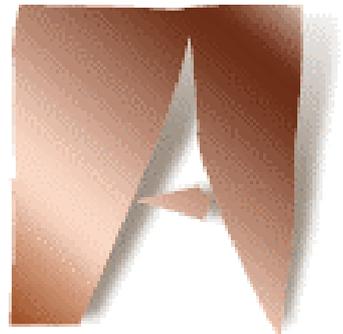




**Laboratoire Automatique et Génie des Procédés**  
Université Claude Bernard, Lyon 1  
CPE, Bât 308G, 43, bld du 11 nov 1918  
Tél: (33) 04 72 43 18 67 Fax: (33) 04 72 43 16 99  
69622 Villeurbanne cédex



**Departament d'Enginyeria Química**  
Escola Tècnica Superior d'Enginyeries  
Universitat Autònoma de Barcelona  
Tel.: 93.581.10.18 Fax: 93.581.20.13  
08193 Bellaterra Spain

# MELISSA

Memorandum of Understanding

ECT/FG/MMM/97.012

Contract Number: ESTEC/CONTRACT13292/98/NL/MV

## Technical Note: 62.6

*Spirulina* biomass treatment and acceptability

**Version: I**

**Issue: 0**

**DESMORIEUX, H.; ALBIOL, J.; GODIA, F.**

**July 2002**

**Document Change Log**

Version	Issue	Date	Observations
Draft	0	07/06/02	Preliminary Version
Version 1	1	30/07/02	Original Version

## **Table of Contents**

1. <i>SPIRULINA</i> PRODUCTION. METHODOLOGY AND RESULTS.....	5
1.1 Introduction .....	5
1.2 Strains .....	5
1.3 Culture medium : .....	6
1.4 Reactors : .....	6
1.5 Conclusion : .....	10
2. CONVECTIVE DRYING OF <i>SPIRULINA</i> .....	11
2.1 Introduction .....	11
2.2 Materials and methods.....	11
2.3 Convective dryer.....	11
2.3.1 <i>Spirulina</i> preparation .....	12
2.3.2 Drying rate curves .....	13
2.3.2.1.1 Establishment of the Characteristic Drying Curve .....	15
2.4 Results .....	16
2.4.1 Influence of air temperature .....	16
2.4.2 Comparison between different types of <i>Spirulina</i> .....	17
2.4.3 Influence of diameters of cylinders .....	18
2.4.3.1 Characteristic Drying Curve of cylinders .....	20
2.4.4 Others specific parameters .....	21
2.5 Conclusions .....	21
3. FREEZE DRYING OF <i>SPIRULINA</i> .....	23
3.1 Introduction .....	23

3.2 Materials and methods.....	23
3.3 Results : .....	25
3.4 Conclusions: .....	27
4. SPIRULINA PRODUCTION FOR HUMAN CONSUMPTION.....	29
4.1 Introduction .....	29
4.2 Results of the interview : .....	30
<b>4.2.1</b> 2.1. Culture : .....	30
4.2.2 2.2. Drying - filtration : .....	30
5. REFERENCES : .....	31

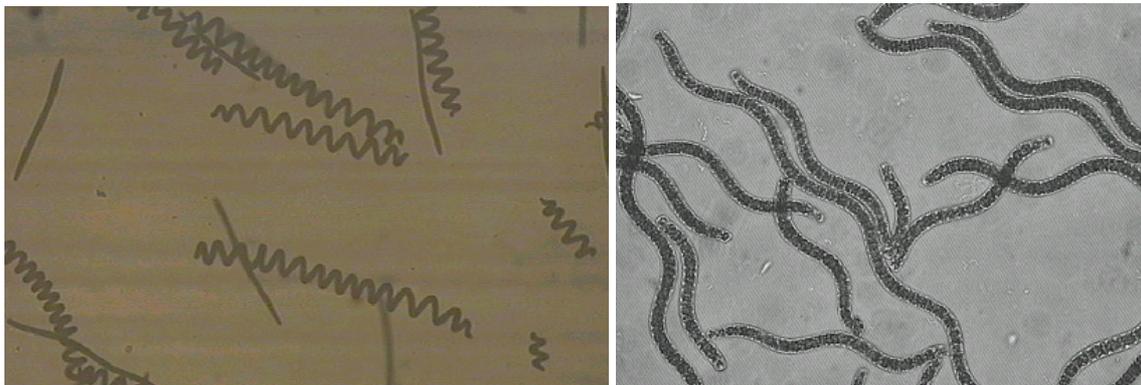
## **1. SPIRULINA PRODUCTION. METHODOLOGY AND RESULTS**

### **1.1 Introduction**

To cultivate the *Spirulina platensis* at the laboratory was a completely new topic in this unit of engineering of the processes. The main task was to learn to start a culture from a small quantity of *Spirulina* and that with simple and manual means. The culture was carried out in two reactors, one of 40 liters working volume and the another one of service output of 25 liters. Many preliminary tests were carried out for sowing in culture. Brutally failures of culture arrived at 4 recoveries, which made that we concentrated the study on the seeding and maintains it in culture.

### **1.2 Strains**

The strain comes from a farm from the south of France. It is spiral, like the *Spirulina* strain observed at the UAB (Pasteur culture collection PCC-8005). The culture contains at the same time filaments right and spiral, figure 62.31.1 a). Currently, we have replaced one of the cultures by that presented on figure 62.31.1 b) that is more corrugated. In the near future a new culture of reference will have to be considered, it is the strain coming from one industry of Saint Nazaire (Alpha - Biotech) which cultivates the *Spirulina*, and who works with the laboratory GEPEA which study the membrane filtration of *Spirulina* for ESA.



a) spiral and straight Spirulina (x25)

b) wavy Spirulina(x50)

**Figure 62.61.1** : Photo of two cultures of *Spirulina* ( in (2001-a) et (2002 b).)

### **1.3 Culture medium :**

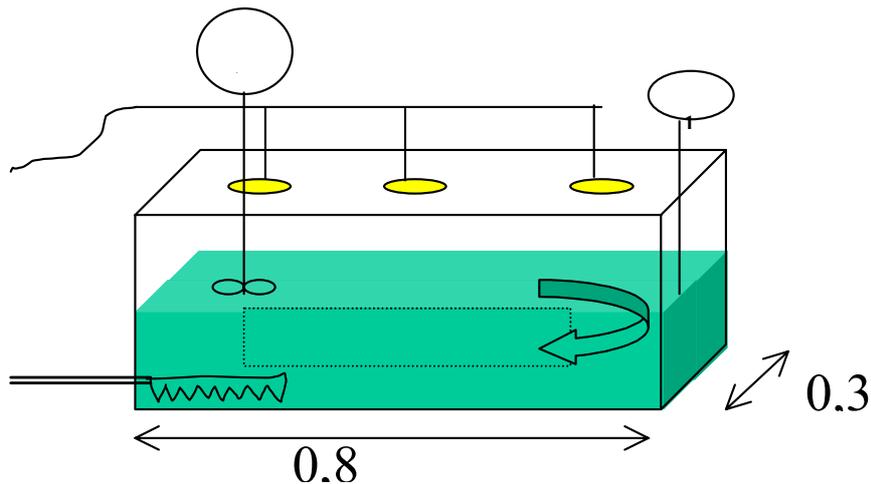
Initially, the culture was carried out under a culture medium simplified, according to the method of J.P. Jourdan [Jourdan, Cultivez votre Spirulina, 2000]. We currently provide ourselves the products necessary to the solutions "A5" et "B6" of the Zarrouk medium.

### **1.4 Reactors :**

Two reactors were installed. They are opened to the air, fed in source of light by the surface of the culture, agitated either by bubble, or by an agitator with pale, heated by aquarium immersion heaters.

#### **1<sup>er</sup> parallelepiped reactor:**

This reactor lays out a used volume of around 40 to 60 liters.



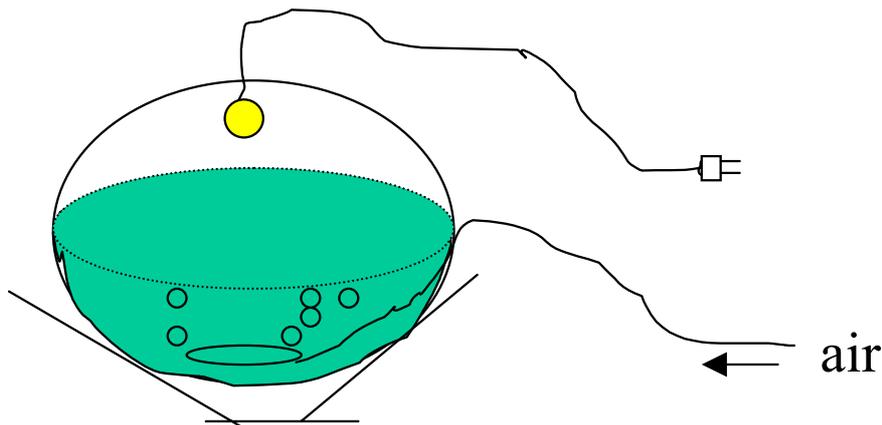
**Figure 62.61.2 :** Principe of the reactor.

