

Pyrolysis of algal biomass obtained from high-rate algae ponds applied to wastewater treatment

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This work presents the results of the pyrolysis of algal biomass obtained from high-rate algae ponds treating sewage. The two high-rate algae ponds (HRAP) were built and operated at the São João Navegantes Wastewater Treatment Plant. The HRAP A was fed with raw sewage while the HRAP B was fed with effluent from an upflow anaerobic sludge blanket (UASB) reactor. The HRAP B provided higher productivity, presenting total solids concentration of 487.3 mg/l and chlorophyll a of 7735 mg/l. The algal productivity in the average depth was measured at 41.8 g·m⁻² day⁻¹ in pond A and at 47.1 g·m⁻² day⁻¹ in pond B. Algae obtained from the HRAP B were separated by the process of coagulation/flocculation and sedimentation. In the presence of alum, a separation efficiency in the range of 97% solid removal was obtained. After centrifugation the biomass was dried and comminuted. The biofuel production experiments were conducted via pyrolysis in a tubular quartz glass reactor which was inserted in a furnace for external heating. The tests were carried out in an inert nitrogen atmosphere at a flow rate of 60 ml/min. The system was operated at 400, 500, and 600°C in order to determine the influence of temperature on the obtained fractional yields. The studies showed that the pyrolysis product yield was influenced by temperature, with a maximum liquid phase (bio-oil and water) production rate of 44% at 500°C, 45% for char and around 11% for gas.

Keywords: high-rate algae ponds, pyrolysis, biofuels, wastewater treatment, bioremediation

Introduction

Biomass is considered worldwide as an important source of renewable energy, including electricity, automobile fuel, and as a source of heat for industrial equipment.

Cultures commonly used for energy production are sugarcane, corn, beans, beets, and many others. There are two main factors that define when a culture is appropriate for this process: good dry matter yield per unit of land (dry ton/ha), low area requirement for cultivation, and low costs of energy production from biomass (Dermibas et al., 2009).

However, some research has condemned the use of biofuels, associating its production with possible high food prices. Algae, among the aquatic biomass feedstocks, are considered one of the most promising sources of biofuels due to their unique characteristics. They can accumulate lipids that can be converted into biofuels, present fast proliferation, have the ability to sequester CO₂ from the atmosphere for growth and do not require agricultural land or freshwater for growth or higher water consumption, and also the whole plant matter can be used in converting biofuels processes

(Dismukes et al., 2008; Brennan and Owende, 2010; Jena and Das, 2011; Pate et al., 2011; Yanik et al., 2013; Zhou et al., 2014; Hognon et al., 2015).

Wastewater treatment associated with algae cultivation can offer an alternative way for sustainable renewable biofuels, since the large amount of freshwater needed for algae cultivation can be saved, becoming an environmentally friendly process (Zhou et al., 2014).

In a sewage treatment system, high-rate ponds are characterized by having high algal biomass generation which is an undesirable byproduct for the environment. Its presence in water bodies decreases water quality.

High-rate algae ponds are raceway-type ponds, in which water, algae, and nutrients are continually mixed. A paddle wheel generates a mean horizontal water velocity of approximately 0.15–0.3 m/s. This movement is necessary to avoid sedimentation and stratification. The maximum biomass production (mostly algae) is achieved through better use of lighting per volume. This is ensured by the low depth of the ponds and the constant movement of biomass through mechanical mixing (Nascimento, 2001; Chisti, 2007).

Usually the algal biomass productivity is determined by the measurement of solids found in the ponds.

The algal biomass production costs are mainly covered by the costs of treatment when using wastewater high-rate ponds resulting in lower environmental impacts in terms of water, energy, and fertilizer needs.

The biomass in high-rate algae ponds assimilates the nutrients needed for its growth and becomes responsible for the removal of nutrients from wastewater. This has the advantage of controlling pollution of water resources which contributes to the sustainable use of this technology on an industrial scale (Park et al., 2011; Passos et al., 2013).

The biomass separation process requires an increase in algal suspension concentration typically from 0.02 to 0.06% total suspended solids (TSS) to approximately 2 to 7% solids, which may be higher depending on the target process objective (Uduman et al., 2010).

