PILOT SCALE HARVESTING, SEPARATION AND DRYING OF MICROALGAE BIOMASS FROM COMPACT PHOTO-BIOREACTOR

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Abstract. *Biodiesel produced from microalgae lipids is gaining a substantial ground in the search for renewable energy sources. In order to optimize the operating conditions of a continuous process, several experiments were realized, both in laboratory and pilot scale. The microalgae cultivation can be conducted in a photo-bioreactor, a closed system which allows parameters control and necessarily involves the aquatic environment. Because of that, the use of separation unit operations is required. The process starts in a proposed compact photo-bioreactor, which consist of a chain of transparent tubes with 6 cm of diameter arranged in parallel where the cultivation media circulate with the help of a pump. This arragement offers a closed culture with less risk of contamination and maintains a minimum contact with the environment. The microalgae grow inside the pipes under incidence of ambient light. In this paper, harvesting, separation and drying were studied, as part of the processes of a sustainable energy plant under construction at UFPR, as shown in Fig. 1. To control the production in a photo-bioreactor in continuous system, it is necessary to monitor the concentration of microalgae growth in suspension. To measure the cell concentration in this equipment, an optic sensor has been developed. The microalgae biomass separation from the culture media is achieved by microalgae flocculation. Several cultivation situations have been tested with different NaOH concentrations, increasing the pH to 10. The system was kept under agitation during the addition by an air pump into the tank. Thereafter the system was maintained static. After a short time, it was observed that the microalgae coagulated and settled. The clarified part water was removed, remaining a concentrated microalgae suspension. Our results suggest that pH increase is a suitable methodology for microalgae separation from the growth suspension. The microalgae sedimentation time was recorded, which allowed the sizing of sedimentation tanks in pilot scale. The next process step, the filtration, was conducted with several filtering media and situations, followed by a incubator or spray drying. The flocculated material was then concentrated by centrifugation and submitted to spray dryer. The dry material (with 4.3 % of humidity) was collected in a vessel at the exit of the side cyclone, which was maintained opened. With this process we obtained, in the end, powdered "Nannochloropsis oculata" biomass, which was submitted to lipid extraction, and thereafter to biodiesel synthesis. The experimental results suggest that the proposed process is a suitable methodology for microalgae separation. The extaction of the oil form biomass bys direct pressing was also tested. These processes were compared and quantified, giving priority to product quality and minimum energy consumption.*

Keywords*: microalgae, biodiesel, unit operation, separation process, flocculation*

1. INTRODUCTION

The world primary energy consumption in 2008 was estimated in fossil fuels (88 %), hydroelectricity (6 %) and nuclear energy (5 %) (Brennan and Owende, 2010).

Greenhouse gases emissions are causing environmental problems and an alternative source that reduces yours emissions must be found. An alternative to the use of fossil fuels is the biodiesel, a renewable and sustainable fuel that reduces environmental impact in comparison to fossil diesel, since it is produced from vegetable or animal oil. Biodiesel production from vegetable oils is a great alternative widely available in the entire world (Meng *et al.*, 2008). Biodiesel can be made from any fat or vegetable oil, but the majority of biodiesel production uses soybean as feedstock, in which is also used as food.

Among many resources for this, microalgae have been found to be very promising (Chisti, 2007) due to their high concentration of lipids in your biomass, a good growth rate and resistance to concentration of salts that allowing the use of any type of water for the culture media. (Hsieh and Wu, 2009).

Microalgae are an attractive and interesting possibility as a feedstock for production of biofuels. In addition, microalgae are a source of biomass, which may have great biodiversity and variability in their biochemical composition, so it can be used in many applications like animal feed, aquiculture, cosmetics and etc. (Pulz and Gross, 2004; Spolaore *et al.*, 2006).