- Lighting is ensured by three halogenous lamps 12V X 50W, model 077 (Iltech mark)
- The temperature is maintained for the moment with 28°C, by local heating by resistance's immersion heaters
- Agitation is ensured by an blades agitator with 32 T/min.

**2<sup>ème</sup> reactor : half sphere :**

It is two half spheres posed one on another, one being useful for the culture, the other is to protect from dust...

The diameter is 56cm and the total volume is 45 liters, 25 liters are used for the culture.



**Figure 62.61.3 :** Principe of the 2<sup>nd</sup> reactor.

- Lighting is ensured by an incandescent lamp of 60W, placed just at the top of the culture. We also used a solar lamp of 75W
- The agitation be ensured by bubble with ambient air in the part lower of container
- The heating be assured either by the power release by the lamp to obtain 28°C, either, in winter, by a immersion heater, to obtain a temperature close of 22°C.

There is no control on line, and the parameters of temperature and lighting are variable. The outside temperature also being able to have an influence.

**New reactor :**

A simple reactor must be built with a possibility of variation of the source of light, making it possible to control, manually, the temperature and the source of light: It will be a vertical one meter length cylindrical reactor. Agitation will be done by bubbling at the bottom of the reactor. It is envisaged to install on a frame, a rape neon (incandescent or fluorescent) coupled to a slope of halogen lamps of 50W each one, on

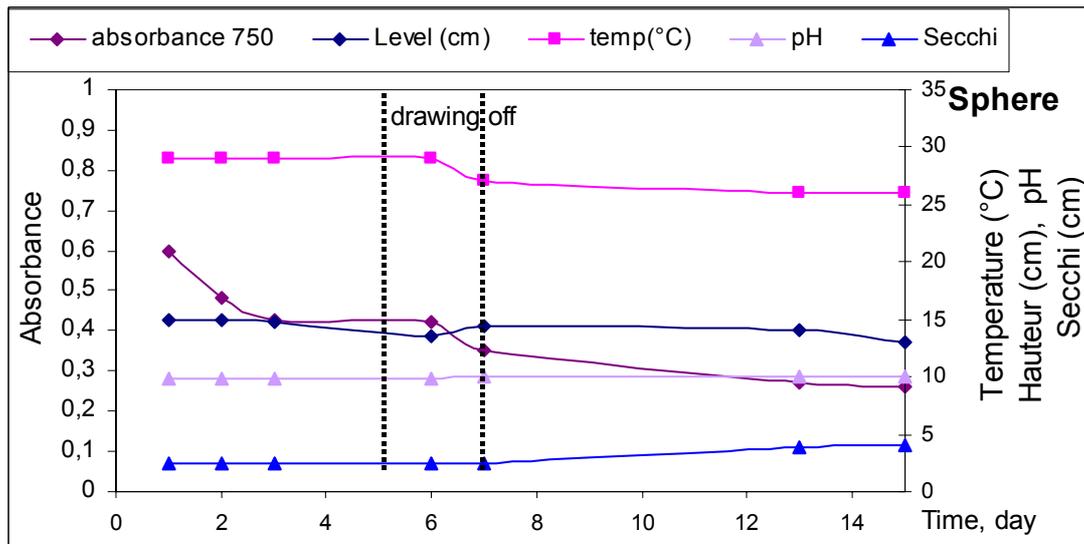
a square section, with regulator. Harvest will be either in bottom of the reactor, or at the top. A possibility of heating by immersion heater or for auxiliary heating is considered for sowing.

The measurement of the pH, the concentration will be done outside the reactor, by taking away.

**5. Follow-up of the cultures :**

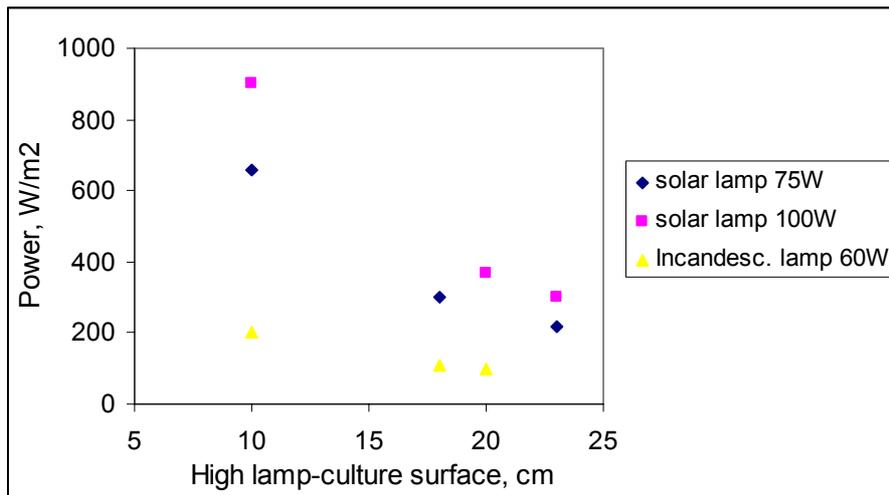
The pH of the two cultures is measured either by paper pH or by glass electrode. It varies between 9,0 and 10, 5. The temperature is measured by alcohol thermometers placed in the culture medium. It is now possible to measure the concentration at the laboratory by spectrophotometer, at 750nm.

Figure 62.61.4 shows an example of the follow-ups of the culture, with a modification due to the taking away for experiments



**Figure 62.61.4 :** example of follow up of the culture .

The light power received by the *Spirulina* was estimated by a pyranometer which records the luminous power on all the solar spectrum. Thus, in the sphere, the power was measured on the diameter of the sphere according to the height, appears on the figure below:



**Figure 62.61.3 :** Light power measured with a pyranometer.

In the rectangular reactor, the average measured power varies according to the place of the measurement (under the lamps or in the corners of the reactor) from 200 to 700 W/m<sup>2</sup>.

## 6. Proteins :

An analysis of the content of proteins of the culture, by the Lowry method, was carried out at the laboratory. That led to a content varying of 30 to 50%. Others measurements will be remade the next months.

## 7. Drying :

Harvest is done by filtration on polyamide filter to 15, 20 or 25 µm, according to the size of the culture, the width of the filaments varies in the course of time in the same culture. The majority of the experiments in 2001 were made with received *Spirulina* from the farms of Mr. Jourdan. Tests were carried out with the *Spirulina* of the laboratory.

Note:

The culture in batch reactors makes that Exopolysaccharides and sludge fall at the bottom (by involving part of the *Spirulina*). A strong agitation can makes them also

reappear on the surface. Thus, one could recover and to dry those sludge while supposing they are with strong content of EPS.

### **1.5 Conclusion :**

The main result is to have improved the *Spirulina* culture, in spite of our inexperience in biology and in the culture of algae. The new reactor should make it possible to get a culture which grow more quickly. This in spite of the absence of follow-up and control on line.

It is necessary to specify the form of the *Spirulina* that is going to be used for the space stations. It appears more significant for us to specify the form (spatial structure) that the name of the strain, since the documents of the agency space presents sometimes a very spiral *Spirulina* or sometimes, a corrugated *Spirulina*. The comparison that we made of the drying of *Spirulina* rather spiral or rather straight show that this parameter doesn't influence the maximum drying rate obtained at the beginning of the drying (WP 62.62). These experiments could not be remade because we do not have *Spirulina* yet perfectly straight (and the danger to contaminate is to be avoided). However, drying being a phenomenon of transport in porous environments, during the first phase of drying or the capillarity intervenes much, a characterization of the average structure of the biomass of the *Spirulina* appears significant. That can take place by analysis of image (counting by microscope) to characterize % of *Spirulina* straight and spiral in the culture, and also to characterize the evolution of the culture.

## **2. CONVECTIVE DRYING OF SPIRULINA**

### **2.1 Introduction**

The aim of this work package is the characterization of forced convective drying of *Spirulina* with varied drying parameters. The quality of the product is the one of the *Spirulina* we get from a producer.

First, we compare with some experiments, if the convective drying of the new product gives the same results or not that we had obtained the last year for the *Spirulina* dried in layer. The *Spirulina* used for the both years come from two different producers in south of France. The type of *Spirulina* is totally different. The first one is a straight form, the over one is a mixed wavy and spiral form.

Then, the work consists to study the drying of small cylinders. The producers in France use this form for drying *Spirulina* and it seems to give a product that is better accepted by the consumers.

