



NTNU – Trondheim
Norwegian University of
Science and Technology

Impact of Recipe Design on Kinetics of Heat Pump Drying of Protein Mixtures

David Illescas-Perez

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Supervisor: Odilio Alves-Filho, EPT

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Department of Energy and Process Engineering

MASTER THESIS

for

DAVID ILLESCAS PÉREZ

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**IMPACT OF RECIPE DESIGN ON KINETICS OF
HEAT PUMP DRYING OF PROTEIN MIXTURES**

Background

Drying is important in the animal feed industry because the dried product has high quality and long shelf life. However, drying is energy intensive taking up to twenty percent of the energy used in the industrial sector. The drawbacks of the available conventional dryers are negative effect on dried product quality and loss in energy losses with detrimental impact on environment.

Some of the current challenges in the pet food industry are developing mixtures that can be dried with acceptable quality. It is important to investigate alternatives to improve drying technologies for attaining higher water removal rates while being energy efficient and environmentally friendly.

These challenges will be studied using protein mixtures and applying a lab scale heat pump dryer. This drying technology has been developed at NTNU and it is energy efficient and environmentally-friendly. It has the added benefits of competitive costs while producing high quality dried protein mixtures.

The objectives of this work are to set up and execution of experimental trials in order to study the influence of drying conditions, protein mixture and geometric parameters on quality, properties and drying kinetics. The specific goals are:

- To study the effect of selected protein mixtures and conditions on quality, properties and kinetics during heat pump drying
- To record and analyze (interpretation) the experimental data
- To propose a protein mixture and related parameters related to better drying kinetics, quality and energy use.

The experimental design and characteristics of samples and drying condition

The experiments will be done using eight protein mixture samples and a lab scale heat pump dryer as listed in Table 1. The protein samples that are boiled are spherical of sizes from 1 to 12 millimeters while the standard protein samples are cubes with sides from 10 to 20 millimeters.

The experimental tasks are:

1. to review the related literature
2. to prepare, mix and the samples for drying
3. to perform tests according to the experimental design
4. to collect data on quality, properties and kinetics
5. to interpret and analyze the data
6. to recommend the treatment, geometry and condition for better drying and quality of the dried protein mixtures.



Table 1. Experimental conditions and characteristics of the products.

Test	SIZ	TEM	CAR	BOI	FRO
1	-	+	-	+	-
2	-	+	-	+	+
3	+	+	+	-	-
4	++	+	+	-	-
5	+	-	-	-	+
6	+	-/+	-	-	+
7	+	-/+	-	-	+
8	+	-/+	-	-	+

The designations in the table are: cube side of 1 to 12 mm (-), 10 mm (+) and 20 mm (++); temperature: -2°C (-), 20°C (+), 25°C (++) ,25°C (+++) and 35°C (++++); standard sample (-), added carbohydrate (+); unboiled (-), boiled (+); frozen (-) and unfrozen (+).

The Master thesis work comprises 30 ECTS credits.

A progress plan (*planned activities and scheduled progress*) shall be submitted to the responsible subject teacher/supervisors for comments within 14 days after the candidate has received the project description.

The work shall be edited as a scientific report, including a table of contents, a summary in Norwegian, conclusion, an index of literature etc. When writing the report, the candidate must emphasize a clearly arranged and well-written text. To facilitate the reading of the report, it is important that references for corresponding text, tables and figures are clearly stated both places.

By the evaluation of the work the following will be greatly emphasized: The results should be thoroughly treated, presented in clearly arranged tables and/or graphics and discussed in detail.

The candidate is responsible for keeping contact with the subject teacher and teaching supervisors.

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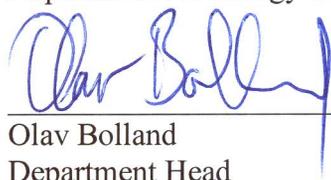
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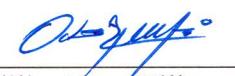
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An executive summary of the thesis including title, student's name, supervisor's name, year, department name, and NTNU's logo and name, shall be submitted to the department as a separate pdf file. Based on an agreement with the supervisor, the final report and other material and documents may be given to the supervisor in digital format.

Submission deadline: June 16, 2012.

Department for Energy and Process Engineering, 16 June 2012.


Olav Bolland
Department Head


Odilio Alves-Filho
Supervisor

Abstract

Drying is important in the animal feed industry because the dried product has high quality and long shelf life. However, drying is energy intensive taking up to twenty percent of the energy used in the industrial sector. The drawbacks of the available conventional dryers are negative effect on dried product quality and loss in energy losses with detrimental impact on environment.

Some of the current challenges in the pet food industry are developing mixtures that can be dried with acceptable quality. It is important to investigate alternatives to improve drying technologies for attaining higher water removal rates while being energy efficient and environmentally friendly.

Experiments in a laboratory scale heat pump dryer were conducted using protein mixtures. This drying technology has been developed at NTNU and it is energy efficient and environmentally friendly. It has the added benefits of competitive costs while producing high quality dried protein mixtures.

The influence of drying conditions, protein mixture and geometric parameters on quality, properties and drying kinetics were studied and discussed later on. A protein mixture and related parameters are proposed to achieve better drying kinetics, quality and energy use.

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Last but not least, I want to thank my family which gave me the opportunity to come to Trondheim to work on my thesis. It was a right decision as I could improve my knowledge in energy and impact on the modern Society in a Center of Excellence in innovative processes and heat pump technology.

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Notations

HPD	Heat Pump Drying
HPDs	Heat Pump Dryers
SMER	Specific Moisture Extraction Rate
q_c	Heat flux
n_w	Evaporation rate
h_g	Heat transfer coefficient
k_g	Mass transfer coefficient
p_v	Water vapor partial pressure
FD	Freeze Drying
VFD	Vacuum Freeze Drying
AFD	Atmospheric Freeze Drying
COP	Coefficient of performance
cond	Condenser
evap	Evaporator
CFCS	Chlorofluorocarbons
HCFCs	Hydro chlorofluorocarbons
X_{wb}	Moisture content in wet basis
X_{db}	Moisture content in dry basis
m_w	Mass of water in the product
m_t	Total mass of the product
m_d	Dry mass fraction of the product

T_{wb}	Wet bulb temperature
X_{cr}	Critical moisture content
ρ_b	Bulk density
ρ_p	Particle density
V_b	Bulk volume of the dried sample
V_{b0}	Bulk volume of the fresh sample
r_a	Rehydration rate
w_t	Weight of the sample at a certain rehydration time
w_d	Mass of the dried sample
PCFs	Proteins, carbohydrates and fats mixture
T_{ev}	Evaporation temperature
STD	Standard recipe
STD+CH	Standard recipe with added carbohydrate
u_{mf}	Minimum fluidization velocity
MC	Moisture content
XR	Moisture ratio
φ_{in}	Air relative humidity at the inlet of the drying chamber
φ_{out}	Air relative humidity at the outlet of the drying chamber

Chapter 1

Introduction

1.1. General drying

Drying is always one of the main food processing technologies widely used in the food industry to preserve food. The main objective of food dehydration is to remove water to a level at which microbial spoilage is minimized while extending the product shelf-life. Indeed, dried products kept under ambient conditions are also characterized by low water activity that inhibits microbial growth and undesirable enzymatic reactions. In addition, there is a significant reduction in mass and volume that also contributes to reduce the cost of packaging, handling, storing and distributing of foodstuffs without involving expensive cooling systems.

Drying is one of the most energy intensive processes that easily account for up to 15% of all industrial energy utilizations. The energy cost is determined by initial and final moisture contents, drying time, operating conditions, the scale of the process and the type of system (forced-air, drum, freeze and vacuum drying). A large fraction of energy is usually lost in industrial drying processes. Therefore, energy management is an essential part of drying process and efficient energy conservation contributes significantly to the overall operating cost [10].

Another important objective of a drying process is to handle difficult materials and to produce a dried product of desired quality at minimum cost and maximum throughput. Therefore, a drying system that is both energy efficient and preserves product quality is desired. This creates challenges for researchers, food industry and dryer manufacturers to develop new technologies to process difficult-sensitive materials and to supply final products with high quality and improved properties.

Product quality is the most important factor to be considered simultaneously with energy conservation. During drying, food materials undergo physical, chemical and biological changes that can affect some natural attributes like texture, color, flavor, and nutritional value. Therefore, an added objective of drying is to produce dried foods of good quality from a nutritional and organoleptic standpoint. Loss of nutritional quality is mainly due to the effect of temperature and dehydration on vitamins and proteins. Organoleptic quality is severely affected since texture, color and flavor are significant attributes used in judging the quality of dried foods [6].

Studies have been done about comparison of quality degradation between heat pump drying (HPD) and conventional drying methods for several materials and it was concluded that HPD offers products of better quality with less energy consumption. Quality parameters as color, density and shrinkage are influenced by the dryer type and dryer conditions such as temperature and relative humidity. Foods like fruits and vegetables consist of water, carbohydrate, lipids, vitamins and minerals. These compounds are easily modified in a high temperature drying condition and result in degradation of product quality and properties [26].

Thus, quality is highly affected by the drying temperature and higher product quality is achieved at lower temperature that leads to a highly porous structure of the product.

Recently, there has been a great interest in utilizing heat pumps in combination with dryers (HPDs) for drying fruits, vegetables and biological materials. The energy efficiency of conventional dryers is generally very low (35% or less) and HPDs consume 60 to 80% less energy than conventional dryers operating at the same temperature. Heat pumps have been known to be energy efficient when used in conjunction with drying operation due to the high coefficient of performance of the heat

pump. The evaporator and the condenser recover heat and reduce the energy consumption of the drying process [10].

One of the main features of the heat pump dryer is its ability to operate in a closed cycle with precise control over the thermal environment of the system that allows regulating the air temperatures at different stages of the drying process to attain the optimum operating conditions. Thus, it is possible to set different combinations of drying conditions, with temperatures in the range from -20 to 100°C, to maximize the performance of the system and improve product quality.

The theoretical maximum specific moisture extraction ratio (SMER) for conventional thermal drying is 1.55 kg/kWh (based on the latent heat of water vaporization at 100°C). A typical SMER value achieved by a heat pump is 3 kg/kWh, which compares very favorably with conventional convective drying, with values ranging from 0.5 to 1 kg/kWh. Thus, heat pump dryers are about 4 to 5 times as affective as traditional drying systems [24].

Therefore, product quality and cost are usually competing factors. The goal is to look for a favorable combination of cost, energy efficiency, quality, and price of the final product. It is important to investigate alternatives to improve drying technologies for attaining higher water removal rates while being energy efficient and environmentally friendly.

The objectives of this work are to set up and perform designed experimental trials applying a lab scale heat pump dryer and to study the influence of different protein mixture recipes, drying conditions and geometric parameters on quality, properties and drying kinetics.

1.2. Outline of the thesis

This thesis is divided in seven chapters that will be described in this section:

The first chapter gives an introduction about the topic and about the objectives of this work and thesis. It explains why drying is important in the industry and the reasons why the quality of the product, energy and environment conservation are the main factors to be accounted for.

In Chapter 2 is a literature review, and provides a summary of the importance of each quality parameter in the final dried product. It covers also a selection of experimental studies on heat pump drying published in the literature and the main conclusions achieved about quality of the product.

Chapter 3 covers fundamentals of drying, drying mechanisms and describes different drying technologies. It describes the advantages and limitations of heat pump drying and defines the dryer performance indicators.

In chapter 4 is on materials, measuring devices and methods and explains how the material was prepared and used in the experiments. Explanations are given on the drying system, the devices used to measure quality parameters and the oven drying method.

In chapter 5 describes the experiments and test conditions, experimental set-ups for each test as well as the parameters to be compared.

Chapter 6 covers the results, analysis and discussion, including the analysis of the drying kinetics and measured quality parameters. This chapter provides discussions about the best recipes and drying conditions for better drying and quality of the dried protein mixtures.

Conclusions and recommendations for further work are summarized in Chapter 7.

Chapter 2

Literature review

Research in atmospheric freeze drying combined with heat pumps has been and is still mainly investigated at NTNU-SINTEF in Trondheim. Several articles have been internationally published in this field by both organizations. This literature review gives a summary of the development in heat pump drying while keeping high quality of the dried products. Drying is done at different drying conditions that may affect positively or negatively the final product quality and properties. The main quality parameters are described next:

- Color

Color is an important quality attribute of dried foodstuffs and it is essential to maintain the original color in the dried product. The first quality judgment made by a food consumer or buyer is focused on visual appearance. Appearance analyses of foods (color, taste and texture) are used as a basis for food quality from the initial through the end of processing. Abnormal colors, especially those associated with deterioration, spoilage and poor eating quality, cause the product to be rejected by the consumer.

Therefore, the goal of this work is to measure and analyze color components in different protein mixtures before and after drying. Differences in color components between fresh, frozen and dried products should be as small as possible to guarantee the quality of the final product.

- Bulk and particle density

During drying the food products undergo deformations that can be characterized by changes in volume, shape, porosity, density and shrinkage. These modifications in the material structure are related to and can be determined by changes in bulk and particle densities. Then, their determinations are extremely important in terms of product quality and characterization of mass and heat transfer phenomena. Optimization of these phenomena taking into account the quality of the material or product and the cost of the processing is required for the development of drying technologies.

In the case of drying shrinkage is a consequence of the evaporation of solvent or moisture contained in the solid porous matrix. If the volume reduction is strictly proportional to the mass loss, the shrinkage is regarded as ideal. On the other hand, if the volume reduction is smaller than the volume of evaporated water, the drying operation generates an increase in the material porosity.

For these reasons, the goal of this work is to investigate and to draw conclusions the effect of drying operation and material geometry on these parameters and to recommend the best recipe and drying conditions for better quality for specific drying times.