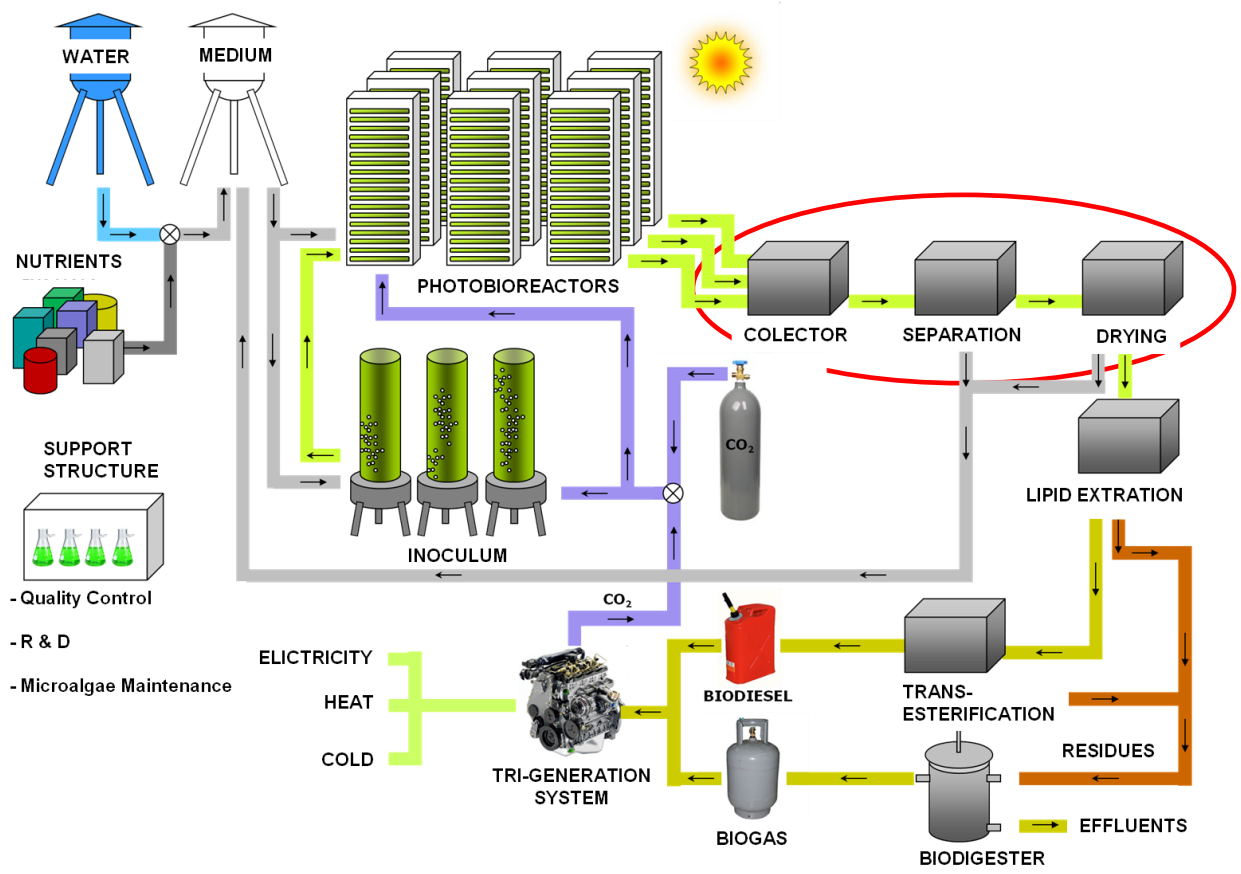

Figure 1. Overview of all processes in the sustainable energy plant under construction at UFPR, with harvesting, separation and drying showed inside a red ellipse.

Photobioreactors are equipments used to cultivated photosynthetic microorganisms such as microalgae (Harun et *al.*, 2010) and consist of transparent pipes that allow the entrance of light and accessories that inject air or carbon dioxide. The efficiency of photobioreactors depends of light incidence because the light energy absorption by algal cultures is a necessary step to improve the algal growth.

One of the most important problems in microalgae biotechnology is the separation of biomass from water and your purify process (Antolin *et al.*, 2002). There are several efforts that must be conducted to develop a process that is economically viable; also development of efficient and economical processes for removing and de-watering of biomass is another area of interest for research.