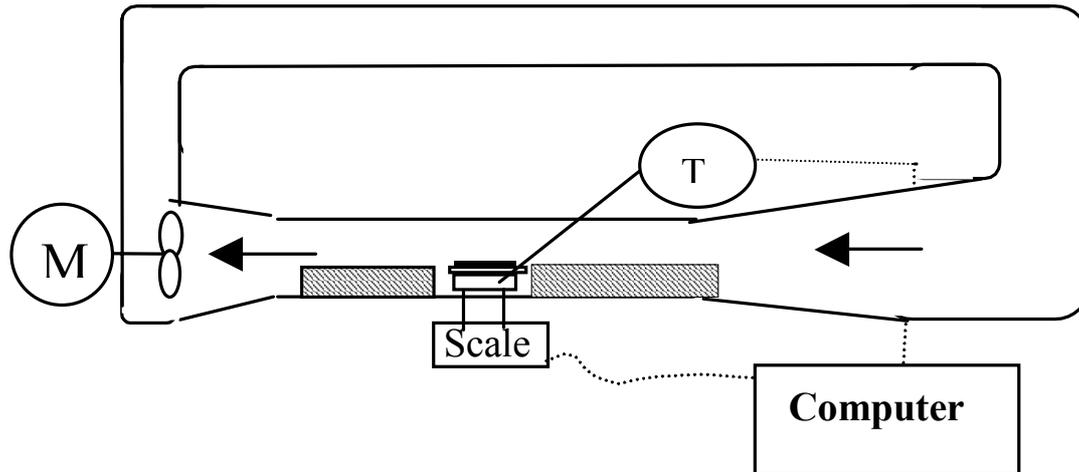
The characterization of each drying is possible by applying the method of the characteristic drying curves concept (CDC). This method includes the drying rate curves in a single curve for one product, with given dimension, structure, arrangement on the support and in a range of air conditions (temperature and velocity).

### **2.2 Materials and methods**

#### **2.3 Convective dryer**

Convective drying is carried out in a closed loop, figure 62.62.1. The temperature and velocity of air are fixed constant during a experiment One experiment is 3 to 5 hours long. Relative humidity of air is measured by a hygrometer and the temperature of by refractometer surface, for the layers. Measurements are established in a range of temperature from 40 to 60°C and an air velocity of 2,1 m/s.

The mass, the temperature in the product is registered during drying time. These measure allows to establish the drying rate curves.



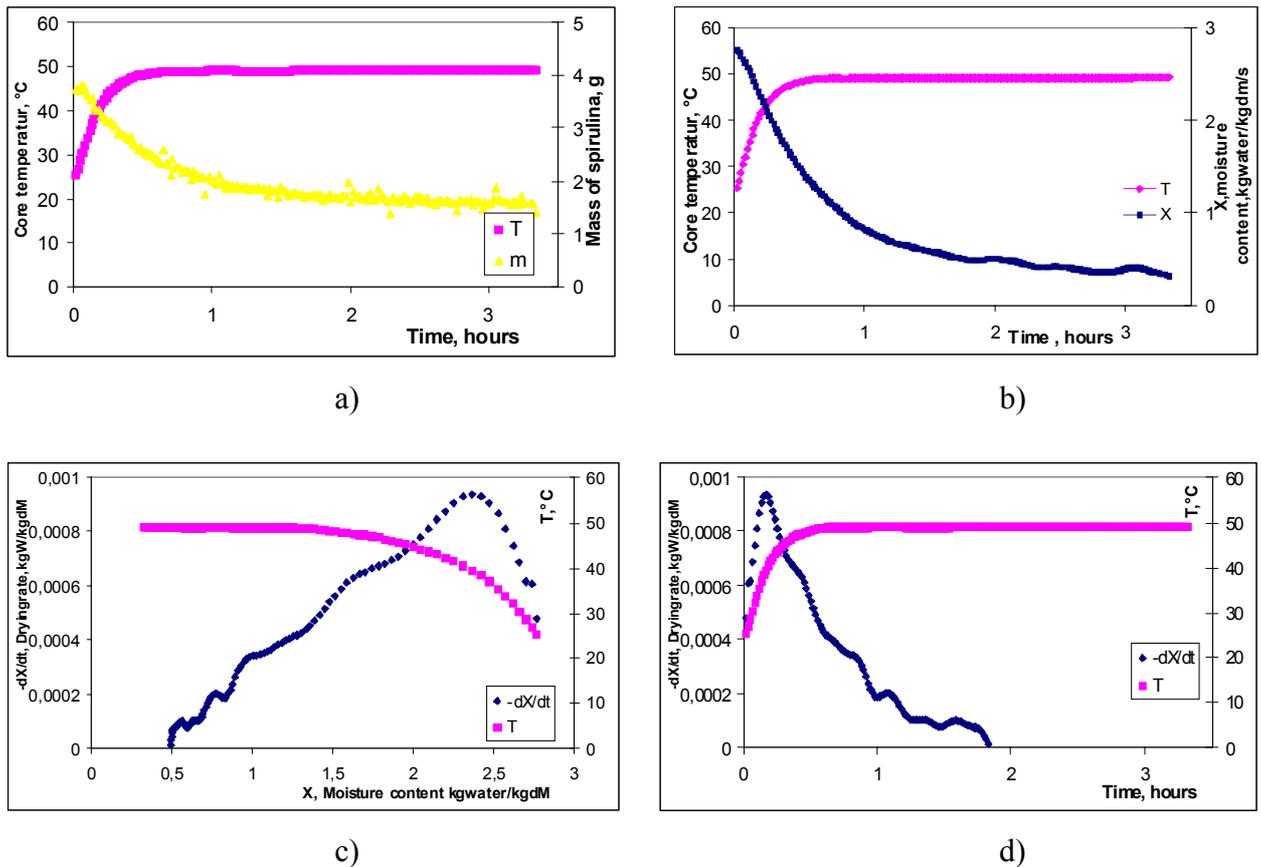
**Figure 62.62.1** : Principe of convective dryer

### 2.3.1 *Spirulina* preparation

The step of filtration and possible pressing make it possible to eliminate most of salt before convective drying, as that is practiced in the small farms. The solvent that have to be eliminate by drying is: the culture medium, the water in the sheath surrounding the cells and the water in the cells. According to the shape of the filaments of the *Spirulina*, rather spiral or rather linear, the water content initial varies from 3,5 to 7 kg of water/kg dry matter for a same pressure of filtration, the straight form retains more the culture medium. The *Spirulina* is spreading out in cylinders of 2 to 4mm. The cylinders are deposited on a plate with a syringe. The spreading out in thin layer is very delicate to realize because of the strong adherence of the *Spirulina* to the support and the support that allows the spreading out. But the thin layer allow to use a greater density of *Spirulina* on the support, than for cylinders. Due to the air flow, greater the mass, lower the noise of the measures. That means a higher initial mass of product.

### 2.3.2 Drying rate curves

After the measure of the dry matter, it is possible to obtain the evolution of the average moisture content according to the drying time. The data are smoothed and after derivation, it is possible to obtain the curve of the drying rate according to the average moisture content, like on figure 62.62.2, that show the evolution of *Spirulina* sample with initially 4 g at 50°C.



**Figure 62.62.2 :** Typical drying curve: **a)** Temperature, mass of *Spirulina* during time; **b)** Temperature, moisture content of *Spirulina* during time; **c)** Drying rate according to the time; **d)** Drying rate according to the moisture content.

The influence of the parameters (temperature of air, diameter) are observed on the curves drying rate versus moisture content, figure 62.62.2.d). This curve let appears the different period of drying :

When the drying rate is increasing : the warm up period.

When the drying rate is maximal, or constant : the first period and constant rate period : the external conditions limit the drying.

And when the falling rate period, when the internal transfer limit the process of drying.

### **External and internal heat and mass transfers**

The figure 62.62.2 a), b), c), d) show that at the beginning of drying, the initial product temperature is equal to the wet bulb temperature 25°C and reach the air temperature at the end of drying.