- Rehydration

The rehydration characteristics of a dried product are used as a quality index and as indicators of physical and chemical changes during drying. It is related to shrinkage and porosity since when drying causes thermal damage to the product it will have higher shrinkage, and it will be less porous with lower capacity to absorb water.

Thus, the additional goal of this work is to study and identify what treatment, geometry and condition leads to a better rehydration capacity.

- Kinetics and drying curves

The kinetics and drying curves of a dried product are measured in terms of moisture content versus drying time. It is important that the product has higher changes in moisture content or higher moisture removal rates in order to reduce drying times, which are related to energy consumption. This is normally achieved by drying at higher temperatures. However, product quality decreases when drying at higher temperatures. Therefore, it is central to achieve a satisfactory solution both for quality and energy consumption.

To sum up, the goal of this work is also to study the effect of drying conditions, product shape and sample size on kinetics, drying curves and drying times. Quality parameters were also analyzed, discussed and conclusions were drawn for recommending which recipe, geometry and drying conditions leads to a better product quality for a given drying time.

There is no research published in the literature about heat pump drying of protein mixtures. Even though most publications on heat pump drying is from NTNU, there are extensive references on general drying. It is useful to examine publications on general drying of different products and to survey the drawbacks and advantages of conventional drying in foods such as green peas and other materials. Therefore, the relevant experimental studies of HPD have been selected and are described next:

The work done by Odilio Alves-Filho, Pablo García Pascual, Trygve M. Eikevik and Ingvald Strommen describes the drying of green peas in a fluidized bed heat pump dryer under atmospheric freezing and at medium temperature drying. Three different types of green peas with two diameters (8 and 10 mm) were used in the trials. The tests were carried out in combined temperatures, the first 9 hours was for freeze-drying followed-up by 3 more hours at 25°C [15].

In addition to the drying kinetics, different product parameters were measured before, during and at the end of the drying process. It was found that:

As concerned to the drying curves, the drying rate was slightly higher for the 8 mm green peas, which was the smallest diameter among the treatments. There was also a significant effect of temperature on the drying kinetics and, consistently, higher drying temperatures produce higher drying rates.

As related to color, the results indicated that atmospheric freeze-drying under temperatures of -5°C or -10°C produced dried green peas with original color. The sample dried at 25°C only had significant reduction in the amount of green color.

As connected to density, the samples dried at 25°C had higher density as expected since shrinkage or reduction in volume of the granules is higher at this temperature.

As concerned to rehydration, the ability to reabsorb water decreases inversely with the drying temperature. The 10 mm samples showed a maximum value at -5°C instead at -10°C , and decreased in the other drying conditions.

U. S. Pal, M. K. Khan, and S. N. Mohanty carried out experiments under controlled conditions for green sweet pepper in a heat pump dryer using drying temperatures of 30, 35, and 40°C . Fresh green sweet peppers were obtained from the local market and they were cut into slices of 5 mm thickness [20]. It was found that:

The drying curves evidently showed that the drying time decreases with increase in drying air temperature from 30 to 40°C . The moisture content of green sweet pepper slices was observed to decrease exponentially with drying time. Heat pump drying at 30°C took the longest time or had a lower drying rate.

The color results showed that chlorophyll content of the final dried product decreased with increase in drying air temperature, which might be due to the temperature sensitivity of this pigment.

The rehydration ratios were lower for product dried at higher temperature of 40°C . This might be due to the structural change in the product at higher drying air temperature that inhibits proper reconstitution.

O. Alves-Filho, T.M. Eikevik and S.V. Goncharova-Alves published a research article on Single and Multistage Heat Pump Drying of Protein. Seven isolated protein drying runs were performed using a laboratory single-stage atmospheric and multistage scale heat pump fluidized bed dryer. Runs 1, 3, 5 and 7 were single single-stage atmospheric freeze-dried using constant drying chamber inlet temperature below the protein's initial freezing point at -5°C . The drying time was 2, 3, 6 and 8 hours, respectively. Runs 2, 4 and 6 were two-stage dried, employing atmospheric freeze drying with reside times of 2, 3, and 6 hour promptly followed by evaporative drying at 25°C for 2 hours [16]. It was found that:

Moisture removal rate increased by shifting from single-stage lyophilization to evaporation. This means that two-stage drying with properly scheduled residence time leads to optimum drying rates, improved dryer capacity and enhanced product quality.

The higher brightness, neutral reddish and higher levels of yellow colors were obtained for two-stage dried samples. The results indicated that color components can be adjusted in single or two-stage drying with a specific scheduled of temperatures and residence times.

The measurements also showed that atmospheric freeze drying produces dried samples with low bulk density characterized by a highly porous structure. Sample bulk density dropped as residence time increased in single-stage and density reduction was more intense when atmospheric freeze drying was immediately followed by evaporative drying.

Finally, the protein powder reconstitution was generally associated with the level of shrinkage during drying. The results indicate that the single-stage dried samples had no shrinkage when the residence time at -5°C was less than 6 hours. Shrinkage in two-stage drying was higher than in single-stage atmospheric freeze drying.

Chapter 3

Fundamentals of drying

Air-drying is an ancient process used to preserve foods in which the material to be dried is exposed to a continuously hot stream of air causing moisture evaporation. This phenomenon involves simultaneous mass and energy transport.

3.1. Mechanisms of heat and mass transfer

Two transport processes occur simultaneously during drying [6]:

1. Heat transfer from the external surroundings to the surface of the food material being dried combined with heat conduction within the material.
2. Mass transfer from inside to the surface of the material followed by external transport of moisture to the surroundings.

Therefore, depending on food product and drying conditions, vaporization may occur either at the surface or inside the product.

The heat transport during drying is influenced by a variety of parameters. A procedure to improve the contact between the particles and the air is fluidization. Some advantages of fluid bed drying are high rates of heat transfer and moisture removal that leads to high thermal efficiency. Nevertheless, care should be taken to avoid damage to the drying product, which may occur in case of intensive fluidization and strong particle to particle collisions.

In the drying process, energy is transferred to the drying material by:

- Convection, when the hot air flowing through the material is used both to supply the heat for evaporation and to carry away the evaporated moisture from the product. Heat is transferred mainly by convection in over 85% of industrial dryers such as tray, fluid-bed and spray drying.
- Conduction, when the material is in contact with a hot surface as in the case in tray, drum or rotary and shelf-vacuum freeze-dryers.

Mass transfer from the product to the flowing air may take place by convection, due to the mixture psychometric differences at inlet and outlet. This includes for instance partial vapor pressure, temperature and relative humidity differences at the boundary layer in the air-product interface. Direct evaporation occurs when the vapor pressure on the surface is equal to the atmospheric pressure as in the case of vacuum-freeze drying.

Under convective drying, the boundary conditions for the heat flux q_c , and the evaporation rate n_w , can be expressed as:

$$\text{Heat transfer: } q_c = h_g(T_{sf} - T_g) \quad (1)$$

$$\text{Mass transfer: } n_w = k_g(p_{vsf} - p_{vg}) \quad (2)$$

where h_g and k_g are the heat and mass transfer coefficients, T is the temperature, and p_v the water-vapor partial pressure. Vapor pressure at the product surface can be evaluated from the sorption isotherm by

$$p_v = f(x, T)$$

Several technologies have been developed aiming at best products at the lowest cost, such as hot air drying and vacuum-freeze drying.

Thermal drying is often conducted at high temperatures. However, many agricultural and food materials are sensitive to high temperatures. Dehydrated products usually may present distinct losses in quality during processing. It is reported that the pigment and vitamin degradation rate increases as the drying temperature increases. Heated air drying is the lowest cost process. However, conventionally air dried products may not rehydrate satisfactorily because of structural changes in the product due to excessive thermal damage. During the last few years, emphasis has been placed on improving the rehydration characteristics and quality attributes of hot air dehydrated commodities. This may be done by changing process variables using pre-drying treatments and low temperature processes. Thus drying at low temperature to enhance the quality of food products has been a procedure with growing interest in recent years [25].

3.2. Vacuum freeze drying

Vacuum freeze-drying (VFD) accomplishes water removal by sublimation directly from the solid phase to the gas phase at very low temperatures and pressures. So the moisture is removed by direct sublimation. It is well known that VFD is an appropriate method for water removal from heat-sensitive materials and to obtain dried products of high quality as compared to higher temperature methods of food drying. It has the ability to retain original structure and color, negligible loss of nutrients and excellent rehydration capability due to the porous structure of the final product.

Conventional vacuum freeze-drying requires a longer drying time, leading to high energy consumption. This combined with a hermetic of the drying system leads to high capital cost. This is partly due to the poor heat transfer rate as the sublimation front moves from the exterior to the interior of the frozen material being dried. Vacuum freeze-drying cost can be 200-500% higher than that of medium temperature air drying in order to achieve the same final moisture content [2].

High value products such as pharmaceutical, biological and food-products, which are extremely heat sensitive, are conventionally vacuum freeze dried. Even though, VFD process is a well-known and established technology that has a long period for research and development, it is an expensive and thermally inefficient drying process. It has the advantage of very highly valued dried products, usually with preserved nutritional characteristic. Vacuum freeze-dried foods have a high porosity and low values of apparent density and variable color but enhanced aroma and taste. The other disadvantage is the high fixed or operational costs and, specially high energy costs. Thus it is normally used for sensitive materials that can be damaged or decomposed at high temperatures.

As a consequence, atmospheric freeze drying was developed in order to reduce the energy consumption and related costs while maintaining a high product quality and properties.

3.3. Atmospheric freeze drying

The newest technology applying atmospheric freeze drying (AFD) combines heat pump, fluidized bed and convective air flow for moisture removal. Drying is done in single or multiple stages and at temperatures below or above the material freezing point. The temperature can be set in the range of -3 to -10°C, hence much higher than values used in vacuum freeze drying. It is well known that lower air temperature at a fixed relative humidity or vapor pressure has low ability to remove moisture. In addition, lower air temperature also requires more energy for cooling and therefore reduces the SMER. It has been stated that drying at temperatures around -10°C is a viable solution considering quality and costs, although a faster drying process is desired. The advantages of the AFD process in comparison with VFD are [4].

- Low initial investment cost since expensive vacuum auxiliary equipment are not required. The drying chamber, loop, blower, connections valves and ancillary equipment near atmospheric pressure while VFD requires structural strength to withstand vacuum.

- The process can be designed for continuous operation with higher productivity and lower operating cost.
- Improved heat and mass transfer coefficients between circulating drying medium and the frozen or unfrozen material.
- High quality and homogeneous quality properties of the dried product with an increased retention of essential (volatile, aromatic and nutritional) compounds in foods.
- It is possible to minimize the product degradation caused by oxidation by applying inert gas drying environment such as nitrogen or helium.
- The application of a heat pump system and different intensification processes added to AFD can decrease both energy consumption and drying time.

However, there are several mutual limitations:

- Products with high internal resistance to mass transfer have much longer drying times for AFD as well as VFD.
- It is difficult to control the threshold residence time at frozen conditions.

Recently, there has been a significant growth in the potential market for heat pump dryers for drying fruits, vegetables and biological materials.

3.4. Heat pump drying

The main heat transfer mechanism in a heat pump dryer is convection, which means that heat is transferred by the convection from the air to the drying material. The HPD is suitable for drying not only solid products but also liquid, solutions and semi-solid products.

Heat pumps used in combination with dryers allow to set the temperatures between those of vacuum freeze-drying and warm-air drying. Also, freeze-drying with heat pump provides increased capacity, lower energy consumption and a high quality

product that is equivalent to that of vacuum freeze-drying. The main advantages of heat pump dryers are the ability to recover energy from the exhaust and to simultaneously control the drying air temperature and humidity. This creates the possibility of a wide range of drying conditions and product quality [26]. Energy consumption is reduced due to the high coefficient of performance of the heat pump dryer if properly designed for high thermal efficiency.

3.4.1. Principle of heat pump dryer

A HP drying process consists mainly of two systems: a HP system and a drying loop with a chamber. The main components of the HP unit are an evaporator, a condenser, a compressor and an expansion valve.

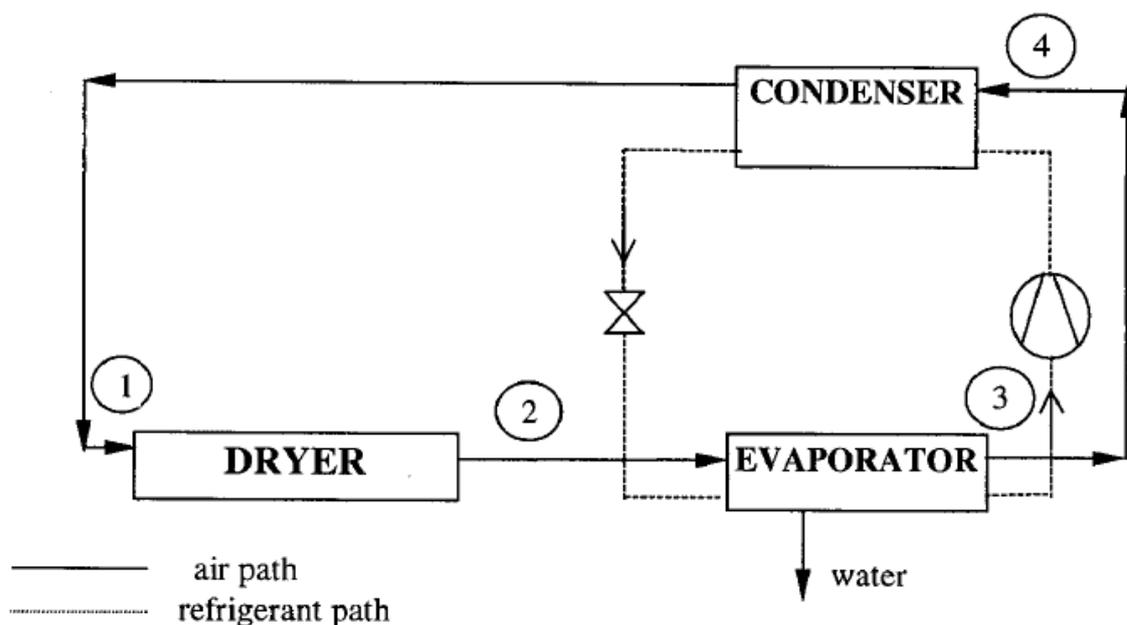


Fig. 3.1: Schematic representation of heat pump drying system [9]

Figure 3.1 represents a schematic layout of the drying loop and chamber integrated with various components of the HP system. The inlet drying air passes through the drying chamber at 1 and picks up moisture from the product. The moisture-laden air at point 2 is then directed to the evaporator. The moisture-laden air is cooled to dew point and dehumidified from point 2 to 3. Sensible heat removal occurs by air cooling to just above its dew point. Further cooling results in latent heat transfer and

water vapor condensation and removal from the drying loop. Then, latent heat of vaporization is absorbed by the boiling of the refrigerant inside the evaporator. This heat is recycled using the condenser. Finally, the drying air is re-heated to the desired temperature as it moves from point 4 to 1.