The Center for Self-Sustainable Energy Research & Development (*NPDEAS = Núcleo de Pesquisa e Desenvolvimento em Energia Autossustentável*) of the *Universidade Federal do Paraná* (*UFPR*) proposes the production of biodiesel from microalgae (Vargas, 2007), using compact photobioreactor to grow the microalgae (Fig. 2).

The production will be in a semi-continuous system through simultaneous addition of new culture media and removing old media.

A fundamental point in the biodiesel production by microalgae consists in choice of the methodologies for the separation and drying of the microalgae biomass and also in the oil extraction process. Methodologies that consume a lot of energy make the biodiesel production process commercially unfeasible. Besides, as the project is aimed at developing sustainable electric power plant, it has become necessary to minimize the energy at several stages of the Different methodologies such as conventional flocculation, electroflocculation, decantation, centrifugation or filtration can be used for the microalgae separation (Lee *et al.*, 2009). After the separation, drying of the material takes place through sun drying, liofilization, spray-drying or even competitive flow. The choice of the methodologies to used in the separation and drying will be based on the efficiency and the cost, which will give the most favorable selection. Separation techniques used in plant oils can be used for microalgae. The methods used include pressing and solvent

extraction. Each one of these processes requires energy consumption and different yields. However, new approaches in the extraction of microalgae lipids are necessary for the total cost of biodiesel production will become commercially competitive. In this way, it is planned to have new approaches in this work for the separation of microalgae lipids. These studies will be taken up after the start of the photobioreator operation and consequently after the production of biomass.

The aim of this paper is to evaluate different techniques for monitoring and processing a pilot-scale cultivation of microalgae for biodiesel production. All alternatives will be evaluated considering the least energy possible.

Figure 2. The NPDEAS compact photobioreactor in pilot scale.

2. METHODOLOGY

After the microalgae cultivation, for the effectively transform the biomass into useful products, a sequence of unit operations is required, who are being studied in lab scale and developed in pilot scale. The principal idea is that the process will be modeled and optimized with the objective to produce biodiesel using the minimum energy as possible. It is important to remember that these unit operations are part of a broader process and with multidisciplinary work, developed by NPDEAS, as can be seen in Fig. 1.

The several stages of this process for microalgae production will be presented separately, in the intent of making them easier to understand.

2.1. Cultivation

Usually, in studies and researches in several fields, the cultivation of the microalgae is made using batch techniques. The unit that performs these activities is the GIA (Grupo Integrado de Aquicultura e Estudos Ambientais = Integrated Group for Aquaculture and Environmental Studies) from the Universidade Federal do Paraná. It was used Nannochloropsis oculata microalgae cultivated in 3 tanks of 200 L, with constant aeration and under ambient temperatures and luminosity. The culture medium used in this experiment was Guillard f/2, inoculated in seawater with 0.5% salinity. The samples were collected after 10 days of cultivation.

For continuous cultivation in larger scale, the best option is compact photo-bioreactors. They consist in several transparent pipes where the culture media is continuously pumped. With light, nutrients and CO2 occurs a considerable increase in the quantity of microalgae biomass. With this purpose, a compact photo-bioreactor was built, with 9

columns of tubes, each of them with 53 horizontal "lines" (Fig. 2). The tubes are made of PVC with 6 cm diameter; they are arranged in the geographic position that allows absorbing the maximum quantity of light that is possible considering the location of the city of Curitiba.

2.2. Sensor

A problem to operation monitoring, is to determine the microalgae concentration, in other words, the number of cells in the aqueous suspension. For this, one of the methods that doesn't interfere in the system and can be used to determine the microalgae concentration is the measuring of the turbidity of the medium, that can be done through turbidimeters. For faster obtaining of this information, an optical sensor was developed and built, which can appropriately measure the quantity of microalgae inside the photo-bioreactor.

The circuit for determination of the optical density was mounted in the exterior of a transparent PVC tube, with nominal diameter of 2.54 cm. It consists of 6 infrared LEDs with 5 mm and the emitted light is captured by a LDR (*Light Dependent Resistor*) located on the opposite side of the tube. The schematic of the sensor can be seen in Fig. 3.

