$) is the temperature above which there is no growth; T ($^\circ\text{C}$) is the broth temperature; and, I ($\mu\text{E}/\text{m}^2 \text{ s}$) is the light intensity that reaches the broth.

The input parameter and the constant variables used for the broth temperature simulation are presented in Table 2. The environmental conditions tested in the original model for validation, such as air temperature, wind speed and sun irradiation remained unchanged.

3. Results and discussion

3.1. Tensile test and flexure test

The results of the tensile and flexure tests show that the addition of HGM to the polymers does not increase the resistance of the composite in terms of elongation at break (%) (Table 3). However, in the Comp. C1, V5 and H6 there is a slight reduction in the maximum tension at break compared to the PR matrix. This is because the HGM is made of borosilicate filled with air which may lead to an increase in the fragility of the material. Yet these results do not indicate that the material with the addition of HGM of the wall of the flat plate photobioreactor should be thicker in order to support the hydraulic pressure of the broth.

Among the three different types of HGM tested the increase in the H6 content leads to higher tension and flexion strength supported by the composite compared to C1 and V5 at break. Comp. C1 can support a lower flexure strength value before break that

Table 2
Parameters and constant variables tested for the broth temperature model.

Definition	Parameter	Unit	Values tested
Ground surface constants for concrete	Ground emissivity	-	0.95
	Ground heat capacity	J/kg K	750
	Ground density	kg/m ³	2.5×10^3
	Ground thickness	m	0.02
	Ground conductivity	W/mK	0.7
Water constants	Water density	kg/m	998
	Water heat capacity	J/kg K	4.18×10^3
	Water latent heat	J/kg	2.45×10^6
Air constants	Air emissivity	-	1.0
	Air conductivity	W/mK	2.63×10^{-2}
	Air heat capacity	J/kg K	2.0×10^3
	Air density	kg/m ³	1.20
	Atmospheric diffusion coefficient	-	0.20
Photobioreactor constants	Reactor emissivity	-	0.97
	Wall thickness	m	3.0×10^{-3}
	Air volume fraction as bubbles	m ³ /s	6.7×10^{-8}
	Reactor height	m	2.0
	Reactor width	m	1.0
	Reactor distance between plates	m	0.1
	Shading factor	-	No shading from 7 am to 7 pm
	Wall transmittance	-	See Fig. 1
	Wall conductivity	W/mK	See Table 4

Table 3

Thermal conductivity and mechanical test results of the polyester resin and composites.

Sample	HGM conc. (vol.%)	Max. tensile strength at break (MPa)	Elongation at break at max. tensile strength (%)	Max. flexure strength at break (MPa)	Elongation at break at max. flexure strength (%)
PR	0	3.86 ± 0.18	16.69 ± 0.14	2.05 ± 0.09	28.7 ± 3.3
Comp. C1	5.0	2.25 ± 0.70	2.48 ± 0.18	1.63 ± 0.07	5.5 ± 0.2
	2.5	2.53 ± 0.49	3.40 ± 0.19	1.50 ± 0.12	6.6 ± 0.5
	1.3	2.86 ± 0.55	2.94 ± 0.38	1.64 ± 0.08	8.8 ± 0.9
	0.6	3.47 ± 0.36	3.39 ± 0.18	1.69 ± 0.14	17.8 ± 3.3
Comp. V5	5.0	2.53 ± 0.51	3.05 ± 0.19	1.59 ± 0.04	9.99 ± 1.4
	2.5	2.69 ± 0.47	4.95 ± 0.14	1.46 ± 0.07	8.2 ± 0.4
	1.3	3.21 ± 0.70	4.81 ± 0.19	1.65 ± 0.12	15.2 ± 0.8
	0.6	3.42 ± 0.51	6.94 ± 0.17	1.81 ± 0.17	22.5 ± 0.8
Comp. H6	5.0	2.74 ± 0.39	3.65 ± 0.19	1.85 ± 0.06	9.45 ± 0.90
	2.5	3.15 ± 0.42	3.80 ± 0.11	1.92 ± 0.08	13.41 ± 1.2
	1.3	3.41 ± 0.54	4.28 ± 0.16	1.89 ± 0.08	16.6 ± 1.9
	0.6	3.50 ± 0.41	4.10 ± 0.29	1.91 ± 0.05	22.5 ± 1.4

could be caused by the size of the sphere and the low crushing strength of the microsphere (Table 1). The addition of HGM H6 leads to higher values for tension and flexure strength which might be due to the concentration of the microspheres in terms of number in the composite and the crushing strength. This indicates that Comp. H6 has more physical resistance and is more suitable for photobioreactor manufacturing processes.