The coefficient of performance (COP) and the SMER are used as indicators for the dryer performance.

The energy efficiency of the heat pump is related to the coefficient of performance defined as the ratio of the heat absorbed in the evaporator or released by the condenser divided by the energy input to the compressor and blower. Then, the COP is given by:

$$COP = \frac{\text{Evaporator heat}}{\text{Work input}} = \frac{\dot{Q}_0}{\dot{W}_c + \dot{W}_f} \quad (3)$$

The maximum theoretical heat pump efficiency is given by the Carnot efficiency as:

$$COP_{carnot} = \frac{T_{cond}}{T_{cond} - T_{evap}} \quad (4)$$

The COP_{carnot} is ideal and cannot be physically accomplished but is used to compare a heat pump system with an ideal value. In practice, the actual efficiency of a heat pump is about 40 to 50% of the theoretical Carnot efficiency.

The performance of the dryer is defined by the specific moisture extraction rate (SMER) as follows:

$$SMER = \frac{\text{amount of water evaporated}}{\text{energy input to the dryer}} \quad (5)$$

The SMER (kg/kWh) depends on the heat pump dryer thermal efficiency, temperature and relative humidity of the air in the inlet and outlet of the evaporator and

condenser. Then, the SMER depends also these components operating pressure and temperature. Therefore, the SMER is:

$$\text{SMER} = \text{COP} * \frac{dx}{dh} \quad (6)$$

dh: specific enthalpy for heating and cooling of air [kJ/kg]

dx: difference in absolute humidity of the air into and out the HPD
[kg water/kg dry air]

3.4.2. Advantages and limitations

The advantages of heat pump drying are [9]:

- Energy efficiency is improved compared to a conventional dryer. The moisture-laden air leaving the drying chamber has a large amount of latent energy. This energy is recovered when the air passes through the evaporator and re-cycled back to the heat pump drying cycle. Low energy consumed is achieved per unit of water removed.
- Accurate and independent control of temperature, humidity and airflow rates are possible. This benefits heat-sensitive materials and produce better product quality.
- The dryer is versatile to dry different types of materials requiring operation in a wide range of drying conditions (typically from -20 to 100°C) and air relative humidities can be generated.
- HPD can be designed accommodating the present trend of using environmentally friendly fluids or natural refrigerants like ammonia, carbon-dioxide and water.

The limitation of this process is:

- Regular maintenance of the compressors, refrigerant filters, heat exchangers etc. are necessary to keep the dryer in optimum operating condition.

3.4.3. Drying kinetics of foods

Experimental data are usually represented as drying curves, which involves the plot of moisture content, X_{wb} , versus drying time, t . These data are the basis to obtain the drying rates (DR) and to plot the DR curves.

The moisture content of a product is expressed in wet or dry basis. The moisture content in wet basis (X_{wb}) is defined as the mass of water in the product (m_w) divided by the total mass of the product (m_t). The moisture content in dry basis (X_{db}) is the ratio of the mass of water to the dry mass of the product (m_d). Therefore, the equations are:

$$X_{wb} = \frac{m_w}{m_t} = \frac{m_t - m_d}{m_t} \quad (7)$$

$$X_{db} = \frac{m_w}{m_d} = \frac{m_t - m_d}{m_d} \quad (8)$$

The plot of moisture content versus time produces the drying curve as shown in Figure 3.2. The curve can be divided into three main periods:

- A-B: initial drying period.
- B-C: constant drying rate period.
- C-D-E: falling drying rate period.

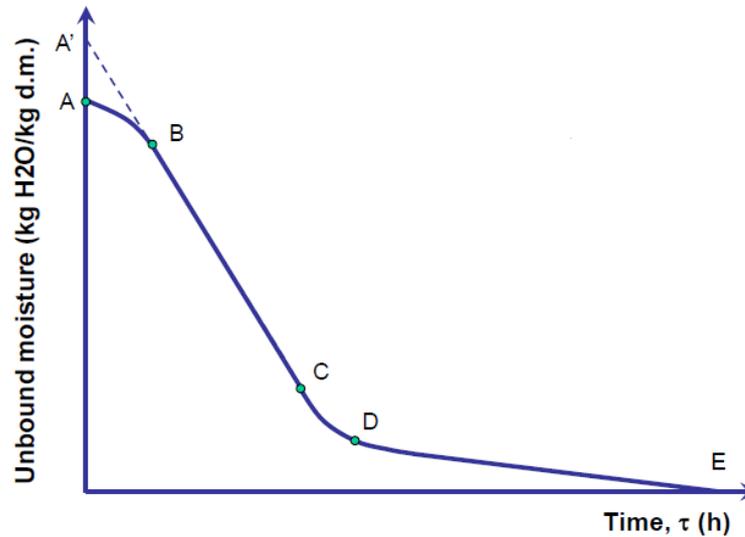


Fig. 3.2: General drying curve with different drying periods

Usually, in the initial drying period moisture evaporation may occur at the surface and the temperature evolves from the initial value to the wet bulb temperature (T_{wb}). In the constant-rate period, the surface is nearly saturated with free moisture and the material remains at constant wet-bulb temperature. The process of evaporation is controlled by the diffusion of water vapor through the boundary layer located at the air-solid interface. All heat transferred from the air is used for water evaporation from the surface. Most food products do not exhibit a constant rate period. This can be explained in terms of shrinkage, the time needed to reach the T_{wb} and the fact that the water is not always unbounded or the surface is not fully wetted. As a result of these effects, a pseudo constant-rate period can be observed in some materials and the drying rate depends on material and is lower than that of pure water.

The falling-rate period starts at the critical moisture content (X_{cr}). During this period the moisture content at the surface decreases and the surface temperature increases above the wet-bulb temperature. The consequence is the development of a non-flat internal moisture (and temperature) profile. The evaporation zone can move progressively from the surface towards the interior of the material. Depending on drying conditions and material properties, either or both internal and external resistances will play important roles in mass and heat transport.

As the moisture content decreases, the internal resistance for mass transfer increases and may become the prevailing step while the product temperature approaches the dry bulb temperature. In this case, a second falling rate period may be observed. The controlling mechanism is the rate at which moisture moves within the product, mainly by water vapor diffusion. The moisture content asymptotically approaches the equilibrium moisture content X_e and the relative humidity and temperature of the drying air [6].

3.4.4. Quality aspects and properties

The main quality indicators and properties investigated in the experiments carried out in this work are color, bulk and particle density, and rehydration capacity.

The color parameters for the color change of the materials can be quantified by Hunter L (whiteness/darkness), a (redness/greenness), and b (yellowness/blueness) values.

Bulk density (kg/m^3) is a property of powders, granules, solids and many other compounds and substances. In the case of drying wet and porous solids, it is defined as the total mass of the material divided by the occupied total volume:

$$\rho_b = \frac{m}{V_b} \quad (9)$$

The total volume includes the particles volume and the air held between them.

Volume change of each sample or bulk shrinkage percent is expressed as the percentage ratio of the initial to the final volume as follows:

$$\text{Shrinkage}(\%) = \frac{V_b}{V_{b0}} * 100 \quad (10)$$

where V_b is the bulk volume of the dried sample and V_{b0} is the bulk or initial volume of the fresh or frozen sample.

Particle density (kg/m^3) is independent of air space and of the degree of compaction of the batch containing the particle. The volume includes particle volume and internal pore volume. A number of “n” particle samples are considered and the mean or average value is determined by:

$$\rho_p = \frac{\sum_{i=1}^n m_{pi}/n}{\sum_{i=1}^n V_{pi}/n} \quad (11)$$

The rehydration rate (kg/kg), is defined as percentage moisture gain after immersing the sample in water. Then, it is calculated from the sample mass difference before and after rehydration as follows:

$$r_a = \frac{w_t - w_d}{w_d} \quad (12)$$

where w_t is the mass of the sample at a certain rehydration time, and w_d is the mass of the dried sample.

Chapter 4

Materials, Measuring devices and Methods

4.1. Materials

The material used in the experiments was a mixture of protein, carbohydrate and fat designated as PCFs. This mixture was blended and grinded in order to obtain a homogeneous mixture for each test. To evaluate drying performances three different recipes were used:

- Recipe 1: standard sample which is basically a original material (PFCs) acquired in the market. A batch of approximately five kilograms of this material was prepared.
- Recipe 2: the standard material containing extra amount of carbohydrate. A batch of three and a half kilograms of this sample was prepared.
- Recipe 3: the standard material which was pretreated by boiling process. A batch of two kilograms of this recipe was prepared.

Figure 4.1 shows the procedure for grinding and blending of the material for the mixtures.



Fig. 4.1: Preparation of the samples for drying

As for the standard recipe containing added carbohydrate, 3418.3 grams of the standard material was mixed with 240 grams of corn starch which contains 2% of total carbohydrate (supplied by Asia Foods and imported by As, Oslo). The amount of corn starch in the sample was 7% (240 grams of corn starch divided by 3418.3 grams of the standard material). As there is only 2% of carbohydrate in the corn starch, the total amount of carbohydrates in the mixture is 4.8 grams that represents 0.14% of total carbohydrates in the sample.

The standard protein material and the mixture with extra carbohydrate were distributed over two methacrylate sheets and two layers of 8mm thickness for both materials were obtained. After that both materials were frozen and kept in a freezer at a temperature of -25°C until the sample layers were completely frozen. The frozen layers were cut into cubes of 10x10x8 mm and 20x20x8 mm. This procedure was done to test different particle sizes and to analyze possible effects on drying rates and quality parameters.

Figures 4.2 and 4.3 show how the material was distributed and cut to the samples that were used in the experiments.



Fig. 4.2: Material layer distributed over a methacrylate sheet before freezing



Fig. 4.3: Detail on cutting of the frozen layer into 10mm long slab shaped particles

Finally, a batch of 2944 grams was separated to prepare the boiling sample during a period of about 15 minutes. The final mass after boiling was 1943 grams. Figure 4.4 shows the result of the pretreatment applied to produce the boiled sample from the standard material.



Fig 4.4: Boiled sample

To obtain data on particle size and distribution a sample of 10 grams of the boiled material was taken and separated into groups based upon individual diameter. The amount of particles on each group was quantified. Figure 4.5 shows the plot of the results on size distribution of the protein samples that were boiled and attained almost spherical shape with diameters from 1 to 12 millimeters.

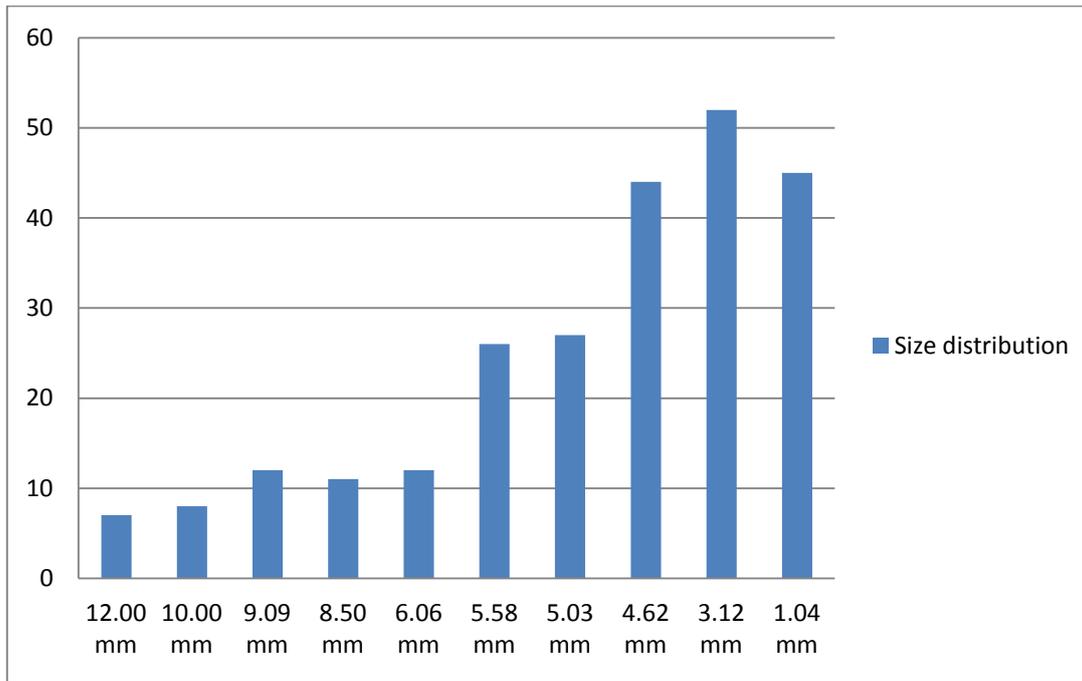


Fig 4.5: Size distribution of the boiled samples

4.2. Heat Pump Dryer

Figure 4.6 shows a simplified layout of the laboratory scale heat pump dryer developed at NTNU and applied for all experiments.

