

SPRAY DRYING OF *Nannochloropsis oculata* MICROALGAE BIOMASS

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Abstract. *Spray drying is used in an ample variety of applications from medicines production to food industries. In this process, the fluid is atomized and dispersed like slim droplets which enter in contact with a hot air flow. These droplets provide an extensive superficial area for heat and mass transfer. However, the cooling by evaporation and the short residence time result a low product temperature. Basically, the sensible heat lost from the hot air provides the latent heat to evaporate the liquid around the product. The purpose of this work is the production of dry *Nannochloropsis* microalgae biomass with a spray dryer system before oil separation and biodiesel production. The microalgae were grown in 0.2 m³ tanks with aeration, in a vegetation greenhouse. After 10 days of development the microalgae were flocculated with NaOH addition. This flocculated material was then concentrated by centrifugation and submitted to spray dryer. The ventilation, compression and heating systems and the peristaltic pump of the equipment were started. The thermostat adjusts and suctioned liquid flow were modified during the experiment, and the atomizer pressure was regulated to 1 kg/cm² absolute. The dry material was collected in a vessel at the exit of the side cyclone, which was maintained opened. At the end of this process we obtained powdered *Nannochloropsis oculata* biomass, which was submitted to lipid extraction with solvent, and thereafter to biodiesel synthesis. The experimental results suggest that the proposed process is a suitable methodology to be applied at Self-Sustainable Energy Research & Development, from Federal University of Paraná.*

Keywords: *spray dryer, microalgae, dry biomass production, biodiesel production*

1. INTRODUCTION

Microalgae can provide several different types of renewable biofuels. These include methane produced by anaerobic digestion of the algal biomass, biodiesel derived from microalgal oil and photobiologically produced biohydrogen (Chisti, 2007).

For biodiesel derived from microalgal oil production its necessary the extraction of the microorganism lipid content. Some of these extractions, like solvent extraction, needs no water presence in the material to a suitable process. According Olaizola (2003), microalgal biomass can be dehydrated in spray dryers, drum dryers, freeze dryers and sun dryers.

Drying is defined, in general, as the liquid remove from a solid by evaporation. In the drying process, the wet material is in contact with the unsaturated air and it's obtained as result the humidity decreases of this material and the air humidification. Two stages define the total drying process: the material heating and the humidity evaporation. So, the drying has as purpose the product humidity reduction to a desirable level (Strumillo and Kudra, 1986).

Water may be evaporated from a solution or a suspension of solid particles by spraying the mixture into a vessel through which a current of hot gases is passed. In this way, a large interfacial area is produced and consequently a high rate of evaporation is obtained. Drop temperatures remain below the wet bulb temperature of the drying gas until drying is almost complete, and the process thus affords a convenient means of drying substances which may deteriorate if their temperatures rise too high, such as milk, coffee, and plasma (Coulson and Richardson, 2002). This way of drying characterize spray dryer.

This paper describes drying experiments of *Nannochloropsis oculata* microalgae using a spray dryer. According Gwo *et al.* (2005), *Nannochloropsis oculata* strain is a small green alga, characterized by spherical or slightly ovoid

cells, about 2-5 μm in diameter. Spray drying is applicable with low size substances, such as powdered milk, for example. Therefore, we assumed the viability of this application for microalgae drying.

Drying tests were made with variation of operation parameters of the spray dryer equipment, such as temperature and inlet mass flow, controlling as dependent variable the humidity of the final product (powdered microalgae).

2. MATERIAL AND METHODS

Nannochloropsis oculata microalgae were grown in 0.2 m^3 tanks by Grupo Integrado de Aquicultura e Estudos Ambientais from Federal University of Paraná, in appropriated culture broth, with aeration, during 10 days, in a greenhouse. After this period, the microalgae were separated from the growth solution by flocculation with pH change, using 1.0 M (molar) sodium hydroxide (NaOH) solution. This causes microalgae flocculation, and then settling. The bottom solution was transferred to 0.02 m^3 vessels, as shown at Fig. 1. The clarified fraction, with low cell concentration, was neutralized and discarded. The solution was centrifuged during 300 seconds, at 3500 rpm, to remove the water excess still present.