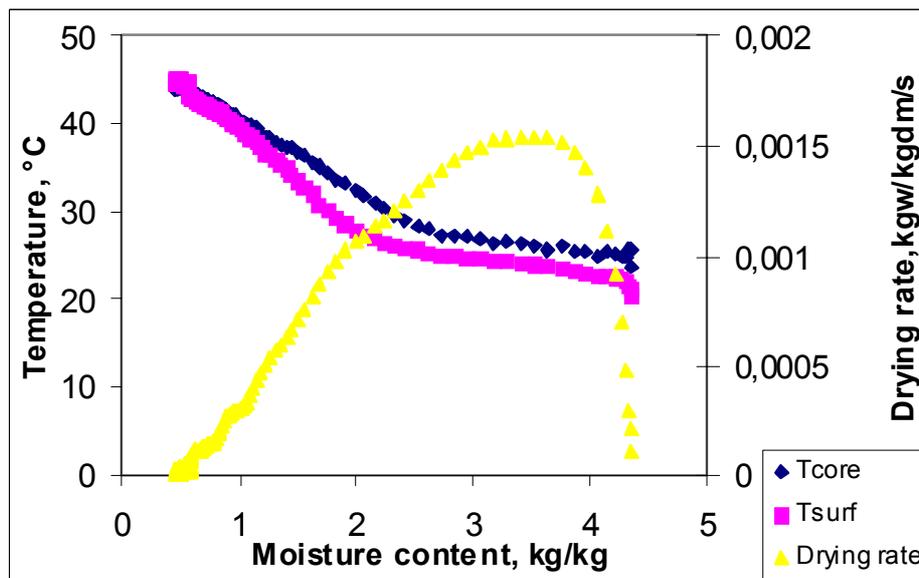
This phenomenon is observable on the curves of drying of layer where the temperature measurement of surface is possible by refractometer. One can then distinctly observe the possible difference in temperature between the core of the layer of *Spirulina* and the surface. The surface temperature can be constant when the drying rate is maximal and constant. During this phase, balance isenthalpic makes that surface is saturated. The surface of the product is at the wet bulb temperature, water is conveyed on the surface of the interior of the product (or the interior of the cylinder) rather quickly to compensate the evaporation on the surface. The transfer is then limited by the external conditions in the boundary layer.

When it is the internal transport which becomes limiting, i.e. which the routing of the water of the interior of the product towards surface does not compensate for evaporation on the surface, internal transport controls the process, the temperature of surface increases until the temperature of the air.

Remark: it be suppose but not check (because it be however difficult of measurement the temperature of a drop) that this same phenomenon intervene in the process of drying by spray drying, where the temperature of air can be very high (200°C) and the temperature of product in output of the dryer doesn't exceed 100°C.

When the drying rate is constant even then the surface temperature of the layer is constant , the curve -surface temperature according to the time- allow to determine  $X_{cr}$ , what is the moisture content between the first and the second phase, i.e. the transition to

which the internal transfer become to be limited. However, the characteristic of the drying of *Spirulina* is that the drying rate does not remain constant whereas the temperature measured on the surface can be constant, like on figure 62.62.3. This involves a difficulty of determining the end of the first phase and thus the critical moisture content. For that, we will refer to works of agricultural foodstuff drying [Fornell, 1980] those consider that the first drying phase is specific and corresponds to the maximum of the drying rate curve according to the water content.



**Figure 62.62.3:** Surface temperature and drying rate by drying of layer at 40°C and 2m/s.

### 2.3.2.1.1 Establishment of the Characteristic Drying Curve

Foodstuffs have heterogeneous structure, bad reproducibility of measurement and various composition throughout the sample. So, the characteristic drying curve method [Keey and Suzuki, 1974] (abbreviated CDC) is chosen to characterize a method of drying and a type of spreading out, with the help of experimental results.

The CDC method consists in reducing the moisture content and the drying rate, by a single normalized drying rate curve of one product with determined dimensions and for various given drying conditions. This curve is obtained by plotting  $f(\phi)$ , that is the drying rate  $v$  normalized by the drying rate during first period  $v_1$

$$f(\phi) = \frac{v}{v_1} = \frac{\left(\frac{dX}{dt}\right)}{\left(\frac{dX}{dt}\right)_1} \quad \text{versus} \quad \phi = \frac{\bar{X} - X_{eq}}{\bar{X}_{cr} - X_{eq}} \quad (1)$$

Were  $\bar{X}$  is the average moisture content,  $\bar{X}_{cr}$  is the average critical moisture content at the transition. As the constant drying rate doesn't appear clearly by some biological materials, the short period where the drying rate is maximum is considered as the first period. In this work, the moisture content at equilibrium  $X_{eq}$ , is replaced by the end moisture content, to get a normalization of the drying rate.  $f$  is the function which is assumed to be a characteristic of the drying of the product with determinate dimensions.

## 2.4 Results

### 2.4.1 Influence of air temperature

The figure 62.62.4 show the influence of the drying air temperature on convective drying of layer with a air velocity of 2m/s . These curve characterize drying of layer because the results are more reproducible with layer than with cylinders (this type of arrangement need more wet matter, the precision of the measure are better).

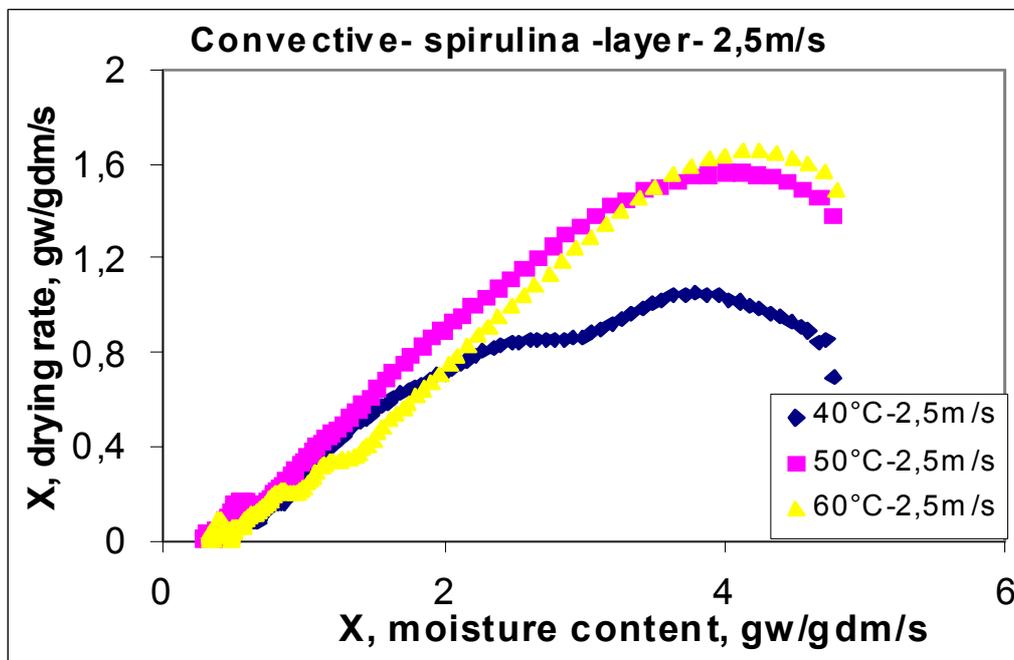


Figure 62.62.4: Drying of layers of Spirulina by 2,5m/s.

As the figure shows, higher the air temperature, higher the drying rate. The maximal drying rate, at the beginning of drying becomes higher, but the influence is not proportional. For low external conditions, the drying rate curves are more flattened.

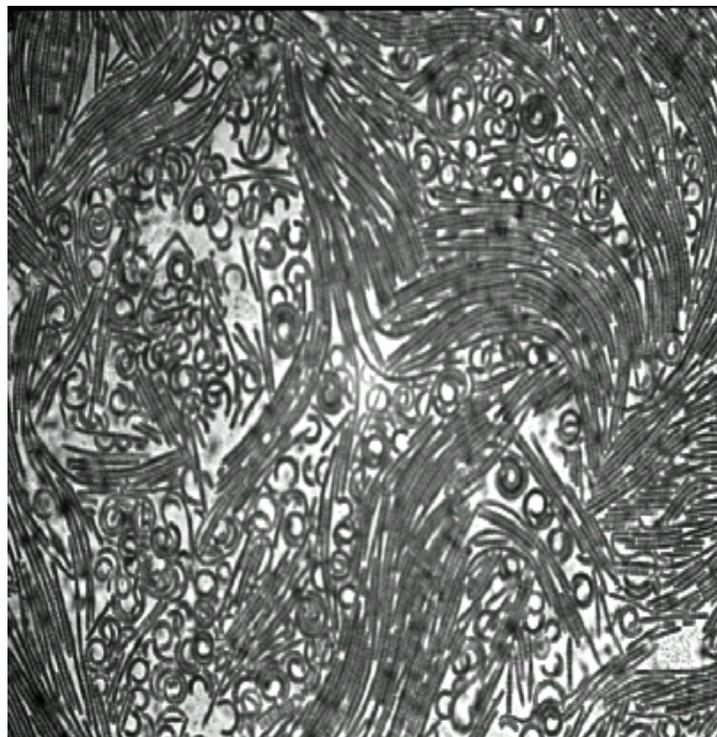
#### 2.4.2 Comparison between different types of *Spirulina*

We don't carry the comparison between the drying of the *Spirulina* in the laboratory and the *Spirulina* used for (precedent) works, due to the actually two small quantity of the *Spirulina* cultivated in the laboratory. For this work, we used another *Spirulina*, that comes from a farms from a producer.