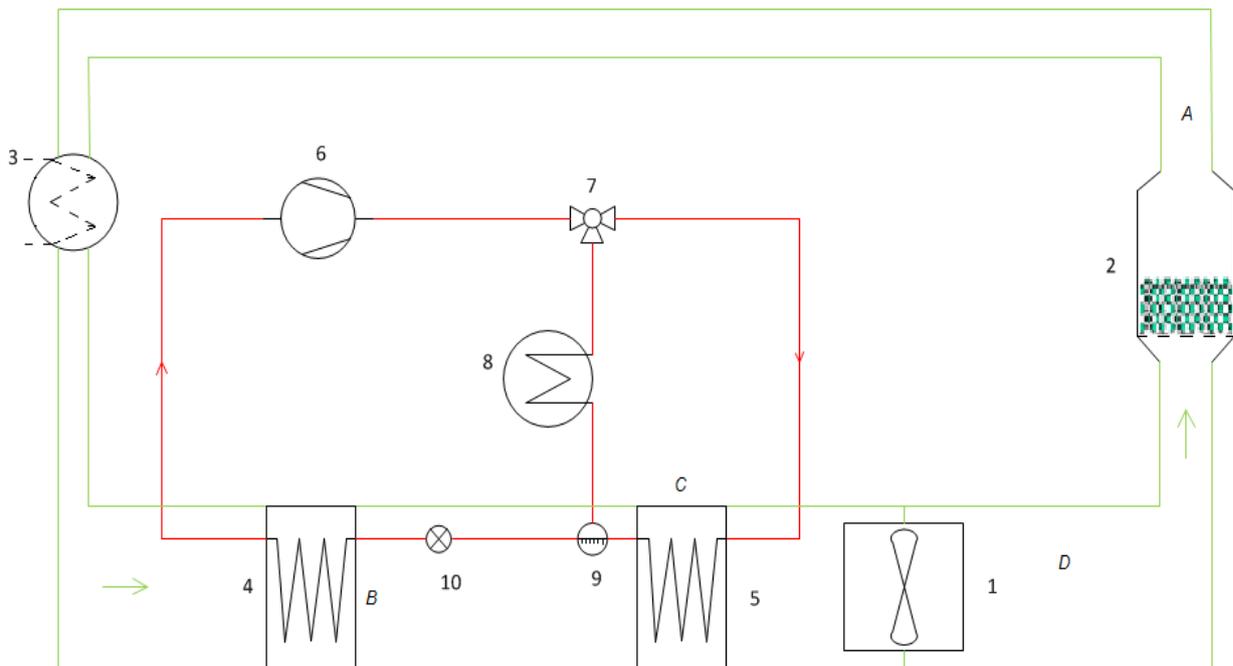


Fig. 4.6: Sketch of the Heat Pump Dryer

1. Blower	6. Compressor
2. Drying chamber	7. Three-way valve
3. Air filter	8. External condenser
4. Evaporator (Air cooler)	9. Receiver
5. Condenser (Air heater)	10. Expansion valve
Green line - air drying cycle	
Red line - heat pump cycle	

The operation starts as air supplied by the blower (1) flows through the bed of material placed in the drying chamber (2). The moisture-laden air is cooled and dehumidified by the air cooler (4). Then, the air is reheated by the air heater or condenser (5) and blower that also recycle it to the drying chamber. The refrigerant

boils inside the air cooler (4), becomes saturated or slightly superheated before being compressed (6). After removing the excess energy by the heat exchanger (8), the refrigerant is further cooled at the air heater (5). After that, the saturated or subcooled liquid refrigerant is throttled by the expansion valve (10). Finally, the refrigerant enters the evaporator repeating the heat pump cycle.

The operating temperature can be adjusted with the condensing and evaporating capacities. The regulation of the compressor capacity through frequency control of the motor s leads to the required air humidity. This setup adjusting the air drying temperatures from -20 to -60°C and relative humidities from 20 to 90%. These settings allow drying of heat sensitive materials either under convective or atmospheric freeze drying conditions. The possibility of sequencing these two operations is especially advantageous when drying food and bio-products because drying under frozen conditions results in minimal shrinkage and better quality, but at lower drying rates. Furthermore, by splitting of the drying process into FD by starting at temperatures around -5°C and finishing at $20-30^{\circ}\text{C}$ makes the process much faster. This also permits to control the quality parameters such as porosity, rehydration rates, product strength, texture, color and taste [24].

The flow rate of the air is adjusted for superficial velocity varying from 0.3 to 7.5 m/s by controlling the blower rotation.

As indicated in Figure 4.6 the fraction of humid air (A) leaving the dryer is cooled in an evaporator to a temperature T_B (B) close to evaporator temperature T_{ev} , which is below the dew point. Consequently, part of the moisture from this air stream is condensed and the latent heat is used to boil the refrigerant in the evaporator. The cooled and moisture-reduced air is mixed with the remaining fraction of humid air from the dryer (C). With the addition of the external work provided by the compressor, the working fluid attains higher pressure and temperature. It is then condensed in the condenser, thus transferring heat to the air mixture (C) and raising its temperature to T_D (D), which is below the condensation temperature T_C . This means that energy is recovered and used to re-heat the air with the energy for the working fluid flowing through the condenser.

Finally, the heated and dehumidified air is directed to the drying chamber. The thermodynamic cycle for the air stream in a Mollier chart is shown in Figure 4.7.

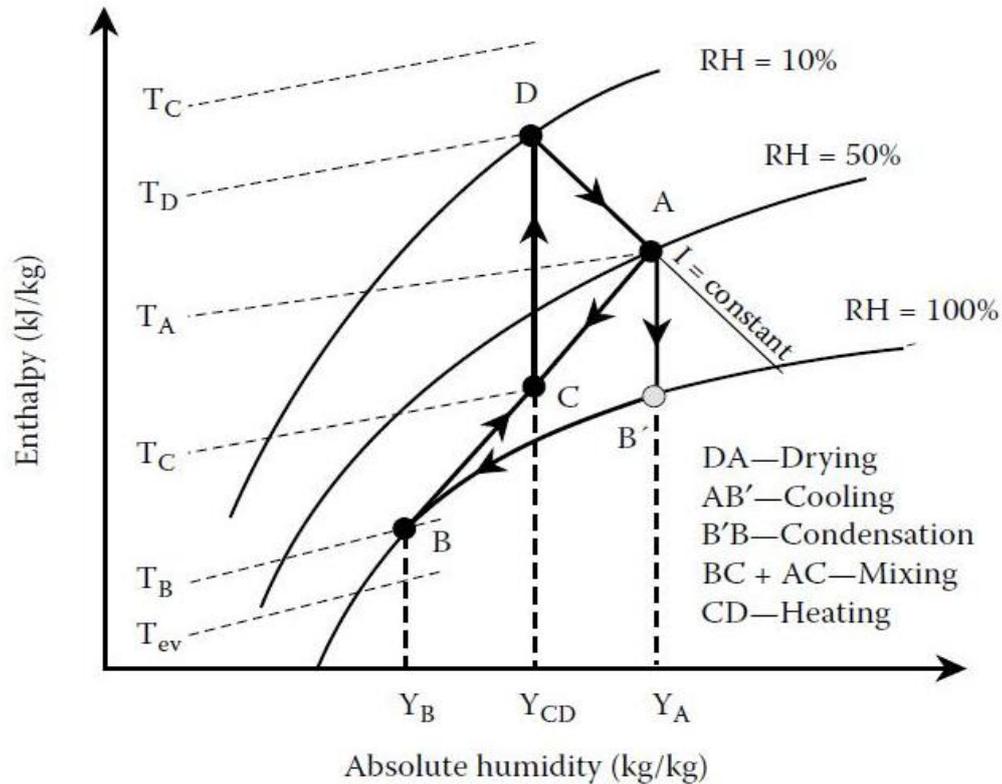


Fig 4.7: Mollier diagram for the air cycle of the drying process [24]

4.3. Drying cabinet and chamber

The drying cabinet and chamber made of plywood and isolated with styrofoam were used for the experiments as shown in Figure 4.8. The cabinet cross section was 80 cm by 80 cm and the height was 1.5m. The drying chamber was made of plexiglas and it was fixed in the drying cabinet by three pin lock-rotation mechanisms. The drying chamber has an internal diameter of 190mm and wall thickness of 5mm. Flexible hoses were installed in proper locations to connect the drying chamber to the heat pump air loop.

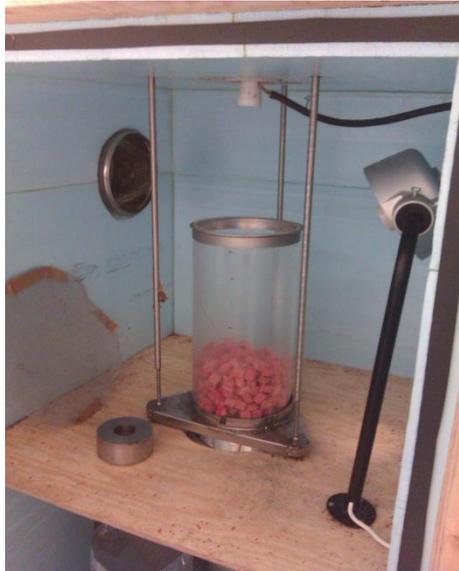


Fig. 4.8: The cabinet and drying chamber with the sample

4.4. Measuring devices

The relative humidity and temperature were measured and recorded in the three positions indicated by A, B and C in Figure 4.6. The air velocity and all data are continuously recorded using a data logger and a computer in order to check the operation conditions in the important points of the processes.

In order to determine how the change in the mass of the experimental samples with time during drying, the whole drying chamber containing the samples was weighted every 20 or 30 minutes time intervals. The scale used was the model “Mettler Toledo XP 600 2M Deltarange” with accuracy of 0.1 grams.

The mass measurements allow calculating the moisture content at each interval, based on the initial and final moisture of the samples that were measured using the moisture analyzer “Mettler Toledo HB43-S”. Both the calculated and measured moisture content values were compared in order to assure reliability of the experimental procedure and to validate that satisfactory results were achieved.

Another important data is bulk density which was measured for the initially frozen and the final dried samples using the standard mass and volume method. This

was done by filling a graduated cylinder with an amount between one or two liters of the material and measuring the net mass of the sample using the scale with an accuracy of 0.01 grams. Bulk density was calculated as the ratio between the mass of the sample and its volume accurately given by the graduated cylinder and scale. In some cases, initial densities were calculated by measuring the bed height of the samples in the chamber, the chamber diameter and calculating the volume by

$$V = \pi D^2 * h \quad (13)$$

The particle densities were measured by taking ten samples of the material and measuring length, height and width for each particle using a caliber and use this data to calculate its volume. A mean value of the ten particle volumes were obtained and then it was also calculated a mean of the mass measured for the ten particles, calculating particle density as the mass dividing by the volume.

A caliber was also used to determine sizes of the spherical protein samples that were boiled.

The color is an essential value for the evaluation of the frozen and dried product quality. The samples were grinded in a mortar to obtain a smooth and uniform sample. Then the color components of the samples were measured in terms of brightness (L), red-green (a) and yellow-blue (b) using a color meter type “X-Rite 948 spectrodensitometer”.

4.5 Drying oven method

The moisture content of the different samples was measured using a Heraeus shown in Figure 4.9. The oven was set and controlled at a temperature of 130°C for 16 hours. The final moisture content values allow comparison with the final values calculated and measured with the moisture analyzer and quite similar results were obtained.



Fig. 4.9: Oven used to determine the moisture content of the samples

To carry out this method, firstly it was prepared a representative portion of the material to be tested (between 8 and 9 grams) which was measured using a scale model “Mettler Toledo PM1200” with accuracy of 0.001 grams.

Then the mass of the test sample is determined and recorded as “wet mass”. The sample was loaded into a tared aluminum container. In this way, the mass of the container and of sample were continuously measured and differences calculated as required to obtain the changes in moisture content.

Finally the samples were dried to constant mass at 130°C. It is considered that constant mass is achieved when less than 0.1% of the test sample wet mass is lost during an additional exposure to the drying process. The nearly constant mass was observed in an overnight period of 16 hours. By removing the samples from the drying oven and cooling them to room temperature, it was possible to measure the mass of the test sample and record it as “dry mass”.

The moisture content of the test samples was determined according to the definition of moisture content in wet basis:

$$X_{wb} = \frac{m_w}{m_t} * 100 = \frac{m_{wet} - m_d}{m_d + m_w} * 100 \quad (14)$$

where the mass of water in the sample (m_w) is calculated by subtracting the dry mass to the wet mass, and the total mass (m_t) is the mass of water plus the dried mass.

4.6 Rehydration measurements

Reconstitution of dehydrated products were performed by immersing an amount (usually two cubes for 10mm particles and one cube for 20 mm ones) of dried samples into hot water at constant temperature of 38°C controlled by a electrical heater and a thermometer. The particles remained immersed for 25 or 30 minutes depending on time needed for the samples stop changing the mass within an accuracy of 0.1 g. This was achieved at about 5 minutes time interval, after which the samples were drained over a paper-filter for about 1 minute. Then, the sample was quickly drained with the paper towels in order to eliminate the surface water and then the mass determined using the scale. A general view of the devices used for these measurements is shown in Figure 4.10.