Figure 1. Microalgae after flocculation, settling and packaging in 0.02 m^3 vessel.

The concentrated solution was dried, using spray dryer, schematically represented in Fig. 2, which are indicated the material inlet (1), the product exit (2) and the hot air exit (3). Thereunto, the ventilation, compression and heating systems, and the peristaltic pump were started. The atomizer pressure was adjusted to 1 kg/cm^2 absolute. The operation temperature (T_{op}) and the entry mass flow (ω) were changed during the experiments, aiming observe the influence in the dried material final humidity. The used values are shown in Tab. 1. Previously published researches with spray-drying of *Dunaliella salina* microalgae shown that an inlet temperature nearly of 473.15 K doesn't causes excessive degradation or composition changes in the biomass (Leach *et al*, 1998), why the less inlet temperature used in the experiments is 473.15 K. The dried microalgae, shown at Fig. 4, were collected in appropriate receivers in the equipment exit.

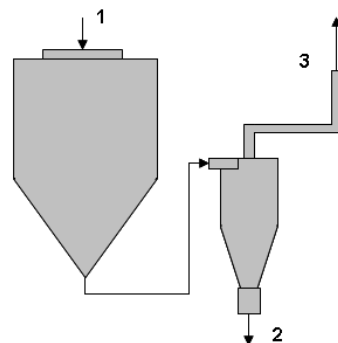


Figure 2. Spray dryer diagram. (1) Inlet; (2) Material exit; (3) Hot air exit.



Figure 3. Spray dryer used at the drying tests.

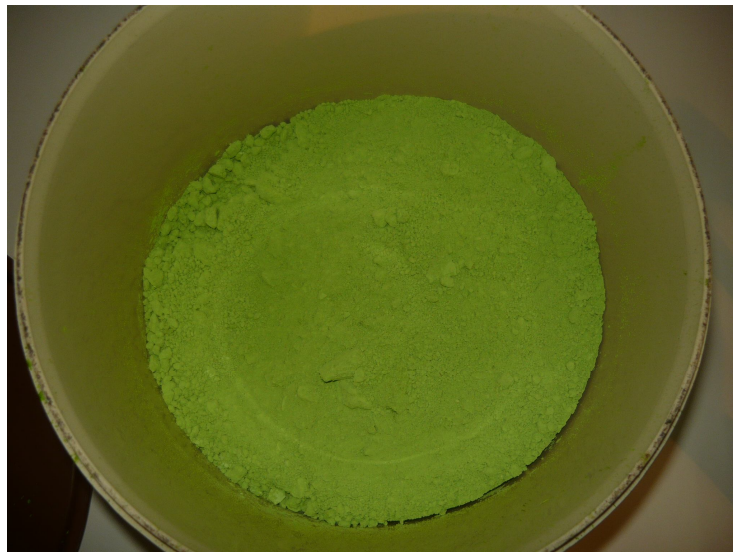


Figure 4. Powdered *Nannochloropsis oculata* biomass, obtained at the end of the drying process.

With a thermocouple type J, positioned in the exit tubing, the material exit flow temperature (T_m) was measured. The measure errors associated with the material exit temperature are available at Exacta Group website, a thermocouple and other temperature sensors producer. These limits meet the ASTM e-230 standards, union reference at 273.00 K, disregarding installation or system errors.

The mass flow values are obtained using an electronic weighing-machine and a digital chronometer.

Table 1. Inlet mass flow (ω), operation temperature (T_{op}) and material exit flow temperature (T_m) values.

Experiment	ω (kg/min)	T_{op} (K)	T_m (K)
1	$0.10 \pm 5 \times 10^{-8}$	523.15 ± 5.00	381.15 ± 2.20
2	$0.06 \pm 5 \times 10^{-8}$	523.15 ± 5.00	381.15 ± 2.20
3	$0.06 \pm 5 \times 10^{-8}$	473.15 ± 5.00	351.15 ± 2.20
4	$0.10 \pm 5 \times 10^{-8}$	473.15 ± 5.00	351.15 ± 2.20
5	$0.08 \pm 5 \times 10^{-8}$	498.15 ± 5.00	366.15 ± 2.20

After dried, the products were reserved in a dessicator with silica gel, and then submitted to humidity tests. For this, were prepared aluminum foil vessels, which were properly identified and kept during one hour in a laboratory oven at 378.15 K, to water removal. After this step, the vessels were placed inside the dessicator, for cooling, and weighted in analytical precision balance, obtaining m_r values. Of each experiment, final product samples was placed into the vessels and carried into the laboratory oven, at 378.15 K during one hour, to humidity removal. Subsequent, the samples were removed into dessicator for cooling, and then weighted, obtaining m_{dr} values, referent of free humidity mass.