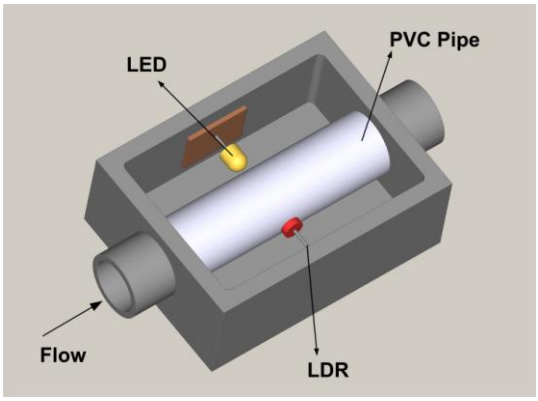

Figure 3. Sketch of the cell concentration sensor.

The experiments for the calibration of the sensor were made in triplicate and have consisted in the microscopic count of the number of cells in a Neubauer chamber. This count was related in the sensor in function of LDR electric resistance. After that, the microalgae suspension was put in the sensor and your resistance was measured with a multimeter.

2.3. Flocculation and Sedimentation

On the sequence of the process, after the growth of the microalgae, it is necessary to separate the biomass and the culture medium. Among the various options, one appears more practical and economical is the sedimentation of the biomass. This operation is slow, so it ought to be speeded up by flocculation. Here two possibilities were analyzed: the conventional flocculation (particle aggregation by pH changes) and the electroflocculation.

In the conventional process, a chemical solution of 1.0 mol/L of NaOH was added in the tanks being mixed by injection of air. This flocculants addition was monitored with the pH measure, realized by a digital pH meter.

A recent technique to facilitate the separation of the biomass is the electroflocculation that, besides "clean", does not require the addition of other substances. This method consists of applying an electric field in the suspension through electrodes, generating a movement of charged particles. The microalgae usually have a negative electrical charge and thus are attracted to the positive pole (anode) of the electrolytic cell, where the agglomeration (flocculation) occurs, thus accelerating the process of sedimentation. In this situation was used an aluminum electrode and a lead electrode, with variable distance between them (Fig. 4). Electroflocculation experiments were performed using two values of continuous electric voltage: 30 V and 15 V, the volume of suspension was 3 L and electrolysis occurred during 30 minutes. Two different cell concentrations were used: 8.1×10^{14} and 2.9×10^{15} cells/m^3. After this operation, the samples were filtered on a previously dried filter paper; the retained cake, along with the filter, was taken to a furnace with air circulation. After 24 hours at 120 °C the biomass was weighed.

2.4. Centrifugation and Drying

After being flocculated and decanted, biomass was centrifuged by 5 minutes at 3500 rpm. This operation, despite the energetic cost, greatly facilitates the next stage: drying.

For this was used a spray dryer located in the Pilot Plant of Chemical Engineering in UFPR. This equipment promotes a fast and efficient drying of the biomass. The flow of the suctioned liquid was adjusted to 50 g/min and the

temperatures were monitored in the operation and output. After, this dried material was subjected to the determination of humidity by weighing in a furnace.

Figure 4. Electroflocculation cell for the separation of microalgae from an aqueous suspension.

2.5. Pressing

In the processing of microalgae, there was the curiosity to know if a pressing wouldn't be enough to extract the lipids of microalgae; this could replace the various operations performed with this same goal.

Focusing on this, was realized a qualitative test with microalgae *Nannochloropsis oculata* and *Phaeodactylum tricornutum*. It was used a helical-type extruder press, commonly used for extraction of vegetable oils.

Despite great expectations, the experiment did not produce good results. The most likely reasons are presence of a kind of "shell" in these microalgae (which hinders the extraction of lipids) and the fact that this type of biomass formed a "cake" that clogged the strainer in the press output.

It is intended to make new pressing tests using microalgae which do not have this “shell”.

3. RESULTS AND DISCUSSIONS

3.1. Optical sensor

The calibration of the sensor showed a good adjustment (with correlation coefficient of 0.97), resulting in the following expression:

$$C = 8.16.R + 3.56$$

where C is the cell concentration ($.10^9$ cells.m^{-3}) and R the electrical resistance ($.10^6$ ohms).