The rehydration capacity (kg/kg) is described as percentage water gain and it was calculated from the sample mass difference before and after rehydration as follows:

$$r_a = \frac{w_t - w_d}{w_d} \quad (15)$$

where w_t is the weight of the rehydrated sample measured at each interval and w_d is the mass of the dried sample. The samples were placed over a paper-filter whose mass was also measured.



Fig. 4.10: A view of the devices used for rehydration measurements

Chapter 5

Experiments and test conditions

A number of studies have been made to investigate the influence of process variables on the drying kinetics. The variables taken into consideration were the composition and properties of the samples such as characteristic size, initial state and air temperature, flow, etc. The drying experiments were carried out at three levels of the characteristic sample size (cubes of 10 and 20 mm, and size distribution of 1 to 12 mm for the boiled samples), three different recipes (standard material, standard with added carbohydrate and boiled samples), two initial states (frozen or unfrozen) and at five levels of air temperatures (-2, 20, 25, 30 and 35°C).

Tests were done either in single or two-stage drying. The two-stage drying mode combines atmospheric lyophilization immediately followed by evaporation at temperature higher than the material freezing point.

The experiments were performed in fixed bed by controlling the drying air just below the incipient fluidization. The material is placed in the chamber having a perforated bottom plate. Air flows through the product and the air velocity is kept in a range that the particles are not moving by the air.

Eight experiments have been carried out in batches divided in three categories (A, B or C) as a function of the material tested (three different recipes) and different drying conditions (temperature, material size and state). The details and experimental conditions of the drying runs in the heat pump dryer are given in Table 5.1.

Run	Recipe	Temperature (°C)	Time (min)	Initial state	Particle size (mm)
A1	Boiled	20	390	Frozen	1 to 12
A2	Boiled	20	390	Unfrozen	2 to 12
B1	STD+CH	20	390	Frozen	10
B2	STD+CH	20	390	Frozen	20
C1	STD	-2	360	Frozen	20
C2	STD	-2/35	120/140	Frozen	20
C3	STD	-2/30	240/140	Frozen	20
C4	STD	-2/25	360/140	Frozen	20

Table 5.1: Experimental design and conditions for all test runs

In the first batch it was tested a boiled protein mixture considering two different states: frozen (A1 test) and unfrozen (A2 test) material. An amount of 875.9 grams of the boiled material was used and loaded into the drying chamber to be dried in single-stage drying using constant drying chamber inlet temperature of 20°C with a residence time of 390 minutes and at a blower air flow set point of 3.6. Because of the small size of the boiled particles, losses through the perforated plate in the chamber at the end of both drying tests were 68.1 and 48.2 grams, respectively. The effect of the initial sample state on the drying kinetics and quality parameters will be analyzed in the next chapter.

In the second batch of experiments, a protein mixture with a specific amount of added carbohydrate was single-stage dried with a residence time of 390 minutes. The tests were carried out considering 854.5 grams approximately of this material and they were dried at 20°C at a blower air flow initial set point of 5.15. This value was lowered during the drying process based on the minimum fluidization velocity (U_{mf}) that dropped slightly during the tests. The air velocity was lowered to 4.8 m/s just below the

U_{mf} to achieve drying at static bed. Two experiments were done under these conditions for initial frozen material considering 10 mm (B1) and 20 mm (B2) cube sides in order to compare drying at different particle sizes. Each test had initial bed height of 45 mm and 58 mm which corresponds to an initial volume of 1.275 liters and 1.644 liters, respectively.

For both first and second batch of experiments, determination of drying curves was done by measuring the mass by a digital scale initially and during drying at time intervals of 30 minutes. The net mass of the material was obtained by difference from the mass of the drying chamber containing the material and the mass of the empty drying chamber. The drying chamber containing the material was taken out the cabinet for each of these periodic mass measurements as quick as possible for rapid recovery of the set conditions inside the drying cabinet.

The third series of batch drying experiments was a combination of a first drying stage and a second drying stage. For the first stage, a batch of 1990.4 grams that corresponds to a volume of 4.5 liters of the standard protein mixture was dried at a temperature of -2°C (test C1). Cube particles with sides of 20 mm were tested for 6 hours at a blower initial set point of 7.5. Every drying time of 2 hours, a certain amount of the drying product was taken away from the drying chamber and keep in the freezer to be dried in a second stage. As a result of this first stage, three different samples of the initial material were obtained after a drying time of 2 hours (test C2), 4 hours (test C3) and 6 hours (test C4). These samples were dried in a second stage for 2 hours and 20 minutes at a drying temperature of 35°C , 30°C and 25°C , respectively. The net mass of the product was measured during the whole drying process at time intervals of 20 minutes. The mass measurements required removal of the drying chamber from the cabinet but the air temperature and velocity were re-established to set points quickly.

Also, for every drying test the air relative humidity, temperature and air velocity were continuously recorded using a data logger and a computer.

Before and after drying tests, a sample (one cube) was taken for measurements of initial and final color, moisture content and particle density measured using either frozen and dried products. The determination of bulk density was carried out at the

ambient temperature of the laboratory that was constant at about 22°C. Rehydration measurements were done at the end of the experiments following the procedure described in the previous chapter.

Chapter 6

Results, Analysis and Discussion

To identify the recipes and process conditions lead to better drying rates and quality dried products, it is necessary to compare drying times, quality parameters and properties of the dried protein mixtures.

6.1. Drying kinetics

The drying kinetics obtained and curves were plotted as moisture content versus drying time. The values of moisture content were calculated from the change in the mass of the product weighed at 30 minutes time intervals for the first drying stage and 20 minutes for the second stage. The total mass of the product was calculated by subtracting the mass measured from the mass of the empty drying chamber. The initial moisture content in wet basis $(X_{wb})_0$ was measured using the moisture analyzer. From this value on, the fractions of dry matter and water were determined and used to calculate the moisture content at each interval. The procedure is:

$$m_t = m_{DC+S} - m_{DC} \quad (16)$$

$$m_d = m_t - m_w = m_t - m_t * (x_{wb})_0 = m_t(1 - x_{wb}) = const \quad (17)$$

This procedure allowed to obtain the drying kinetic curves that were plotted based on the moisture contents calculated for the given drying times or time intervals.

$$x_{wb}(\%) = \frac{m_w}{m_d} * 100 \quad (18)$$

The final moisture content of the product was obtained for the final time or the last interval. The calculated value was compared with the final moisture content measured with the moisture analyzer at the end of each test in order to verify the validity of our calculations.

The drying kinetics resulting from the different recipe mixtures were done considering different temperatures and sample sizes and will be now discussed. Moisture content and drying rates are all important indicators for the effectiveness and performance of the drying process. The drying rate is related with the moisture content.

As each test run has different initial moisture content and moisture ratio (XR) that were represented and compared for the different tests.

Moisture ratios are obtained by calculation as the difference between the moisture content at each interval and the final value divided by the difference between the initial and final moisture content:

$$XR = \frac{X - X_f}{X_0 - X_f} \quad (19)$$

As for the final moisture content approaches an equilibrium value for relatively large drying time. The minimum final moisture value for every test has been taken into account and the analysis was assumed that the final or equilibrium moisture content, at the end of each drying period, approached 4% wb.

Table 6.1 shows the temperature and relative humidity average values for each run at the inlet and outlet of the drying chamber.

run	inlet (A)		outlet (B)	
	T [°C]	φ_{in}	T [°C]	φ_{out}
A1	20.6	11.2	19.8	14.8
A2	20.7	10.8	19.6	13.2
B1	20.3	11.4	20.1	12.4
B2	19.9	10.9	20.0	12.3
C1	-1.9	35.4	-0.5	35.6
C2	34.5	8.0	33.3	8.8
C3	30.0	7.1	29.7	7.6
C4	25.0	8.7	24.7	9.2

Table 6.1: Specification of the experimental conditions

Table 6.2 shows the initial and final moisture content values for each test to have an overall view of the moisture content changes for different recipes and drying conditions.

Experiment	Temperature (°C)	Initial MC, X_{db} (kg/kg)	Final MC, X_{db} (kg/kg)
A1	20	71.41	8.64
A2	20	68.33	6.69
B1	20	44.71	22.17
B2	20	44.71	28.65
C1	-2	62.81	51.51
C2	-2/35	62.81/57.61	42.38
C3	-2/30	62.81/54.19	38.72
C4	-2/25	62.81/51.51	37.64

Table 6.2: Summary of moisture content and drying runs

Final moisture content values were also calculated for each test by applying drying oven method to compare these values with the ones obtained in the heat pump dryer. An amount between 2 or 3 cubes (9 grams) of each sample were prepared and dried in an oven at a temperature of 130°C. Samples of the standard recipe dried at -2°C were not available and could not be measured because all the material was used for the second drying stage. Table 6.3 shows the moisture content values obtained from the oven method as well as the values obtained by using the heat pump dryer.

Experiment	MC, X_{db} (kg/kg) Heat Pump	MC, X_{db} (kg/kg) Oven Method
A1	8.64	8.998
A2	6.69	6.919
B1	22.17	23.611
B2	28.65	30.779
C2	42.38	35.445
C3	38.72	37.216
C4	37.64	37.633

Table 6.3: Moisture content values obtained for two different drying methods

Final moisture content values obtained for samples dried in the oven are quite similar to that measured during the experiments in the heat pump dryer. Slightly higher values are normally achieved in the oven except for the test where a second drying stage was applied.

Therefore, moisture removal rates are equivalent to those ones obtained in the heat pump but quality parameters analysis should be carried out in order to validate this drying method to be accepted in the industry.

Figure 6.1 shows the plots of moisture ratio for the five tests involving single stage drying. Each test has been run considering different mixture recipes, drying temperatures and sample sizes. This experimental design allows studying the influence of these variables on the drying rates.

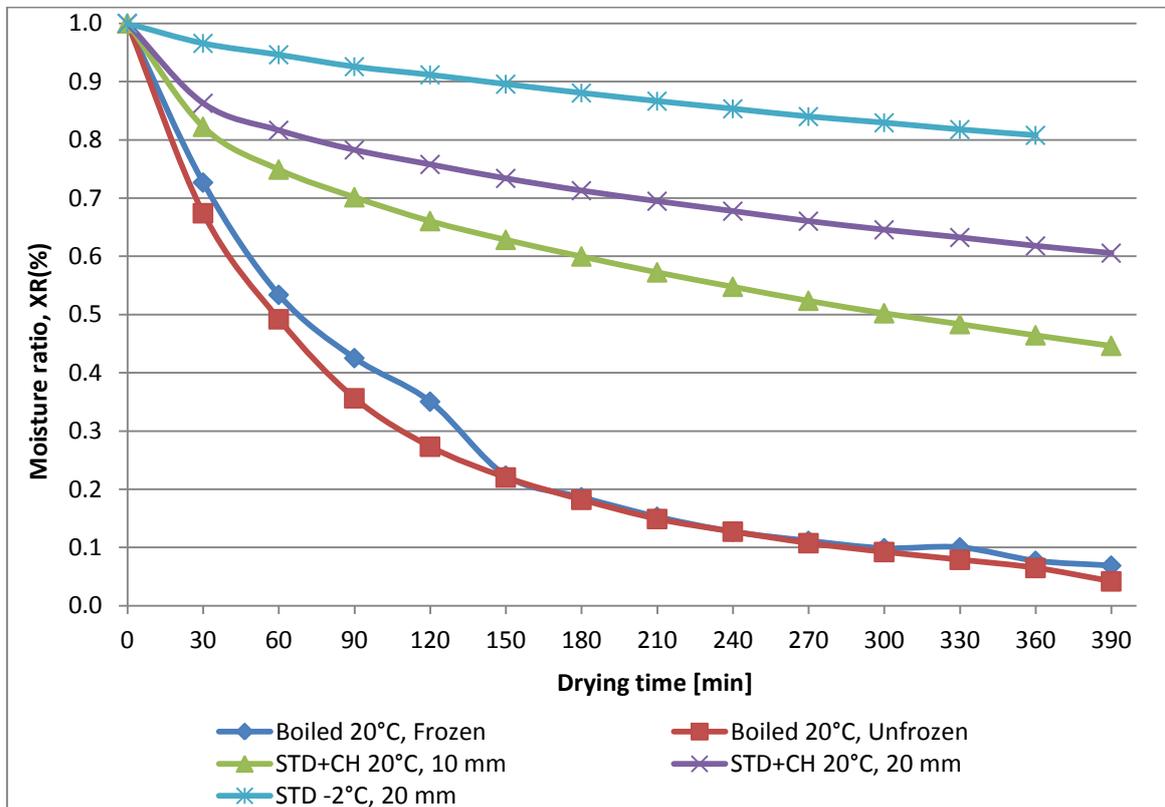


Fig 6.1: Moisture ratio for different mixture recipes and the drying conditions

The drying rate of food materials generally decreases as drying progresses. Figure 6.1 shows that mass transfer improves as the air temperature increases. For similar materials, the drying rate is lowest for the standard sample dried at -2°C and highest for the sample dried at 20°C . In other words, higher moisture removal rates are achieved at higher temperatures.

The results also show a rapid increase in drying rate with reduced size or dimension. The smallest particles are the boiled ones with a size distribution from 1 to 12 mm and they have the highest drying rates while the lowest moisture removal is for standard 20 mm particles. Thus, the drying time increases and the rate drops as sample size or thickness increases.

The final moisture ratio difference between the standard 20 mm sample and the standard 20 mm sample with added carbohydrate is about 0.2 while between standard sample and boiled sample is about 0.74. Standard 20 mm samples when operated with drying temperature at -2°C took 360 minutes to reduce the moisture ratio from 1 to 0.81 while standard 20 mm samples with added carbohydrate dried at 20°C took 60 minutes to achieve the same value. Boiled frozen samples dried at 20°C took less than 30 minutes.