The algal cells have reduced size, sometimes <30 μm and their density is similar to water with a low sedimentation rate, so to be successful in separation, it is necessary to aggregate the cells. Generally, the process comprises of two steps: the first involving destabilization of algal cells using coagulation followed by sedimentation or flotation. In the second step of the process, it is necessary to increase the biomass content, which is often done by filtration, centrifugation, or thermal processes (Molina Grima et al., 2003; Granados et al., 2012; Cai et al., 2013; Udom et al., 2013).

The algae cell has a negative surface charge, which prevents aggregation. This charge may be reduced or neutralized by the addition of flocculants or multivalent cations, such as cationic polymers that change the zeta potential, which is a measure of particle stability, reducing the repulsive forces. So the action of the attractive Van der Waals forces allows algae agglutination (Wessler et al., 2003; Granados et al., 2012). Salts used for this purpose should be non-toxic, low cost, and have high effectiveness at low concentrations (Molina Grima et al., 2003).

Another advantage of using coagulation/flocculation process is nutrient removal. The presence of nutrients in wastewater,

particularly nitrogen and phosphorus, is a serious environmental problem and is receiving increasing attention. Nitrogen in the form of ammonia can be volatilized and cause air pollution. Phosphorus can permeate into the soil and cause damage to the underground water (Chen et al., 2012). When there are excessive levels of nutrients in the wastewater, they cause eutrophication of water sources, possibly damaging the ecosystem (Cai et al., 2013).

The algal biomass, after thickening, may reach 5–15% solid content, and, being perishable, it must be processed as soon as possible. Essential processes such as thickening and drying usually involve high operational costs. Thus, these steps are considered determining factors regarding the economical feasibility analysis of the overall process (Brennan and Owende, 2010; Uduman et al., 2010). The methods commonly used for thickening biomass are centrifugation and filtration followed by different drying techniques, such as natural, oven, spray, and fluidized bed drying.

There are three basic components in algae biomass: proteins, carbohydrates, and lipids. These oils can then be extracted and converted in to biofuels (Um and Kim, 2009).

The pyrolysis process appears to be an excellent alternative for energy conversion, it presents the advantage of using different sources of organic matter, not being limited by the lipid content, as with biodiesel production processes. The pyrolysis process is based on decomposition of organic compounds present in the total biomass under a controlled environment in the absence of oxygen and atmospheric pressure, resulting in different phases: liquid (bio-oil), gas, and solid (char). It is an endothermic reaction that occurs at a temperature of 300–700°C depending on the characteristics of the material to be pyrolyzed (Martini, 2009; Hognon et al., 2015).

Biomass pyrolysis is considered a renewable process, because biomass is turned in several gases when pyrolyzed. Carbon dioxide, one of the gases formed, is absorbed by the algae for its growth, making the process self-sustainable with no serious contribution to greenhouse effect. The relative yield of each phase generated in the process depends on operating parameters (temperature, heating rate, residence time, and flow rate of inert gas), properties of the biomass (the particle size as well as its moisture), and type of pyrolysis used (slow, fast, or flash pyrolysis) (Balat et al., 2009; Martini, 2009; Akhtar and Amin, 2012; Yanik et al., 2013; Hognon et al., 2015).

In order to obtain high yields of aqueous products, fast pyrolysis is normally used, which is characterized by higher heating rates (1000°C/min) and lower residence times of volatiles (10–20 s). In order to favor solid char formation, slow pyrolysis process with lower heating rates (5–80°C/min) and longer residence times (5–30 min) must be used (Van de Velden et al., 2010; Jena and Das, 2011; Yanik et al., 2013).

The bio-oil generated by biomass pyrolysis is generally cleaner than that from fossil fuels, due to its lower nitrogen and sulfur content. The biomass vaporizes, passes through a process of cracking and condensation, producing a dark brown liquid, consisting of a complex mixture of many different hydrocarbons. This process is most successful in fluidized bed reactors due to high heating rates, rapid devolatilization and easy control (Doshi et al., 2005; Martini, 2009).

Materials and Methods

Biomass Production

Two high-rate algae ponds were constructed in the IPH/UFRGS experimental wastewater treatment unit, at São João Navegantes Wastewater Treatment Plant. This plant is responsible for handling the sewage of the north area of Porto Alegre/RS.

The ponds were operated in closed circuit with the following dimensions: overall height: 0.9 m, length of the straight sections: 30 m, width: 5 m (at the upper edge of the slope) and surface area 320 m², as can be seen in **Figure 1**.