These *Spirulina* is different from the *Spirulina* that we used the year before.

Then, we compare the convective drying of thin layer, with the both strains.

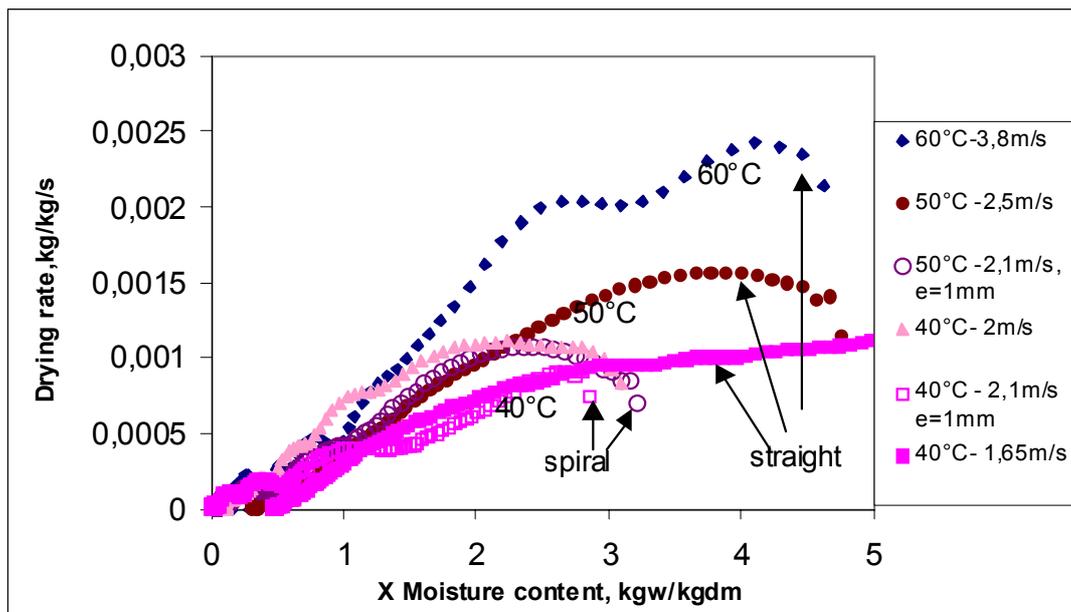
The figure 62.62.6 show the result that are done on 1999 and the results in 2001, with the both different *Spirulina*. These both *Spirulina* are different, specially in they shape. One *Spirulina*, used in 1999, comes from a farms, it has a high percent of straight shape, as showed on the figure 62.62.5. The other *Spirulina* used in 2001, from another farms, is typically spiral and wavy.



**Figure 62.62.5** : microscopic view of the used straight *Spirulina*.

These result allows also to see the influence of the shape of the filament.

The figure 62.62.6 show that it doesn't appears difference due to the shape of the *Spirulina* or due to the different origin of *Spirulina*. Although the drying of the reference of *Spirulina* have to be study, these results show that the external conditions are deciding at the beginning of drying.



**Figure 62.62.6 :** Drying rate curves of the two type of *Spirulina* in layers, by 40, 50 and 60°C.

#### 2.4.3 Influence of diameters of cylinders

First, the observation of the dried cylinders results to limits the dimensions that have to be respected. Then influence of the diameter of the cylinders in this range is observed by the drying rate curves. With beyond a diameter of 4mm, the external crust of the cylinder led to a too great heterogeneity of the product between surface and the interior, the *Spirulina* can then remain wet in the interior and time drying to become much longer. The shrinkage of the product during drying is not equal to the evacuated water volume but is definitely lower. Because of the shrinkage of the product and also by a possible fermentation, the cylinders of *Spirulina* presents a " tunnel " to the internal. The dry cylinders, initially of 4mm, are hollow rolls. With the break, these pores seem to be unfavorable with the visual quality of the product. It is thus interesting to dry of the small diameters, which in addition decreases the time of drying. A diameter lower than 2mm is possible in practice but not possible for the study of forced

convective drying because the phenomenon of shrinkage breaks the product and the fine particles of cylinders can be carried by air flow. The thickness of the cylinders is thus limited between 2 and 4 mm.

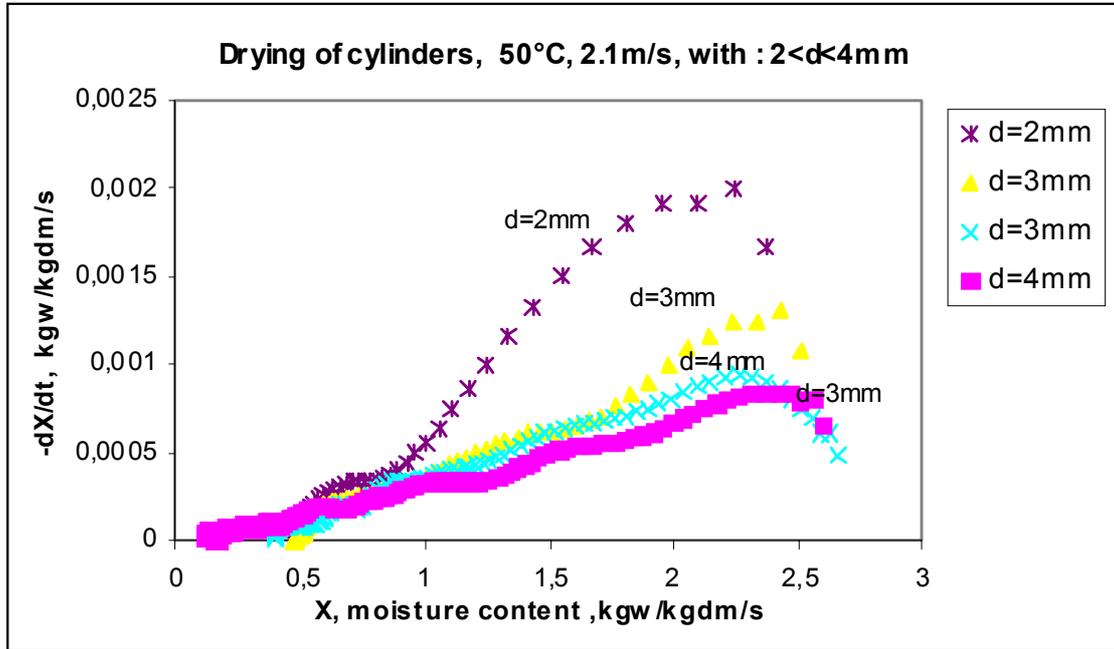


Figure 62.62.7 : convective drying rates, by varied diameters of *Spirulina* cylinders, at 50°C.