Is important to emphasize that the spray dryer used to drying executions is working since 1966, as shown at Fig. 5, and the instruments for operation temperature measuring cannot effectively be precise.



Figure 5. Spray dryer specifications.

3. RESULTS AND DISCUSSION

Behind the drying, the humidity tests were made, how said previously. The medium weighing values, and the values of calculated humidity, are presented in Tab. 2. Humidity is a gravimetric analysis, and the final values are calculated just linking the mass difference of the materials, before and after placed in the laboratory oven, during one hour at 378.15 K, and presented as percentage values.

Table 2. Mass values of vessels (m_r), samples (m_{wt}) and dry samples (m_{dr}), and humidity results.

Experiment	m_r (kg)	m_{wt} (kg)	m_{dr} (kg)	Humidity (%)
1	$6.622 \times 10^{-4} \pm 1.633 \times 10^{-7}$	$7.965 \times 10^{-4} \pm 2.216 \times 10^{-7}$	$7.637 \times 10^{-4} \pm 6.403 \times 10^{-8}$	$4.12 \pm 4.35 \times 10^{-4}$
2	$4.985 \times 10^{-4} \pm 1.155 \times 10^{-7}$	$1.162 \times 10^{-3} \pm 2.708 \times 10^{-7}$	$1.110 \times 10^{-3} \pm 6.994 \times 10^{-8}$	$4.43 \pm 3.04 \times 10^{-3}$
3	$6.047 \times 10^{-4} \pm 1.826 \times 10^{-7}$	$7.520 \times 10^{-4} \pm 1.000 \times 10^{-7}$	$6.916 \times 10^{-4} \pm 8.699 \times 10^{-8}$	$8.03 \pm 2.60 \times 10^{-4}$
4	$6.193 \times 10^{-4} \pm 2.449 \times 10^{-7}$	$1.081 \times 10^{-3} \pm 5.774 \times 10^{-8}$	$1.026 \times 10^{-3} \pm 5.774 \times 10^{-8}$	$5.10 \pm 5.22 \times 10^{-5}$
5	$6.121 \times 10^{-4} \pm 1,826 \times 10^{-7}$	$1.103 \times 10^{-3} \pm 5.000 \times 10^{-8}$	$1.015 \times 10^{-3} \pm 6.473 \times 10^{-8}$	$8.00 \pm 3.50 \times 10^{-2}$

To facilitate the results observation, Tab. 3 presents the relation of drying standards used in the operation, and de percentage of humidity in the final products.

Table 3. Operation parameters values and humidity results.

Experiment	ω (kg/min)	T_{op} (K)	T_m (K)	Humidity (%)
1	$0.10 \pm 5 \times 10^{-8}$	523.15 ± 5.00	381.15 ± 2.20	$4.12 \pm 4.35 \times 10^{-4}$
2	$0.06 \pm 5 \times 10^{-8}$	523.15 ± 5.00	381.15 ± 2.20	$4.43 \pm 3.04 \times 10^{-3}$
3	$0.06 \pm 5 \times 10^{-8}$	473.15 ± 5.00	351.15 ± 2.20	$8.03 \pm 2.60 \times 10^{-4}$
4	$0.10 \pm 5 \times 10^{-8}$	473.15 ± 5.00	351.15 ± 2.20	$5.10 \pm 5.22 \times 10^{-5}$
5	$0.08 \pm 5 \times 10^{-8}$	498.15 ± 5.00	366.15 ± 2.20	$8.00 \pm 3.50 \times 10^{-2}$

Table 3 analysis allows see that higher humidity values in spray dried products are from lower mass flows (0.06 and 0.08 kg/min), being the experiments 3 and 5 with higher humidity values (8.03 and 8.00 %, respectively). These experiments were performed with lower operation temperatures. At experiments 1 and 4, with 0.10 kg/min mass flow, there are values considerably lower than experiments 3 and 5. Experiment 2 was made with low mass flow, but with high operation temperature, and presented second lower humidity value.