3.2. Transmittance

Fig. 1 presents the results for transmittance measured at UV–vis to near-infrared wavelength. The transmittance of the control with pure polyester resin and catalyst is 99.6% with a standard deviation of less than 3%. The three types of HGM are white and they are not transparent due to their small size. The concentration tested in the PR HGM composites is based on the total volume of the matrix, thus, Comp. C1 has fewer microspheres in terms of number than Com. V5 and H6, respectively. Because of the lower number of

microspheres in Comp. C1, the results show a higher transmittance for all four different concentrations than the other two composites (Fig. 1). The presence and the distribution of the microspheres in the composite affect the transmittance results by differentiating the performance of the tested samples. This may explain the results from Comp. H6 with 5.0 and 2.5 vol.% are very similar (Fig. 1c).

3.3. Thermal conductivity

According to the results presented in Table 4 the thermal conductivity of the different composites are similar. There is a slight variation in the composites with the concentration of 5 vol.% of HGM, however, this does not have a significant impact on the heat transfer from the photobioreactor wall to the broth. Higher concentrations of HGM may lead to lower rates of thermal conductivity. On the other hand, a higher concentration of HGM will reduce even more the transmittance of the photobioreactor wall and may affect biomass production negatively.

The reduction in the thermal conductivity comparing the PR and Comp. C1, V5 and H6 ranges from 5% to 9% which represents a reduction of 1–2 °C in the broth temperature. Liang and Li (2007) observed a decrease from 25% in the HGM composite thermal conductivity with 20 vol.% of HGM. The thermal conductivity at 25 °C of the HPDE control tested by Patankar and Kranov (2010) reduced from 0.52 W/mK to 0.37 W/mK with the addition of 30 wt.% HGM.

As expected, because of the lower thermal conductivity of HGM, the resultant HGM composites have significantly lower thermal conductivity and this thermal conductivity decreases with the increase in HGM content in the composite (Table 4). The performance of three HGM tested were similar in terms of thermal conductivity.

3.4. Broth temperature and the growth rate

Fig. 2a, c, e and g shows the results for the broth temperature using polyester resin and HGM composite input data in the photobioreactor constants in the mechanistic model. The addition of

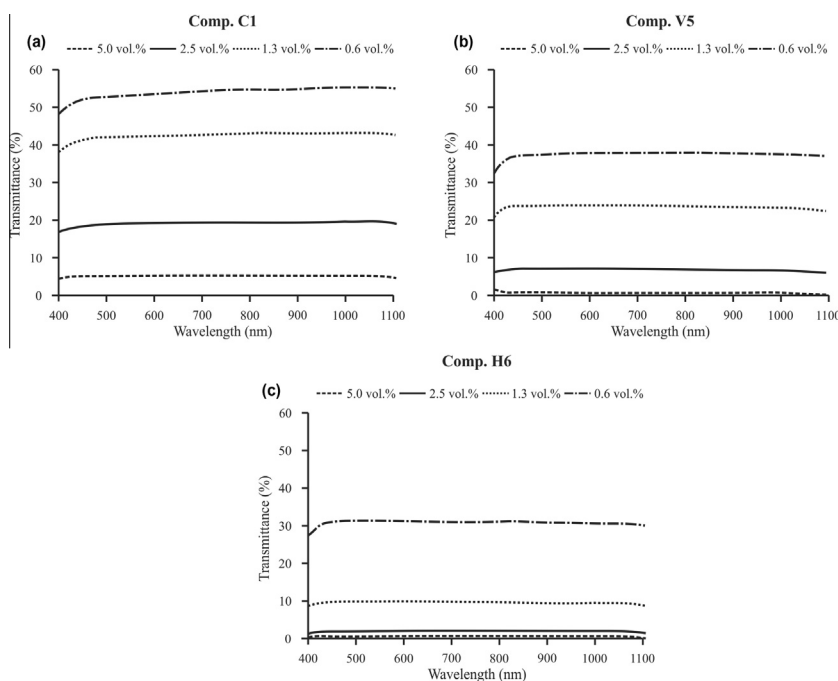


Fig. 1. Transmittance results of the PR HGM composites.

Table 4
Thermal conductivity of composites and the specific growth rate. Simulated results from photobioreactors made with HGM composites.

Sample	HGM conc. (vol.%)	Thermal conductivity (W/mK)	Simulated specific growth rate (day^{-1})
PR	0	200	0.89
Comp. C1	5.0	182.05	0.72
	2.5	185.40	1.07
	1.3	188.31	1.22
	0.6	190.73	1.22
Comp. V5	5.0	182.05	0.23
	2.5	185.49	0.80
	1.3	188.31	1.12
	0.6	190.73	1.21
Comp. H6	5.0	182.05	0.21
	2.5	185.49	0.46
	1.3	188.31	0.89
	0.6	190.73	1.18

5 vol.% of HGM led to a 15 °C reduction or a 33% drop in the broth temperature in the photobioreactor for three composites. However, using a concentration of 0.6 vol.% the average reduction is 5 °C for Comp. C1, 7 °C for Comp. V5 and 9 °C for Comp. H6.