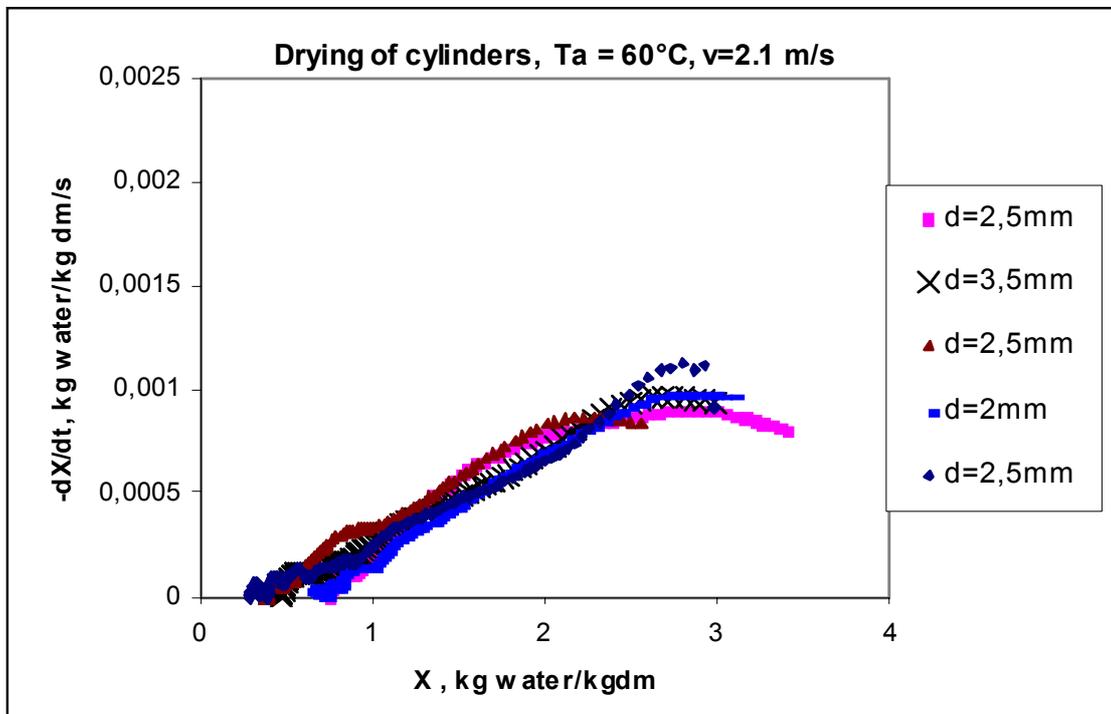
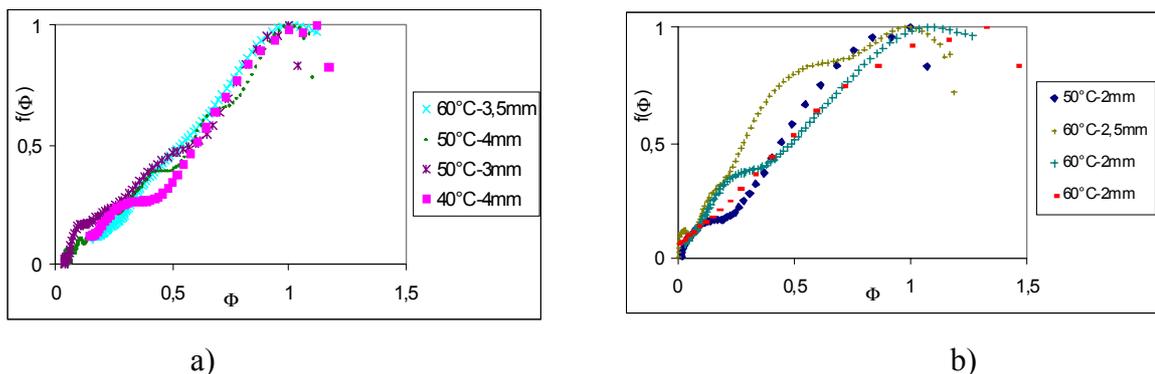


Figure 62.62.8 : convective drying rates, by varied diameters of *Spirulina* cylinders, at 60°C and 2,1m/s.

The comparative curves on figure 62.62.7 show that, for a temperature 50°C, the maximum drying rate decrease when the diameter increases. At 50°C, for 3 and 4 mm, the difference between the cylinders become lower and tend towards a limit. However, for 60°C, figure 62.62.8, we have not obtained these results, but an almost constant speed of drying. This can be due to reactions on the surface which return more quickly the transfer internal limiting for temperatures higher than 60°C. It is interesting to dry the Spirulina into thin cylinders, lower than 2 mm, obtained by extrusion. However, the effect of carrying drying with small cylinders, on the cells, must be studied.

### 2.4.3.1 Characteristic Drying Curve of cylinders

Applying the CDC method allows to regroup the curves for the Spirulina, for one dimension either in cylinders or in layers and for one type of drying. It is possible to represent the falling rate period for each type of drying and spreading out.



**Figure 62.62.9** : CDC for convective drying of cylinders,

- a) of cylinders 3-4 mm diameters
- b) of cylinders, 2mm diameters

The figure shows that it is possible to regroup the drying rate curves, by normalization.

The influence of the size appears clearly on these characteristic curves (CDC). The CDC are concave for convective drying of cylinders of small diameters.

The drying of cylinders of diameters of 3-4 mm led to linear function close to the diagonal. This means that the diffusion coefficient of water inside thick cylinders is

going to be very low at the end of drying [Moyne, 1989]. This dimension is thus not interesting on the level output.

#### 2.4.4 Others specific parameters

Face with the crust problems, we emitted initially, the assumption that the EPS can be responsible for this phenomenon. For a first analysis, we dried the sludge produced by the *Spirulina*, by supposing them rich in EPS, the content of EPS was not measured. However, the results are irregular, according to the type of spreading out (spreading out in cylinders or in layer), air velocity and temperature. The results are not similar. For more conclusive results, it will be necessary to analyze the composition of dried sludge.

## 2.5 Conclusions

### **Main results :**

- We have studied the drying of *Spirulina* (two types) coming from south of France.
- The result were poorly reproducible for the cylinders, but with good reproducibility for the layers
- The influence of temperature of air and of diameter is observable and generally coherent, higher the temperature and lower the diameter, higher the drying rate.
- A first drying rate appears, it have to be confirmed.
- For a given diameter of cylinders, the curves can be grouped under a CDC (characteristic drying curve). This curve is close to the diagonal. That means that the *Spirulina* behaves like a " thick " product, for which the diffusivity of the water through the medium is weak.
- The effect of the structure has not been theoretically studied. The structure can have a great influence on the facility with the phenomena of transport (between a stacking of springs or an arrangement of rods, the characteristics of the formed porous environment are very different). However, we dried two types of the *Spirulina*, one rather right and the other rather spiral, the drying rate curves are similar. Then the shape of the filament would not be a restrictive parameter.

- Problems during the work :
  - It happens that results are not in conformity with the precedents. That can be due to the precision of manipulation or errors of handling. But it seems that others parameters can intervene. Those clearly not identified. It can be the content of EPS, the culture parameters, or the crusting which intervenes more by high temperature (60°C) than at low temperature (40°C), or it can be the variation of the composition of the *Spirulina* during the growing time (batch reactor).
  
- **Possible continuation :**
  - It is imperative to establish the drying rate of the *Spirulina* cultivated in our laboratory. The batch culture is not enough reproducible of the culture under the real conditions (for example, increase in the concentration in EPS and sludges). The development of a new reactor should allow a more flexible culture (draining, adjustment of the luminous power).
  - It would be interesting to locate where is the resistance of heat and mass transfers, in the arrangement of the filaments or the membrane, to contribute to the choice of the process which makes it possible to decrease resistance to the transfers (to decrease energy consumption) and which preserves organoleptic and biochemical qualities .
  - It is necessary to characterize the shrinkage to model drying and identify the phenomenon of crust. This phenomenon also appears with infra red and in spray drying of it
  - Perform organoleptic quality measures
  - The continuation of the research on convective drying depends on the choice, studied in parallel, of the process

### **3. FREEZE DRYING OF SPIRULINA**

#### **3.1 Introduction**

Freeze drying is generally used to preserve a product which will have good qualities of taste, appearance and of dissolution. The aim of this part is especially to carry some experiments of the freeze drying of *Spirulina* to see if it will be interesting or not to develop this research for the spatial station and on which conditions this type of drying can be conceivable.

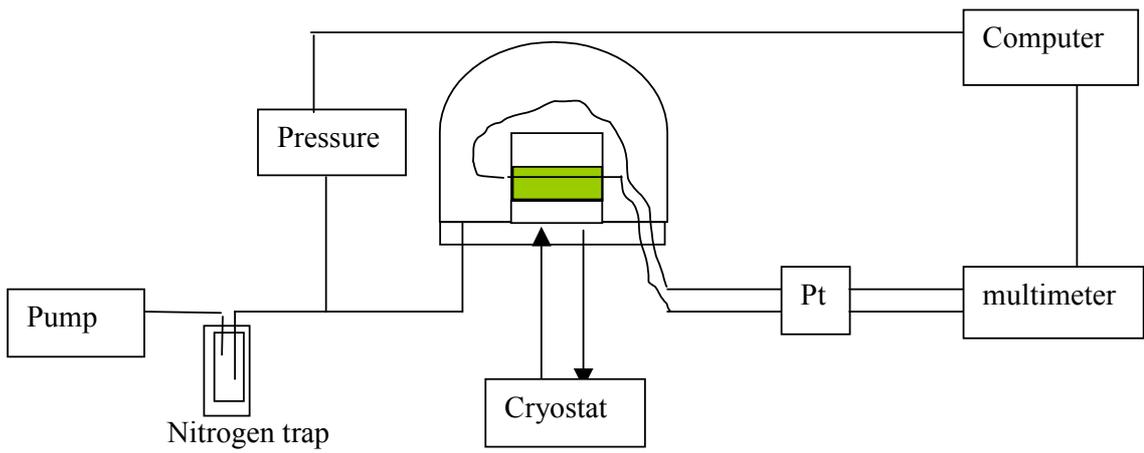
#### **3.2 Materials and methods**

Two installations of freeze-drying are usable at the laboratory for two very different capacities.