Thus, the highest moisture removal or drying rate is for the boiled protein mixture dried at 20°C followed by the standard recipe with added carbohydrate dried at the same temperature.

The results of this study demonstrate that pretreatments such as boiling may significantly increase drying rate, and therefore decrease drying time of material for a given final moisture content.

The moisture contents versus the drying times for the single stage drying tests as described before are plotted in Figure 6.2. The aim is to compare the differences the initial moisture contents and effects on the drying curves for the different mixture recipes tested.

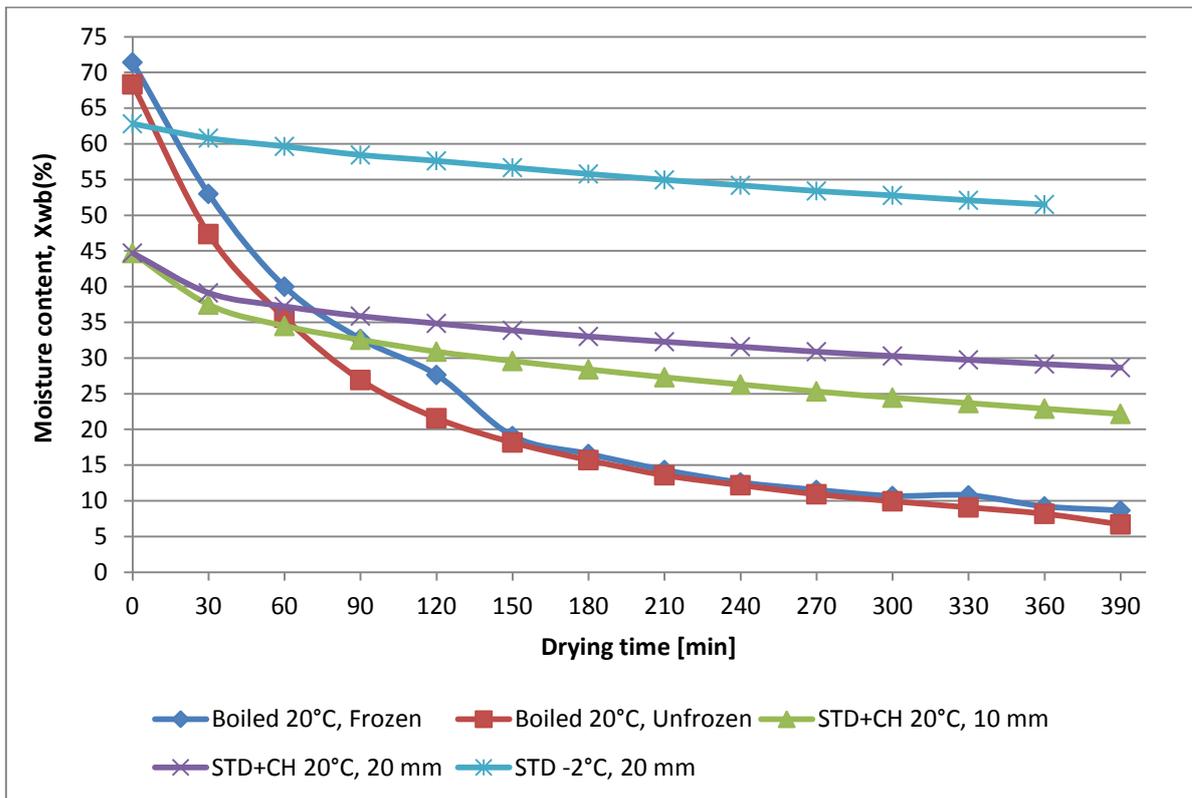


Fig. 6.2: Drying kinetics for different recipes and drying conditions

The initial moisture content for the standard recipe is 62.81%. Application of 15 minutes hydro-thermal treatment (boiling) leads to a higher moisture content in the sample (70% approximately). The addition of a certain amount of corn starch (moisture content around 15%) to the standard sample reduces the initial moisture content down to a value of 44.71%.

Analysis was made on how moisture content was affected by material initial state that was either frozen or unfrozen. The comparison for the boiled samples is shown in Figure 6.3.

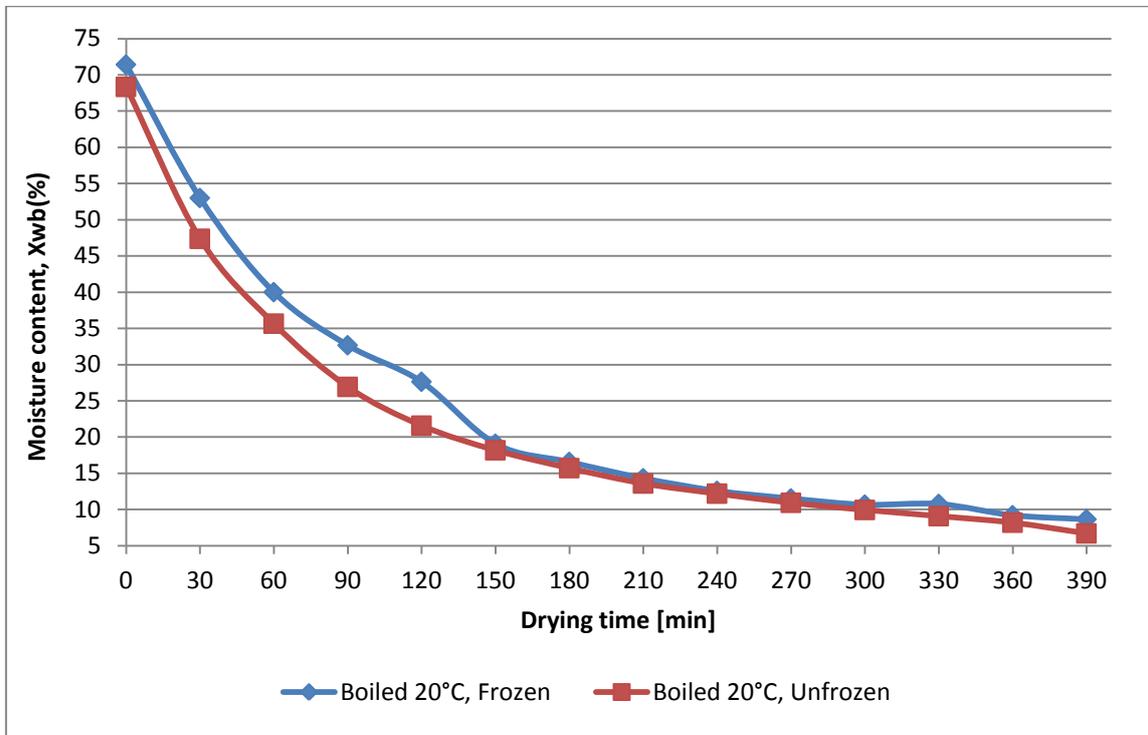


Fig. 6.3: Drying kinetics at 20°C for the boiled samples

For this case, the moisture content at each interval was calculated based on the final moisture content measured in the analyzer.

It is showed that the drying rates for the unfrozen material are slightly higher than for the frozen material. The reason may be that the energy provided in the drying air was used to heat up the grain to the drying temperature. In other words, the unfrozen material dried quicker because for the same drying temperature and particle size the initial drying period to preheat this sample is shorter because the surface temperature is closer to the drying temperature.

In order to analyze how moisture content is affected by size, tests were done with 10 to 20 mm cubes using the standard samples with an extra amount of carbohydrates. The results are plotted in Figure 6.4.

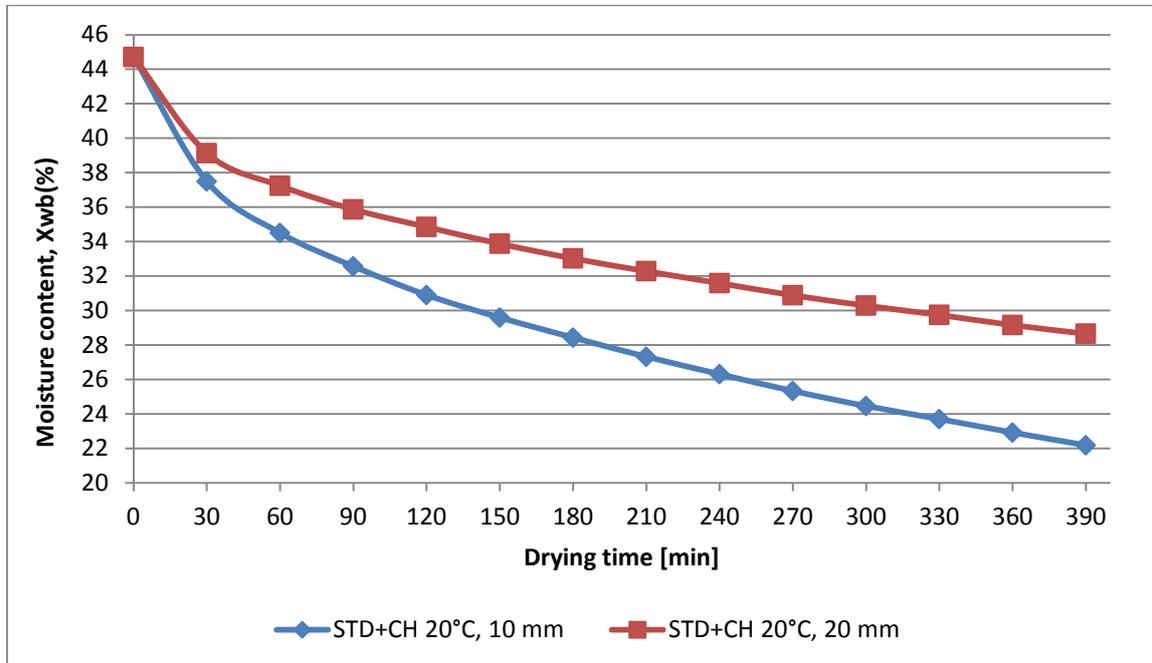


Fig. 6.4: Drying kinetics of the standard samples with extra carbohydrates

As it has been considered the same recipe, both samples with the same initial moisture content and the tests were done by filling the chamber with the same initial mass. It can be observed that the smaller particles (10 mm size) dried quicker than the larger particles under the same drying temperature and considering the same recipe. Moisture content is reduced by 22.54% in 10 mm particles and 16.06% in 20 mm ones. The main reason is that smaller particles have lower mass transfer resistance than larger cubes. Also the higher number of smaller particles provides a larger surface area for heat and mass transfer leading to a higher drying rate and shorter moisture content for the same drying period.

To reduce moisture content from 44.71% to 28.65%, drying time was decreased by 210 minutes (from 390 minutes to 180 minutes) for 10 mm particles compared to the drying time for 20 mm samples dried at the same temperature. Thus, the results indicate that the smaller the recipe sample size or thickness, the shorter the time and the quicker the product dries.

In order to analyze how moisture content is affected by size (particles of 10mm, 20mm or size distribution between 1 and 12mm in the boiled ones) two different recipe samples were dried at the same temperature. The tests were done considering an initial frozen material and the results are plotted in Figure 6.5.

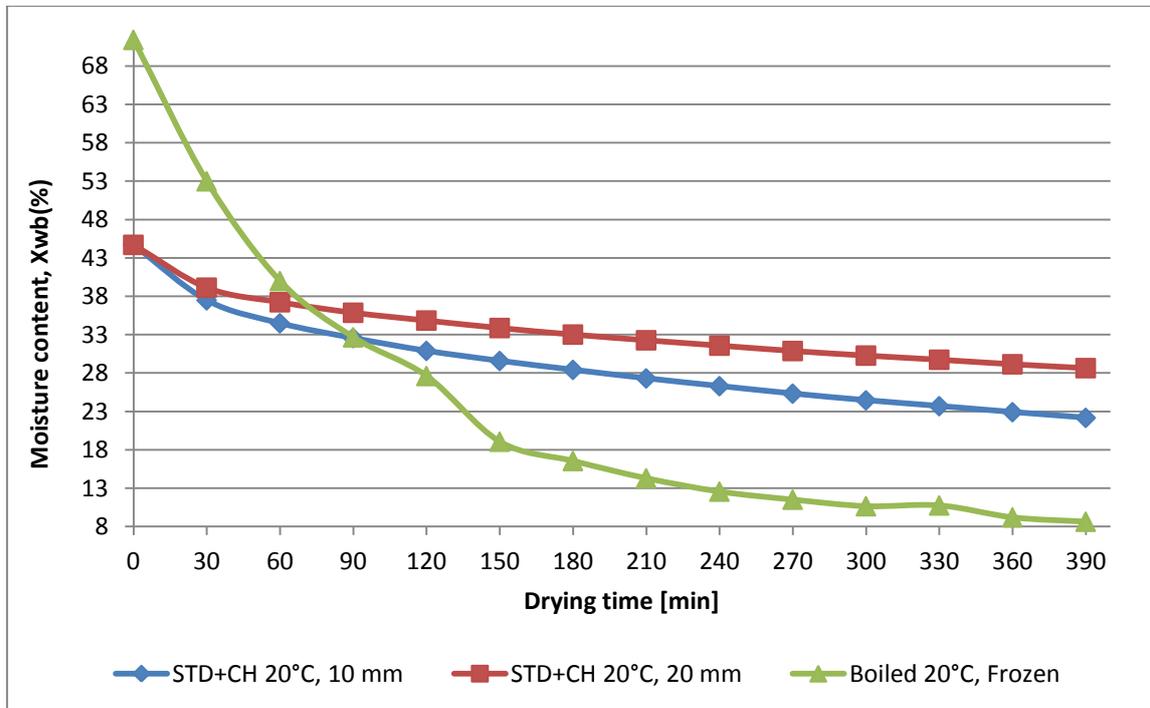


Fig. 6.5: Influence of particle size on drying kinetics for different recipes

The plot clearly show that the final moisture content difference between 10 mm STD+CH particles and boiled ones is about 13.53%wb while between 20mm and boiled particles is 20.01%wb. The drying rate is highest for the boiled sample and lowest for the sample containing extra carbohydrates with a particle size of 20 mm.