This result is highly interesting, because it is a simple, practical and reliable way to know the value of a important variable to the process (the number of cells per volume of media). Surely this device, which was developed and built specifically for this study, will be very useful in further researches.

3.2. Conventional flocculation

The performed experiments allowed to conclude that, from a certain amount of NaOH added the pH remains almost constant at 10. For the calculation of the minimum amount of flocculant needed for the process, this analysis will enable a lower requirement of this substance, especially for larger scale experiments. Moreover, the flocculation increases the speed of the sedimentation. These facts will be better analyzed when the optimization of the process has been done.

3.3. Electroflocculation

The electroflocculation tests were performed as previously described. The recovery percentage was estimated by comparison with samples subjected to conventional flocculation. The results are shown in Tab. 1.

These are initial results, but it may be noted that even with high levels of recovery, the masses obtained by electroflocculation are smaller than those obtained by conventional flocculation. It can also be concluded that higher

voltages and higher cell concentrations lead to a greater percentage of biomass recovery. The next step is to carry out more experiments, in order to effectively compare the two types of flocculation.

Table 1. Microalgae electroflocculation: comparison of biomass recovery in relation to conventional NaOH flocculation.

Cell concentration (cells / m^3)	Electric voltage (V)	Recovery (%)
$8.1 . 10^{14}$	30	94.3
$8.1 . 10^{14}$	15	88.8
$2.9 . 10^{15}$	30	97.6
$2.9 . 10^{15}$	15	89.3

3.4. Drying

In previous experiments, drying the microalgae biomass in natural incubators was tested; this process has the advantage of being extremely economical, but has the disadvantage of the long time required. Opposing to that, the drying with spray-dryer is quickly, but has power consumption. The following results were obtained:

Inlet temperature of the drying air = 250 ° C

Outlet temperature of the material = 108 ° C

Final humidity content of the material = 4.3 %

The low humidity levels achieved for the material encourages the use of this equipment in the drying of biomass. This advantage will be confronted with energy spending, to assess its possible use in larger scale.

4. CONCLUSIONS

Aiming the obtainment of biodiesel and other products of interest, the microalgae are being studied as a source of lipids. This study is being done on a pilot scale and not only in laboratory, because the expansion of scale has many advantages, such as better visualization of the process, the practice with equipment similar to industrial ones, simulation, optimization in the most realistic way and achieving of the mass and energy balances with values more closer to those used industrially.

A pilot photo-bioreactor has already been built with transparent tubes 6 cm in diameter, positioned to receive the maximum luminosity. Then, thanks to the culture medium and the presence of CO2, there will be growth of microalgae. The monitoring of cell concentration with optical sensor proved to be a good way to monitoring the progress of the process.

To separate the biomass from its suspension, flocculation tests were performed with the use of NaOH. It was noted that the final pH was around 10 and this procedure facilitates the next operation which is sedimentation. With the results already obtained for the flocculation and settling time, a tank is being scaled to perform this separation. Also other factors that influence the settling of the biomass will be studied. Currently tests are being conducted with electroflocculation and results look promising.

The electroflocculation is a new process that is still rarely used, but appears to be promising. The biomass recovery achieved by this process was, on average, is over 90% of the obtained by conventional NaOH flocculation. Despite this little “loss” and of the consumption of energy, electroflocculation has the advantages of being a cleaner process because there are no chemical substances added to the medium. In addition, other studies are currently being done to conclude that the consumption of electricity in electroflocculation is small.

After decanting, the biomass should be dried. Experiments with a spray-dryer have been realized, in which were achieved values of final humidity content of around 4 %, considered excellent for the process.

Thus, the process of cultivation of microalgae in a compact photo-bioreactor with the consequent separation of biomass is being studied, so it can be fully done in pilot scale. It is expected that in a short time, the lipids from microalgae are a viable source of the biodiesel production on a large scale.

This work was being realized thought that all these operations must to consume a little energy as possible, because the goal of this process is self-sustainability.

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