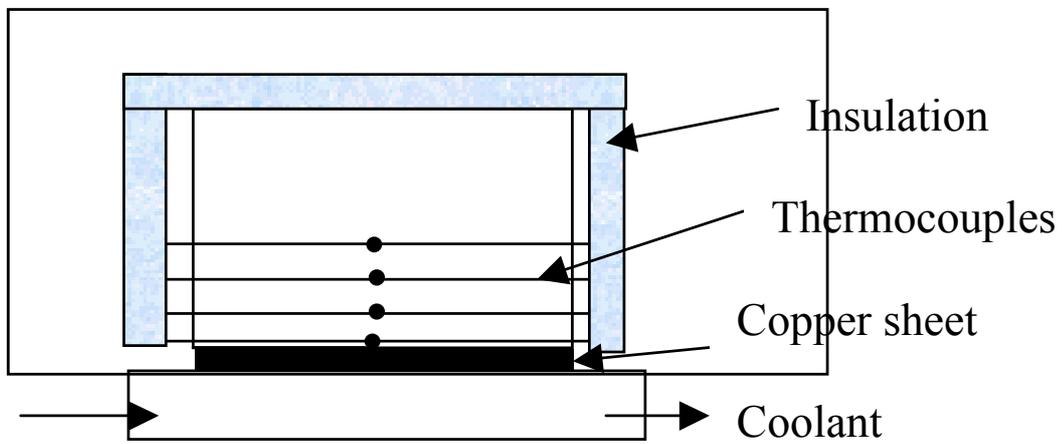
An apparatus was recently acquired, the lyophiliser (Freeze dryer SMH45 von USIFROID) which allows freeze-drying and conditioning directly batch sample. The installation that we mainly used is schematised below and makes it possible to measure the evolution of the temperature profiles during freezing phases and during sublimation of ice. The product is frozen on a circular copper plate, the thickness of the product can vary from few mm to 7-8 cm (approximately 150ml).

The product is frozen by conduction through the cooling of the copper plate, which is it even crossed by the cooling agent. It is also possible to freeze the *Spirulina* by *underwater block* setting in liquid nitrogen, then, to place it on the copper plate for the next phase of sublimation of water. The cooling rate can be varied, the minimal temperature of freezing too. On these two parameters depends mainly the size on the formed crystals.

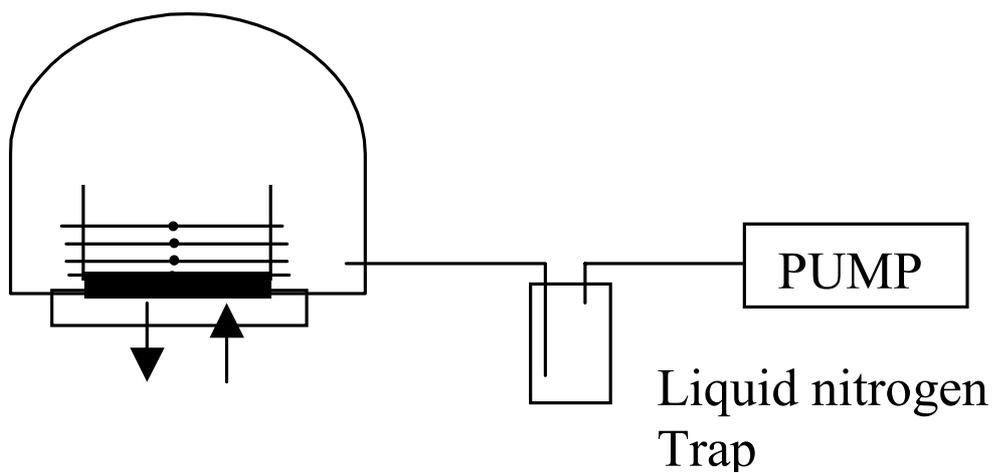
On this size of the crystals can depend organoleptic qualities and the solubility of the product. Times of freezing can be optimised to obtain a desired quality of product. Freeze-drying is carried out by vacuum setting (below 0,6 mbar) of the enclosure which contains the product, with a trap with condensation of the vapour (figure 1.c). The weight of the product is not recorded. The end of each stage corresponds to the end of the stages of temperatures.



a : Freeze dryer installation.



b : Cells for profile temperature measurement

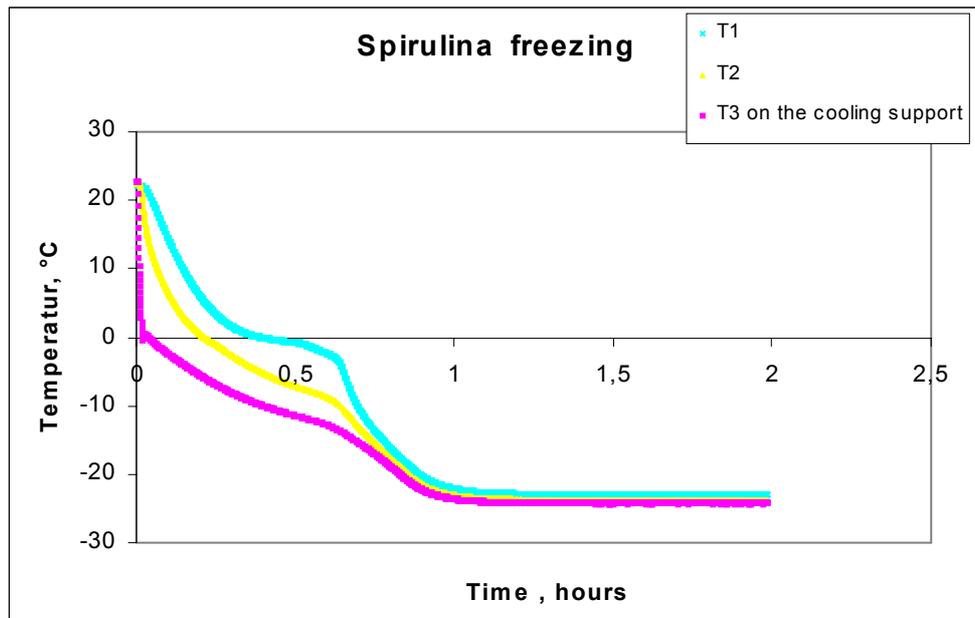


c : Vacuum installation

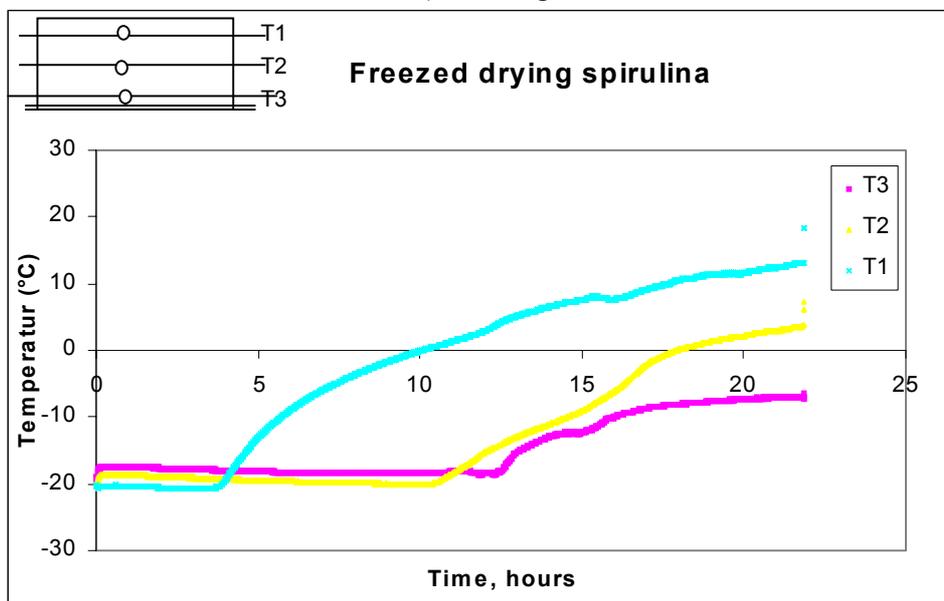
**Figure 62.63.1 a b c** : Freeze dryer installation and details.

### 3.3 Results :

We carried out tests (brutal freezing in liquid nitrogen or freezing with progressive low temperature), of which a test with the measurement of the profiles of temperature. The appearance is light, characteristic of the freeze-dried products. The very fragile product is easily friable and can thus be powder tiny room.



a) freezing



b) Vacuum drying

Figure 62.63.2: Temperature profile of *Spirulina* during freezing and vacuum drying.

To get temperature profiles, the experience need approximately 200 ml *Spirulina*, of which we added water. It is better to freeze-dry the not wring *Spirulina*, to get a freezing time and a freeze-drying time smaller than by wring *Spirulina*. It is necessary that the *Spirulina* is wet, that means with a moisture content higher than that of the beginning of convective or radiative drying, that means more than 7kg/kg.