The high-rate algae ponds were operated under two feeding conditions: pond A was fed with raw sewage after pretreatment (screening and grit removal) and pond B was fed with effluent from an upflow anaerobic sludge blanket (UASB) reactor. In order to maximize the process of biomass production, the operating parameters of the ponds were useful depth (Hu): 0.3 m, longitudinal flow speed: 0.3 m/s, and hydraulic detention time (HDT): 3 days.

The pond samples were collected in 20 l plastic containers, directly from the body of the ponds, to provide enough biomass for the pyrolysis experiments.

In order to determine algae biomass productivity, total solids, turbidity, and chlorophyll a were measured weekly.

All experiments to determine these parameters were carried out according to Standard Methods for the Examination of Water and Wastewater [American Public Health Association (APHA) and Awwa (2005)].

Algae Separation and Thickening

Experiments of coagulation/flocculation were performed using the effluent from pond B, which showed better performance in terms of algal biomass production.

The equipment used was VELP Jar Test model F.6/S, composed of 6 jars of 2000 ml each, with agitation and controlled independently.

To evaluate the separation process and the removal of nutrients two coagulants were used, Aluminum Sulfate and Ferric Chloride and two flocculants, Sulfloc 1001 and Tanfloc SL. Their concentration ranges are shown in **Table 1**.

After the separation and removal of all the supernatant from the jars, the algae sludge was submitted to centrifugation for 20 min at 2500 rpm to obtain a sample of about 15–20% of dried solids. After

centrifugation, the biomass was dried at 105°C. Finally, the dried algae were ground in a mortar and stored separately according to the reagent used in the separation process.

Nutrient Removal

Experiments were performed to determine the concentration of nitrogen and phosphorus in effluent ponds before and after coagulation/flocculation. Thus, it was possible to determine the effect of algae upon the separation in the removal of nutrients.

Biomass Pyrolysis

The experiments obtaining biofuel via biomass pyrolysis were performed in a tubular quartz reactor, with the dimensions described in **Figure 2**.

The experiments were run in batches, to allow solid char removal. The process flow used in this work, presented in **Figure 3**, was based on Zhang et al. (2011).

In the process, the inert atmosphere was generated by nitrogen gas (1) and the heating process was provided by an external furnace (2). The condensation was performed in (3), where two condensers in series were immersed in an ice bath. The exit of non-condensable gases was in (4).

The pyrolysis reactor was fed manually with 7 g of dried and ground biomass obtained from the previous step of the process. The biomass was inserted in the reactor using an aluminum foil capsule. After it has been charged, the reactor was closed and the inert atmosphere was provided by a 0.06 l/min nitrogen gas flow.

The pyrolysis runs were started by placing the reactor in a programmable tubular furnace, with a heating rate of 20°C/min. All the runs were made in two steps: heating the sample and an isothermal reaction step, maintaining the desired temperature (400, 500, and 600°C) for 60 min.

The vapors generated passed through two condensers in series, immersed in ice baths maintained at a temperature of about 0°C. At the end of each experiment, the aqueous phase generated by condensation was collected, combined, weighed, and stored. The non-condensable gases were measured by difference. Following the 60 min reaction time, the system was turned off and cooled to room temperature.

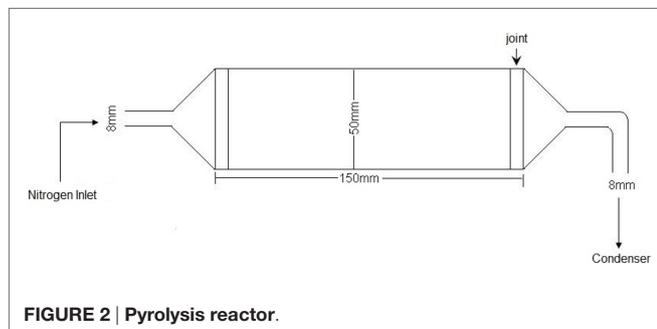
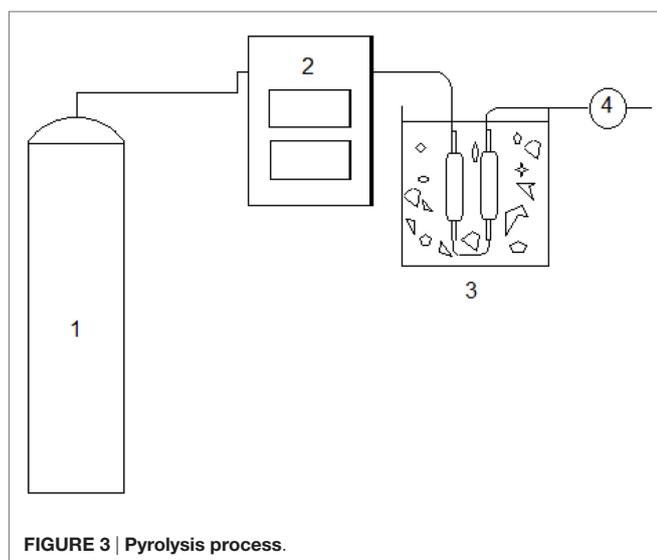
After reaching room temperature, the reactor was opened and the solid fraction (char) was collected, weighed, and stored. The