In general the HGM composites behave in a similar way. The average difference of broth temperature is 2 °C for each increase of 100% tested in the microsphere content in the photobioreactor wall. The average broth temperature in Fig. 2 is below 35 °C for the three type of HGM. This is the optimal temperature for most microalgae culture. The lowest values for broth temperature were observed in the Comp. H6 which also has the lowest transmittance values. This indicates that the effect on the broth temperature is influenced mainly by transmittance values of the photobioreactor wall and that the thermal conductivity has a less significant effect.

The results from the microalgal growth simulation are presented in Fig 2b, d, f and h using the parameters values from *Chlorella pyrenoidosa* in the model developed by Bernard and Rémond

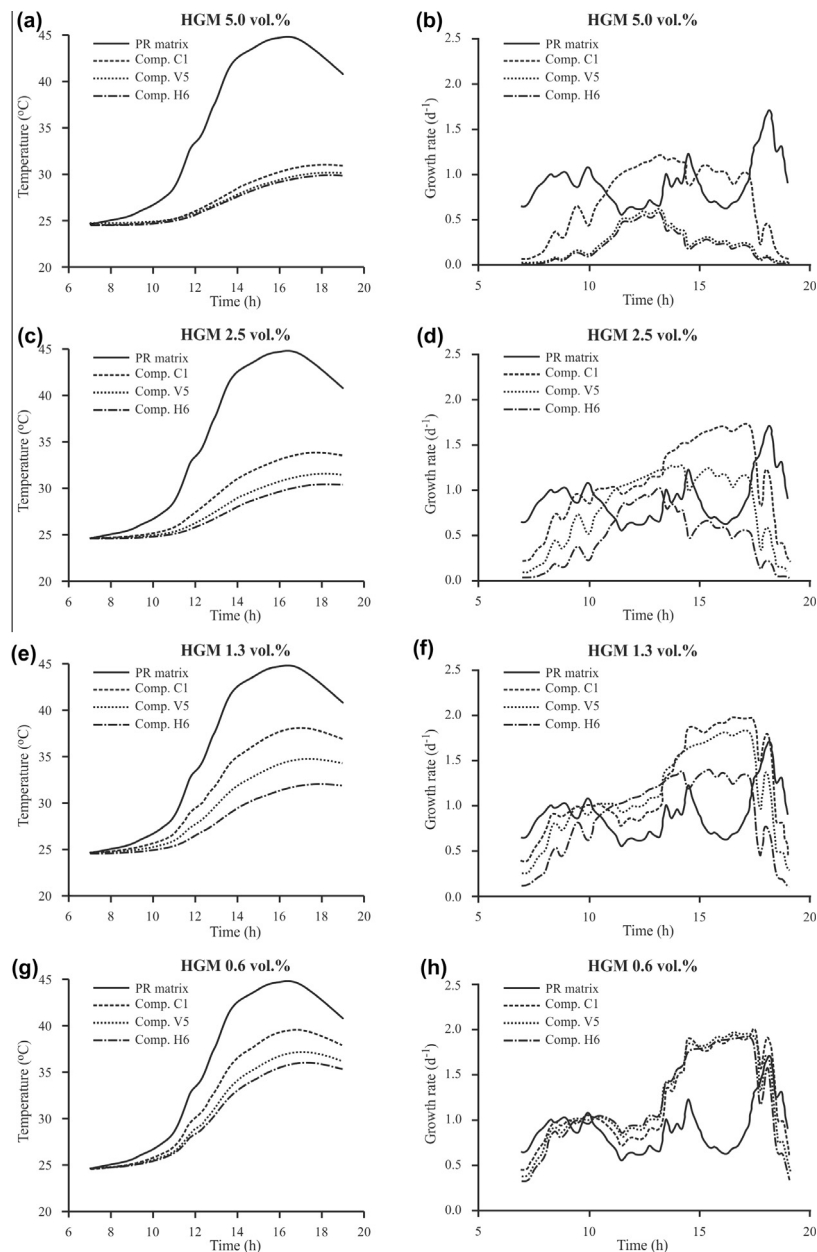


Fig. 2. The simulation results for the broth temperature (a, c, e and g) and specific growth rate (b, d, f and h).

(2012). The input data for irradiance used in this simulation are the same data used in the mechanistic model for the broth temperature. However, a reduction in the irradiance values were applied according to the transmittance values presented in Fig. 1 for each composite.