At the same time, comparing the different used temperatures, when associated with the same mass flow, higher temperatures values result in lower humidity. The temperature influences can be related with the fact that, with higher system temperatures, there is greater heat amount, which provides more energy for evaporation of material water.

Is possible to say that temperature is the main factor that influences the final humidity content, since comparing same mass flows, there are substantial difference of humidity between different temperatures.

The microalgae tank cultivation requires a considerable framework, and demands a relative long time to get enough quantity to spray drying tests. There are many conditions that influences the growth time, like the sun, temperature, etc., which may further delay the process. So, taking into account also that the drying process requires a lot of biomass to obtain concise results of humidity analysis, there are not many variety of results. Moreover, the main objective of this work is to prove the applicability of *Nannochloropsis oculata* drying with a spray dryer system, for your potential application at Center for Self-Sustainable Energy Research & Development, from Federal University of Paraná. Preliminary assessments show that spray dryer system can operate using the hot air flow obtained from an exhaust of a generator of electricity, reason why this methodology may be considered suitable.

4. CONCLUSIONS

As supposed, spray dryer is an effective equipment for microalgae drying. Low humidity values were obtained, allowing the execution of some kind of extractions, as solvent extraction, and further biodiesel production. Though the spray dryer operation needs a substantial energy charge to evaporate the water, this work shows that microalgae spray drying is possible. The low energy consumption objective refers to use heat from a generator, provided at Center for Self-Sustainable Energy Research & Development, from Federal University of Paraná, who support this work. So, with this application, spray drying can be used without other forms of energy expense.

5. ACKNOWLEDGEMENTS

To Center for Self-Sustainable Energy Research & Development, from Federal University of Paraná. To National Counsel of Technological and Scientific Development (CNPq) and to Nilko Metallurgy. To Grupo Integrado de Aquicultura e Estudos Ambientais, from Federal University of Paraná, by microalgae cropping and cooperation with flocculation procedures. To Dr. Marcos Rogério Mafra and Dr. Maria Lucia Masson by the laboratories lending for tests execution.

6. REFERENCES

- Chisti, Y., 2007, "Biodiesel from microalgae", *Biotechnology Advances*, No. 25, Elsevier, pp. 294-306.
- Coulson, J. M., Richardson, J. F., 2002, "Chemical Engineering", Vol. 2, Particle Technology and Separation Processes, Ed. Butterworth-Heinemann, Oxford, USA, 1208 p.
- Grupo Exacta, 2006, "Limite de erro para termopares de isolação mineral", 10 Aug. 2009, <http://www.exacta.ind.br/tecnicas_limites_aplicacao.shtml>
- Gwo, J.-C., Chiu, J.-Y., Chou, C.-C., Cheng, H.-Y., 2005, "Cryopreservation of a marine microalga, *Nannochloropsis oculata* (Eustigmatophyceae)", *Cryobiology*, No. 50, Elsevier, pp. 338-343.
- Leach, G., Oliveira, G., Morais, R., 1998, "Spray-drying of *Dunaliella salina* to produce a β -carotene rich powder", *Journal of Industrial Microbiology & Biotechnology*, No. 20, Society for Industrial Microbiology, pp. 82-85.
- Olaizola, M., 2003, "Commercial development of microalgal biotechnology: from the test tube to the marketplace", *Biomolecular Engineering*, No. 20, Elsevier, pp. 459-466.
- Rodrigues, M. I., Jemma, A. F., 2005, "Planejamento de Experimentos e Otimização de Processos – Uma estratégia seqüencial de planejamento", Ed. Casa do Pão, Campinas, Brasil, 326 p.

Strumillo, C. Z., Kudra, T., “Drying: Principles, Applications and Design”, In Hughes, R. Topics in Chemical Engineering, Vol.3, Gordon and Breach Science Publisher, UK, 1986.

7. RESPONSIBILITY NOTICE

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