The main reason for the difference in drying kinetics is that particle size and availability of larger surface area promotes and intensifies moisture removal. It is evident that, for the same initial mass, the number and surface area in a bed of boiled particles is higher due to small size distribution. Consistently, the boiled particles have a size distribution ranging from 1 and 12 mm and dried quicker than larger particles.

In order to analyze how moisture content is affected by drying temperature it was considered the standard sample after a first drying stage. The results are shown in Figure 6.6.

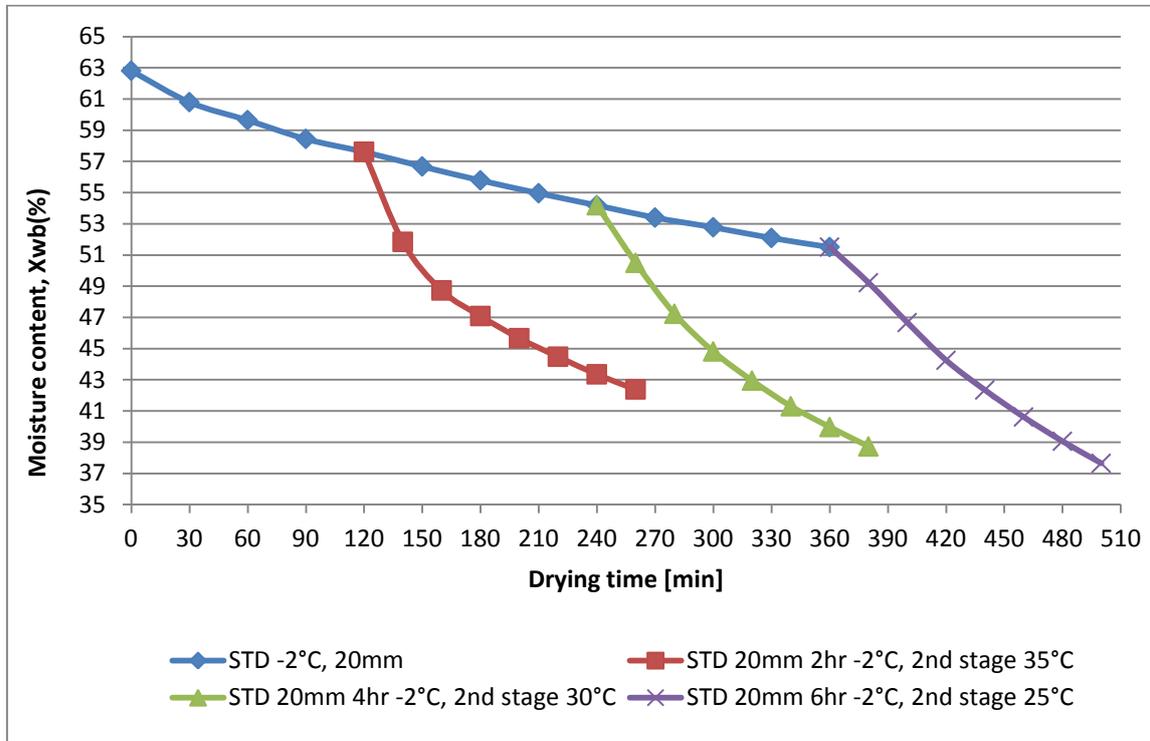


Fig. 6.6: Influence of drying temperature on drying kinetics for the second drying stage

Figure 6.6 shows moisture content plots for single-stage drying of the standard recipe after 6 hours drying at a temperature of -2°C , as well as the second drying stage for the three cases studied at different drying temperatures. As expected, there is acceleration of the two-stage drying process due to the increase of the temperature of the drying air from 25 to 35°C . This is obvious during the first periods of drying indicating that mass and heat transfer rates improve as the air temperature increases and higher moisture removal rates can be achieved. Thus, the greater the difference between the product and drying medium, the higher the drying rate.

In addition, by shifting single-stage to two-stage atmospheric freeze drying the effective diffusivities and water removal rates increased. During the first drying stage at -2°C , moisture removal rates are 5.25, 8.62 and 11.3%wb with residence times of 120, 240 and 360 minutes, respectively. As for the second drying stage, moisture removal

rates are 15.23, 14.47 and 13.87%wb for a drying period of 120 minutes and at drying temperatures of 35, 30 and 25°C, respectively. As a result of the two-stage drying, moisture removal rates of 20.43, 23.09 and 25.17%wb are achieved for drying times of 260, 380 and 500 minutes, respectively. Only 11.3% of water removal was achieved for single-stage drying at -2°C with residence time of 390 minutes.

A first drying stage at a temperature below the initial freezing point can be applied for a short time to remove unbound moisture. This stage can be followed by a second drying stage at a higher temperature to remove the residual moisture that results in higher dryer capacity. Therefore, a first drying stage of 120 minutes at -2°C followed by a second stage of 140 minutes at 35°C is recommended to increase drying rates and dryer capacity. However, it could be preferable to carry out a second drying stage at a lower temperature of 30 or 25°C if the material is sensitive to higher temperatures. For this reason, it is important to analyze quality parameters to determine the best drying process in order to achieve higher drying rates and a material with a desired quality.

6.2. Quality parameters

A better product quality is normally achieved by applying atmospheric freeze drying compared to drying with temperatures above the material freezing point.

Better quality indicators are: fast rehydration capacity, low bulk and particle density, small shrinkage, higher porosity of the product and an attractive color. These improvements could increase the current degree of acceptance of dehydrated products in the market.

In this study, the effect of recipe design, drying temperature and particle size on color, density and rehydration of protein mixtures was analyzed and evaluated.

6.2.1. Color analysis

Color is an important attribute in quality of dried foodstuffs. Hunter color parameters have previously proved valuable in describing visual color deterioration and providing useful information for quality control.

Initial and final color was measured using a color meter based on the Hunter color scale. The components are redness (a), yellowness (b) and brightness (L), which were measured and used to analyze color changes between the original material and the dried sample. The color requirement for dried protein mixtures are brightness equal or slightly lower than in the fresh material, similar red values and neutral in yellow ones.

Color changes have been studied in the standard samples containing additional carbohydrates and drying at the same temperature (20°C). Both samples have similar initial color component values because they are originated from the same mixture recipe.

In order to analyze changes in particular sample regions, the final color measurements for the dried samples have been measured at two different locations: an overall zone (white) and a proteic zone (red). The reason is that our sample has two characteristics zones because the standard sample was red and the corn starch added gives it a white component.

Some pictures of the final drying samples for both tests are shown in Figures 6.7 and 6.8.



Fig.6.7: STD+CH 10 mm samples dried at 20°C



Fig 6.8: STD+CH 20 mm samples dried at 20°C

The main color of the recipe product is red and brightness. Then, brightness (L) and redness (a) are the main color components to be analyzed and, if possible, they should be kept unchanged. Also, these quality aspects should approach the original values or be in a better range as specified for final product acceptance in the market.

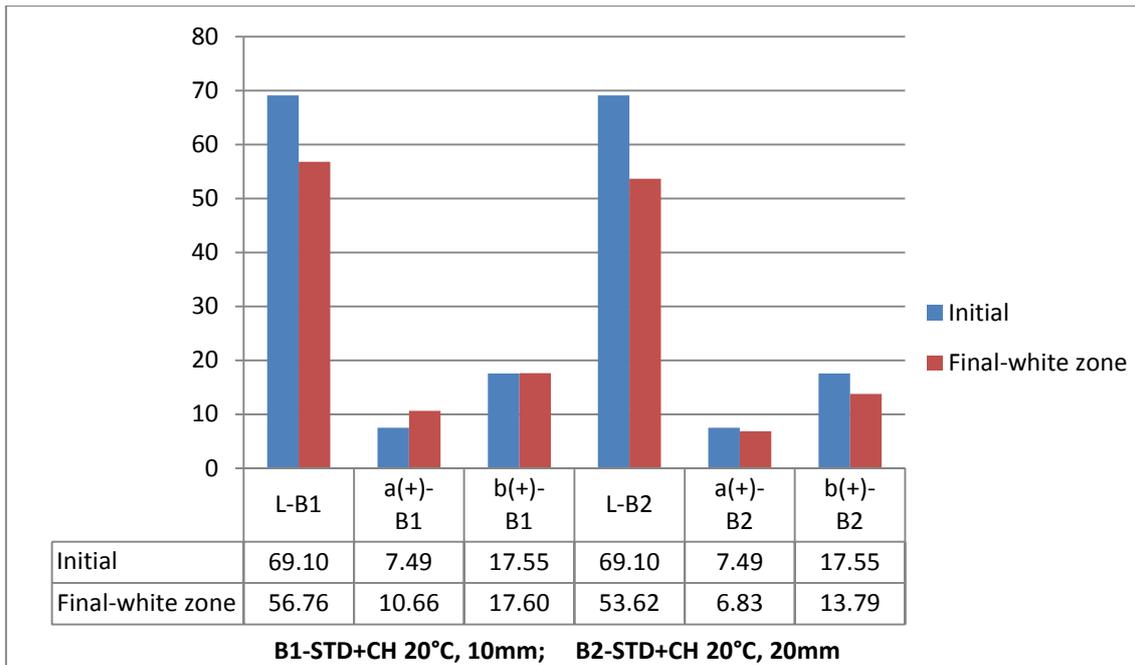


Fig. 6.9: Color measurements comparison in the white zone for different sample sizes

The measurements show how brightness (L) for both samples drops after drying in the white zone. This indicates that HPD process causes a color shift towards the darker region. Thus the white zone of the dried samples is a bit darker than the original recipe color and it is more significant in larger particles of 20 mm.

On the other hand, red color component (a) increased for 10mm particles and decreased slightly for the larger samples.

Finally, yellow component (b) increased slightly for 10mm particles and decreased for 20 mm samples.

The color is affected by particle size, as shown in the plot. The dried smaller particle of 10mm has final color closer to the original frozen sample because the final material is less dark and redder than in the case of the bigger particles. Therefore, the results show that quality of the smaller particles in the white zone is higher what is better accepted in the market.

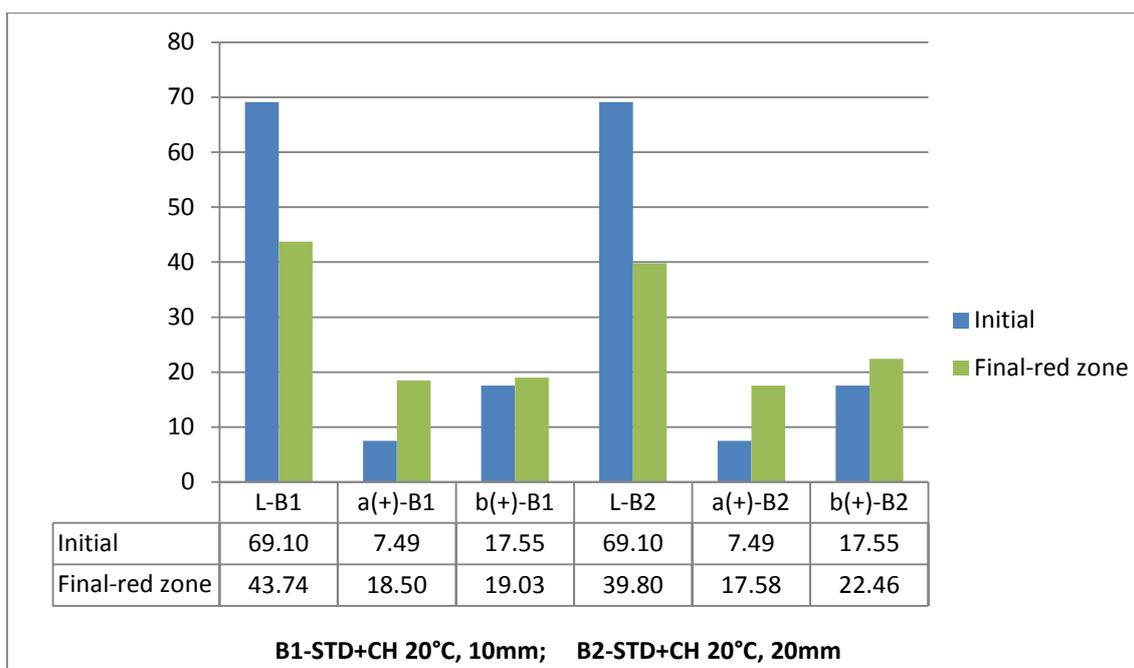


Fig. 6.10: Color measurements comparison in the red zone for different sample sizes

As for L values in the characteristic red zone, brightness is again lower in the dried material. It is also shown that the red value is lower in the final 10 mm sample than in the 20 mm sample so smaller thickness particles are not as dark as the larger ones which is preferable. On the other hand, the red component is slightly higher for smaller particles whereas yellowness is lower.

Therefore, in the proteic zone which is mainly red, brightness and redness are the main components to be taken into account. Both values change in a not negligible range in the final dried product so the standard protein mixture recipe with added carbohydrates has not the most desirable results in terms of color when dehydrates. However, slightly better color values are achieved for smaller particles of 10mm and they will be better accepted for costumers in terms of quality.

better accepted in the market due to the initial color of the standard material was mainly red.

On the other hand, yellow color component (b) is also preserved for all samples although this is not the main component in terms of quality for the protein mixture that it has been considered.