FIGURE 1 | High-rate algae ponds.

TABLE 1 | Concentration range used.

Product	FeCl ₃ 10%	Al ₂ (SO ₄) ₃ 10%	Sulfloc 20%	Tanfloc 10%
Concentration range (mg/l)	200–300	100–150	250–300	50–100

**FIGURE 2 | Pyrolysis reactor.****FIGURE 3 | Pyrolysis process.**

reactor final mass was also determined, in order to measure the losses by wall adhesion.

The pyrolysis evaluation was performed through yields measurement. Each fraction was determined from the ratio of the weight of respective fraction to initial weight of biomass, expressed as percentage yield, according to Eq. (1).

$$\text{Yield (\%)} = \frac{\text{Fraction mass obtained after pyrolysis}}{\text{Initial algal biomass}} \times 100\% \quad (1)$$

Results and Discussions

Biomass Production

The results of solids, turbidity, chlorophyll a and productivity are shown in **Table 2**, comparing the performance of both biomass production ponds.

In the experiments, we considered the concentration of solids present in effluents and turbidity caused only by the presence of algae. From **Table 2**, it can be noted that pond B in all evaluation parameters showed higher values than those obtained from pond A. Such behavior is explained by the fact that effluent from UASB

TABLE 2 | High-rate ponds performance.

Analysis	Pond A	SD	Pond B	SD
Total solids (mg/l)	433.2	59.2	487.3	56.1
Turbidity (NTU)	41.9	8.9	63.3	13.4
Chlorophyll a (mg/l)	2338	NA	7735	NA
Productivity (g·m ⁻² day ⁻¹)	41.8	NA	47.1	NA

NA, not applicable.

TABLE 3 | Algae biomass productivity.

Authors	System	Biomass productivity (g·m ⁻² day ⁻¹)
Nascimento (2001)	HRAP	21.8
Riaño et al. (2012)	Photobioreactor	1.54
Sturm and Lamer (2011)	Open ponds	12
Terigar and Theegala (2014)	Open tanks	43.4

TABLE 4 | Crops productivity [adapted from Trzeciak et al. (2008)].

Crops	Harvest (month/year)	Biomass productivity (g·m ⁻² day ⁻¹)
Cotton	3	0.38
Peanut	3	0.55
Canola	3	0.60
Sunflowers	3	0.55
Dendê (<i>Elaeis guineensis</i>)	12	6.84
Mamona (<i>Ricinus communis</i> L.)	3	0.41

reactor provided low solid concentration, which facilitated higher solar irradiation in the body of the pond, an essential factor for biomass growth. Thus, the effluent selected for tests of separation, thickening, and the tests for obtaining biofuels was collected from the pond B. **Table 3** shows a comparison among biomass productivity obtained in this work and others presented in the literature.

Table 4 shows the comparison between the productivity of crops commonly used in the biofuels production.

As we can see from both tables, high-rate algae ponds can be a competitive source of biomass, with higher productivities and without need of arable land and fresh water. This system high-rate algae pond (HRAP) presents no seasonality and the biomass can be harvested all year, without competition with food crops.

Algae Separation, Thickening, and Nutrient Removal

The results of algae separation are shown in **Table 5**, based on separation efficiency related to the chemical dosage used. This table also shows the evaluation of nutrient removal for each product.