The use of the composites with addition of 5 vol.% HGM to construct photobioreactors have no positive impact on the specific growth rate. A small increase in the growth rate for 2.5 vol.% HGM is observed for Comp C1 (Table 4). Both have transmittance values of around 20%, however, only Comp C1 has a positive average growth rate (Table 4). The results from the effect of transmittance in the composites is clearer in Fig. 2d and f where the amplitude between the curves for the growth rate is higher. There is a slight difference in the case of 0.6 vol.% HGM (Fig. 2h) due to the similarity in the thermal conductivity and transmittance of the samples.

The irradiance data in the mechanistic model at 7 AM starts at $50 \mu\text{E}/\text{m}^2 \text{ s}$, at 10 AM it is around $1400 \mu\text{E}/\text{m}^2 \text{ s}$, it reaches a peak at 12 AM with $4400 \mu\text{E}/\text{m}^2 \text{ s}$ and then falls during the rest of the day. The reduction in the specific growth rate for the photobioreactor made of polyester resin can be observed from 12 AM to 4 PM. During this time the broth temperature also reaches the maximum value (Fig. 2). However, the addition of HGM in the photobioreactor wall shows that this can be minimized and the productivity increased in the cultivation system.

The density of the cells in the culture medium is not considered in the mechanistic model and the reactor emissivity considered is 0.97 (Table 3). On the first day of cultivation when the biomass density is lower, a reduction in temperature is expected, as presented in Fig. 2. However, the reduction in broth temperature can be even greater for a high biomass concentration due to decrease of the emissivity. The balance between emissivity and photobioreactor wall transmittance could be evaluated to reduce the HGM content in the composites even more.

The average reduction in the thermal conductivity of the photobioreactor wall is 8% and the reduction in transmittance ranges from 95% to 45% according to the HGM content. The use of HGM of a bigger size and lower density in the composite to construct photobioreactors seem to produce better results in terms of increase in growth rate. However, the size and density of the microsphere seem to have no influence in the composites with 0.6 vol.% HGM as observed in Fig. 2h.

In this study the best balance between thermal insulation and transmittance which leads to a higher biomass concentration is observed for Comp. C1 with 1.3 or 0.6 vol.%. However, the manufacturing process of a photobioreactor made from a HGM composite may require the use of more resistant HGM for extrusion and thermoforming processes. In this case Comp. V5 or H6 at 0.6 vol.% are the best option.

The external cooling systems such as water-spray on the reactor or internal heat exchangers to control broth temperature are associated to high amount of energy consumption. Using these temperature control system in a production unit of biodiesel from microalgae the total energy required is higher than the energy produced (Ras et al., 2013). In addition some solution as water-spray on the photobioreactor surface require controlled systems to turn on the spraying when the broth temperature is higher than the optimal condition.

HGM is an inert material and can be used as additive to any industrial plastic polymerization process. According to Norsker et al. (2011) the costs to a flat plate photobioreactors plant made of polyethylene sheet is € 9.76 per kg of DW biomass for 1–100 ha plant scale. The average cost of HGM is € 9.0 per kg (based on data from 3 M, Brazil). The additional cost for a polyethylene ($\rho = 0.94 \text{ g}/\text{cm}^3$) photobioreactor is € 0.09 per unit using 0.6 vol.% HGM within the dimensions described in Table 2. As observed,

the average broth temperature reduction using 0.6 vol.% HGM C1 is 5°C (Fig. 1a). The energy required only for a heat transfer process to reduce 5°C in the broth temperature is approximately 1.2 kW h. The average cost for electricity for industrial users in Europe is € 0.15 per kW h. Other additional costs as pumping, pipe lines and maintenance have to be considered for cooling systems by internal heat transfer.

Another alternative for broth temperature control are shade net structures that reduce the incident light by 30–80%. The average costs for this solution is € 2.7 per m^2 including installation (based on data from Grupo Nortène, Brazil), however, the maintenance and extra labor costs also need to be included. In terms of costs the addition of HGM in the photobioreactor construction material is an low cost alternative to control broth temperature, does not have a significant effect on the growth rate and there are no additional costs to be considered as equipment, energy, maintenance and labor to control the broth temperature.

4. Conclusion

The three different HGM concentrations were tested in polyester resin matrix for a composite material to be used as construction material for photobioreactors in order to reduce the broth temperature and increase growth rates in the microalgae culture. The broth temperature in the photobioreactor made of a HGM composite material has a significant impact on the use of a heat exchanger. It reduces the amount of energy required for broth temperature control significantly. Therefore, the use of HGM composites in the manufacture of reactors is a way to reduce costs and make large scale outdoor microalgae production feasible.

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