The heat transfer passes especially through the water(more conducting than the biomass), then if the moisture content is too low, the heat transfer during freezing become down during freezing. In the same way, during sublimation, the draining of the vapour of the *Spirulina* through the pores is more difficult, because the pores are smaller if the water content starting is weaker. However, a high initial moisture content involves a higher energy expenditure.

In the case of the *Spirulina*, the optimum initial moisture content is has to determine.

The figure 62.63.2 show the results of the freeze drying of *Spirulina*. Freezing time is two hours with a temperature of -22°C.

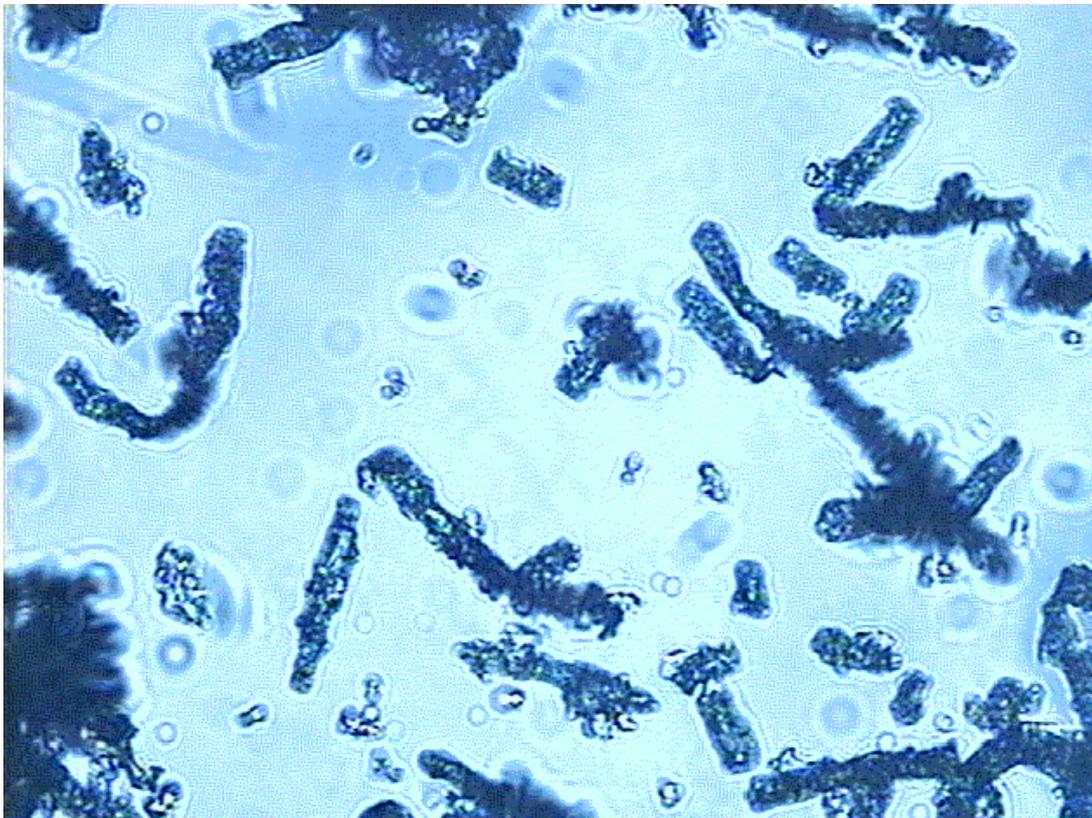


Figure 62.63.3 : photo of freeze drying *Spirulina* (nov 2001).

During the first phase (pink-mauve curve), the *Spirulina* is cooling, then during freezing close to zero degrees, the temperature is quite constant. Then the product is under cooling until the given temperature. During sublimation, the temperature remains constant, equal to the initial temperature, until total sublimation of the free frozen water. When the temperature of the *Spirulina*, placed near the support, increases, one can consider that all the free water is evacuated, it remains adsorbed water which can still be evacuated. For that, the sample is heated by conduction with the copper support by warm fluid circulation.

The photo n° 62.63.3 watch of the grains resulting from the powder obtained after freeze-drying. It seems that many filaments are cut .

### **3.4 Conclusions:**

#### **Main results:**

- The installation makes it possible to freeze-dry *Spirulina* and to get the temperature profiles;
- The freeze-drying of the *Spirulina* requires the optimisation of the initial moisture content;
- The photos show that the cells seem to be broken, this remain to be checked;
- The aspect of the freeze-dried piece of *Spirulina*, colour and texture, " is appreciated ".

#### **Possible continuation:**

The study of freeze-drying of *Spirulina* is possible

if:

- The fast dissolution which involves the release of the colour is not awkward;
- To bring a lyophilisator into operation is adaptable to the space conditions, in particular for the more significant need of energy on the ground than others processes of drying (cooling < 20°C + vacuum under P < 0,6mbar).

by:

- Determination of the optimal initial moisture content which makes it possible to reduce the drying time and the energy cost;

- The optimisation of the temperature and the freezing rate to get a given quality of *Spirulina* according to the identified need;
- The influence of these parameters on the solubility of the dry product, on the biochemical or structural characteristics of the *Spirulina*;
- The modelling of the heat and mass transfers during freeze-drying.

Studies in 2002 will make it possible to measure the influence of freeze-drying on the biochemical quality of the *Spirulina*.

#### **4. SPIRULINA PRODUCTION FOR HUMAN CONSUMPTION**

##### **4.1 Introduction**

Because the spatial stations are looking for drying with small production, the study concerns also the acceptability of *Spirulina* by the consumer. Then, it will be interesting to meet the actual producers (who dry *Spirulina*) and visit their installations, to discuss with them about quality, problems, etc... That was realized during a travel of two or three days in the region of Montpellier and Pont St Esprit.

The visit and the observation of the producers' work with *Spirulina* make it possible to raise elements of the know-how of the producers and eventually to create a link between the results of scientific search and those of the know-how.

We have visited 3 farms:

- Mr Philippe Callamand at Lodève (department Hérault, 34)
- Mr Robert Nogier at Pont St Esprit (department Gard, 30)
- Mr Jean Paul Jourdan at Mialet (department Hérault, 34)

And meet:

- Mr Ripley Fox at Saint Bauzille de Putois (department Hérault, 34)

The observations were centered on the harvest and on the drying of *Spirulina*. The results are gathered in the table and take again the expressions of the producers.

**The parameters :**

The measuring units are those of the operators. For ex: the capacity of the culture is expressed in m<sup>2</sup> exposed to the sun, not in m<sup>3</sup> . The indirect light source is always the solar radiation, through a plastic greenhouse.

**4.2 Results of the interview :**

**4.2.1 2.1. Culture :**

Farms	« Structure » of <i>Spirulina</i>	Production
Callamand	Wavy	4kg dry <i>Spirulina</i> /day
Nogier	Rather straight	
Vitar	Wavy	
Jourdan	Wavy and spiral	

**4.2.2 2.2. Drying - filtration :**

	Used drying method	Filtration	Pressing	Problems
Callamand	Forced convection	Manual , on sieve 120 yarn per inch 1 yarn = 20µm 1 pore = 20µm Pre layer of <i>Spirulina</i> .	Manual  know-how to preserve the broken of cells	
Nogier	Spray drying (failure)	1)Centrifuge filtration on sieve on 50 µm, 2)Without filtration		If the <i>Spirulina</i> is damaged, it passes through the sieves, and then come problem of joining on filter
Nogier	Forced convection	Without filtration	Without	
Vitar	Forced convection			
Jourdan	Forced convection	Manual filtration on 20-25µm filter	Soft pressing to allow evacuate the water without damage the <i>Spirulina</i>	Difficult filtration if too much EPS or "sludge"

Therefore for the pressing (which can be a centrifugal filtration), the producers point out that it intervenes together: the value of the pressure difference applied to the suspension and also the "speed" of filtration. The diameter of pores of the filter varies between 20 and 50 $\mu$ m.

It seems that :

- the *Spirulina* can constitute a pre- layer on the filter,
- the size (the width) of the *Spirulina* varies from one culture to another.

It is thus significant to define an average diameter (with standard deviation) of the used *Spirulina* and the possible variation of their width during time.

No producer rinses the *Spirulina* after filtration.

## **5. REFERENCES :**

Fornell A., Bimbenet JJ, Almin Y., Experimental study and modelization for air drying of vegetables products, *Lebensmittel Wissenschaft und Technologie*, vol.14, p96-100, 1980.

Moyne C., Roques M., Wolf W., A collaborative experiment on drying beds of glass spheres, *Physical Properties of Foods-2*, Elsevier Applied Science, p27-48, 1989.

Keey, R. B., and Suzuki, M., On the characteristic drying curve. *International Journal of heat and Mass transfer*. N° 17, p.1455-1464, 1974.