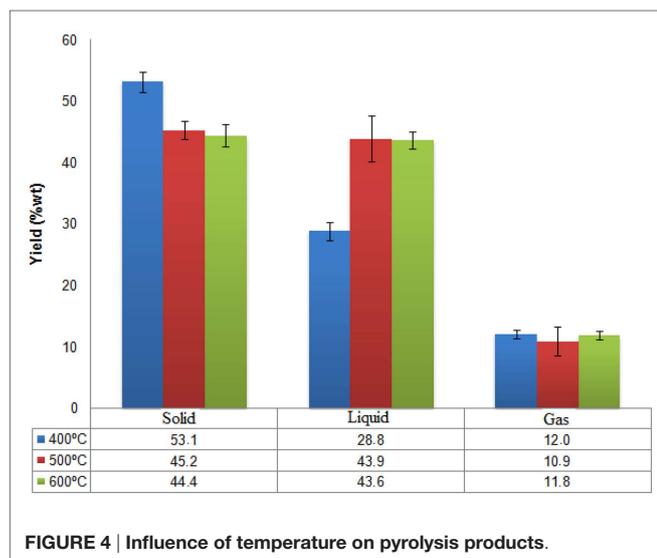
Thus, according to the results shown in **Table 5**, the biomass separated with aluminum sulfate, which was selected as the most convenient chemical due to lower dosage requirement, showed better separation and nutrient removal. The biomass was dried and crushed to be used in the pyrolysis experiments. The efficiency of N and P removal were similar when using Sulfloc 20%, but the dosage required was higher than with sulfate.

Biomass Pyrolysis

The influence of temperature (400, 500, and 600°C) on pyrolysis results are shown in **Figure 4**. According to the results, the

TABLE 5 | Removal obtained and dosage used.

Product	Ferric chloride 10%	Al sulfate 10%	Sulfloc 20%	Tanfloc 10%
Maximum separation (%)	88.4	97.9	94.5	97.5
Concentration (mg/l)	300	150	290	100
P Removal (%)	100	100	100	37.9
N Removal (%)	–	5.5	5.5	–

**FIGURE 4 | Influence of temperature on pyrolysis products.**

temperature of 400°C favors solid phase formation, with an average yield of 53.1%. The aqueous and gaseous phases obtained average yields of 28.8 and 12%, respectively. At 500°C, the yields for solid and aqueous phases were similar, however aqueous phase formation was slightly higher, composed of bio-oil and water. The average yield was 43.9 for solid phase 45.2 for the aqueous phase and 10.9% in the gas phase. At 600°C we can see similar yields between solid and liquid formation, 44.4 and 43.6%, respectively. The average for gas formation at this temperature was 11.8%.

The liquid phase, comprising of bio-oil and water, has a reddish brown color, with a strong and distinctive smoky smell, which

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confirms the information in the literature about products obtained in the pyrolysis (Jena and Das, 2011; Yanik et al., 2013).

As described in the literature, temperature plays an important role on the yield of the fractions obtained in the pyrolysis process. Studies show that temperatures between 450 and 550°C maximize the yield of bio-oil and, at very high temperatures, secondary reactions of the volatiles may occur, thus decreasing the yield of the liquid phase, which can be seen in **Figure 4**; at 600°C, a small decrease in the aqueous phase yield was observed (Yanik et al., 2013).

For related data, we use an ANCOVA analysis (Analysis of Covariance) with a fixed factor (oven temperature) and a covariate (initial mass of algae) to identify differences in the char mass production. Five replicates were performed for each factor and the software used was SPSS version 18.

The data do not present heteroscedasticity, using the Levene test (p -value of 0.235), the tested factor was significant at a p -value of 0.01. So we went to the *post hoc* analysis, which showed a significant difference between the means of groups, the 400°C group is different from other groups and the 500°C and 600°C are not statistically different from each other.

Conclusion

In this work, the association of wastewater treatment and biofuel production through pyrolysis of algal biomass obtained in high-rate algae ponds was studied. The algal productivity, at the average depth was measured as 41.8 g m⁻² day⁻¹ for pond A and as 47.1 g m⁻² day⁻¹ for pond B. The algae were pyrolyzed in a tubular furnace system with external heating at different temperatures. Studies have shown that the pyrolysis process is efficient and the fractions yields are greatly influenced by temperature. Operating under mild conditions, it was possible to obtain maximum yields of 45% at 500°C for aqueous phase (bio-oil and water), 44% for char, and about 11% for gas. As we can see, through this process, it is possible to offer a promising alternative for environmental pollution control with potential economic return.

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