The results indicate that lower temperature produce color closer to the initial color of the fresh or frozen sample than a higher temperature. Furthermore, a single drying stage at a temperature below the protein's mixture initial freezing point followed by a second stage above the freezing point enhance color properties in the dried product.

The pictures of the final dried samples subjected to a second drying stage are shown in Figures 6.12, 6.13 and 6.14.



Fig. 6.12: Standard samples 20 mm dried at 35°C

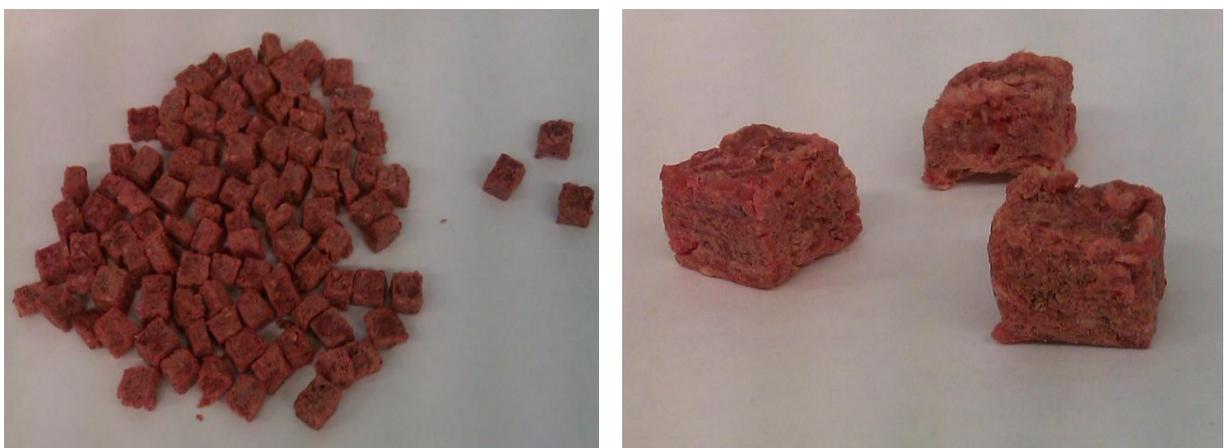


Fig 6.13: Standard samples 20 mm dried at 30°C



Fig 6.14: Standard samples 20 mm dried at 25°C

6.2.2. Bulk density

Initial and final bulk densities were measured at the start and end of the drying tests for each sample. This was done to study how the density of the product changes when drying. Each sample had different final moisture content due to varying drying temperatures and residence times.

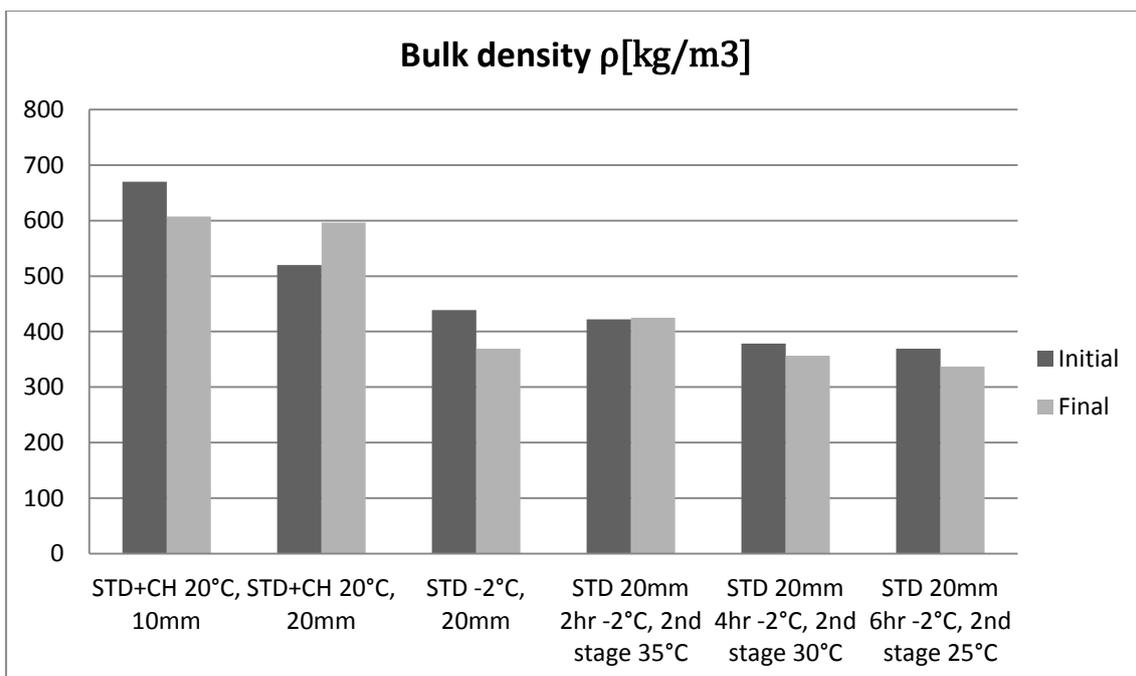


Fig 6.15: Bulk density measurements before and at the end of each drying process

Figure 6.15 shows the bulk densities for six drying tests. The higher density of standard of the 10 mm thick samples with extra amount of carbohydrate is due to higher porosity and less bulk void since the smaller particles fill the voids of the graduated cylinder used to measure bulk density. It is also observed that initial bulk density for standard samples with added carbohydrate is higher than that of standard samples because corn starch contributes to a higher value.

On the other hand, significant bulk density change is achieved for the standard 20 mm samples that was dried at -2°C and the bulk density was reduced by almost 19% (from 438.76 to 369.29 kg/m^3). The reasons are that water removal from samples containing corn starch is accompanied by volume reduction and the samples dried at higher temperatures experienced higher shrinkage. Both occurrences may cause the final density to be more similar or higher than the initial value

Low values of bulk density at the end of drying compared to initial values are achieved in almost every test. The main reason is that the matrix volume reduction is smaller than the equivalent volume or mass of the evaporated water and, as a consequence, the product porosity increases and the bulk density drops. This means that shrinkage in the products is lower for atmospheric freeze dehydrated samples and that this process does not cause structural damage to the product.

However, the 20 mm samples containing extra carbohydrates had increased bulk density probably due to reduced heat and mass transfer in these larger particles with lower surface area. In such case the water removal during drying was lower than the volume change or shrinkage causing the bulk density to increase.

In addition, the case of the samples subjected at a second drying stage at a higher temperature of 35°C leads to a higher final bulk density. This dependence may be related to shrinkage that is more pronounced during high temperature drying. That produces a less porous product with a lower final volume making the final bulk density to increase. This product may be less accepted by consumers compared to better samples obtained in the other tests.

It is also shown that the samples subjected at a second drying stage that the initial bulk density is lower for samples dried during 6 hours at a temperature of -2°C . This is because a long drying time at a given temperature allows higher moisture

removal and the product tends to shrink. However, shrinkage at temperatures below freezing point is less significant. The reduction in final density is higher in the samples dried at 25°C (almost 10% whereas reduction in samples dried at 30°C is around 6%) because there is a critical temperature where shrinkage peak, where the shrinkage is maximum. Thus, a sample dried above (or below) this temperature has a lower density.

6.2.3. Particle density

Initial and final particle densities were measured at the start and end of the drying process. The particle density for each sample is shown in Figure 6.16.

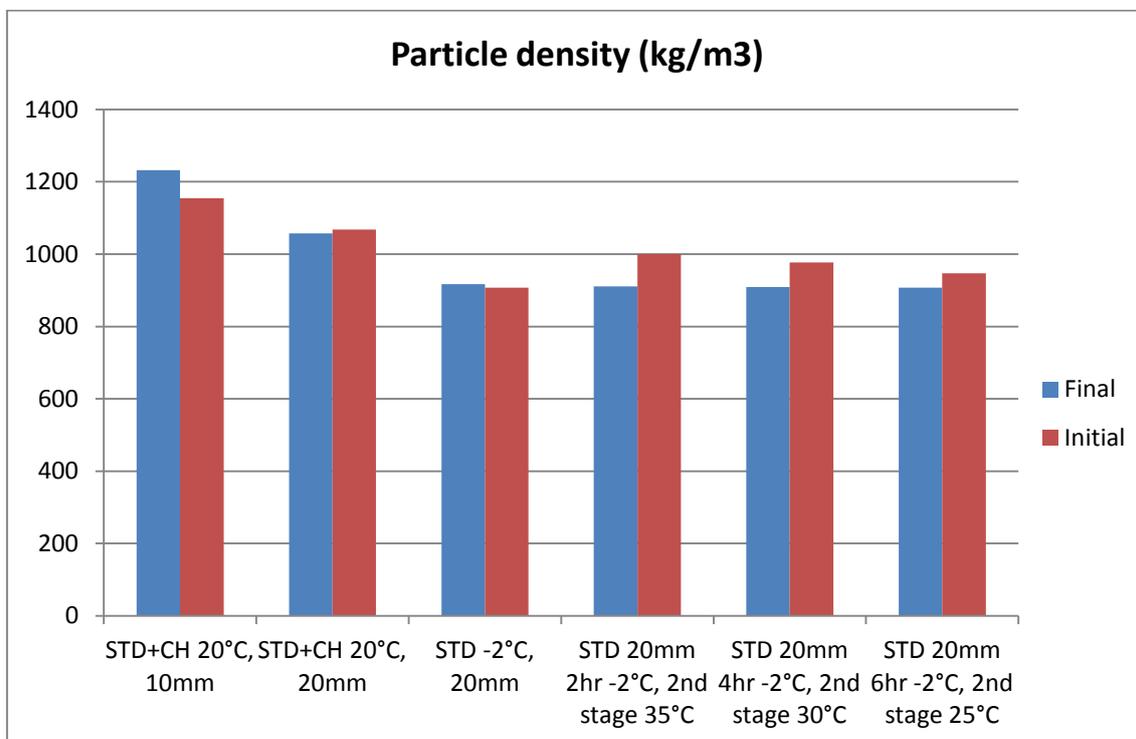


Fig. 6.16: Particle density measurements before and at the end of each drying test

The results show that samples containing corn starch as extra carbohydrate have higher particle density than standard samples.

Higher values of particle density at the end of drying compared to initial values are observed for almost all tests on the contrary of bulk density measurements. The reason is that particle density does not include the air between the particles and the

change in volume of each particle due to shrinkage becomes more significant than the corresponding water removal. This is more evident in the 20 mm samples dried at higher temperatures.

The samples subjected to a second drying stage at 35, 30 and 25°C had increased particle density from their initial values by 8.9, 7.0 and 4.2%, respectively. Only the STD+CH 10mm thick samples dried at 20°C and the STD 20mm particles dried at -2°C achieved lower final particle densities due to lower shrinkage and higher heat transfer rates, respectively.

6.2.4. Rehydration capacity

The rehydration characteristics of a dried product are important and often used as a quality index. Reconstitution of dehydrated products allows elucidation of how a material is affected by the shrinkage, porosity and structural damage after drying.

The results for the rehydration capacity for different recipes were measured for all samples dried for different drying conditions. The measured data were organized in tables and results of rehydration versus time are shown in Figures 6.17, 6.18 and 6.19.

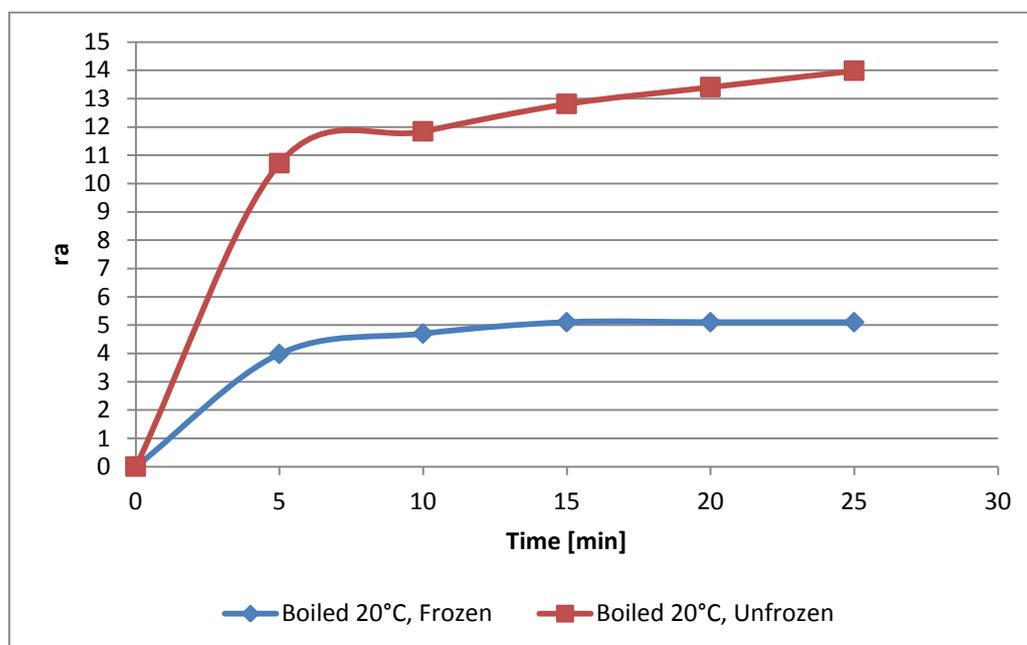


Fig 6.17: Rehydration rates for the boiled samples

The plot indicates that the boiled samples have excellent rehydration capacity by having an improved structure and particularly high porosity. Furthermore, it is observed that particles that were initially unfrozen rehydrates better than frozen particles because moisture removal and final porosity were higher.

Therefore, rehydration capacities of pre-treated particles, such as boiled samples, were higher than for untreated samples.

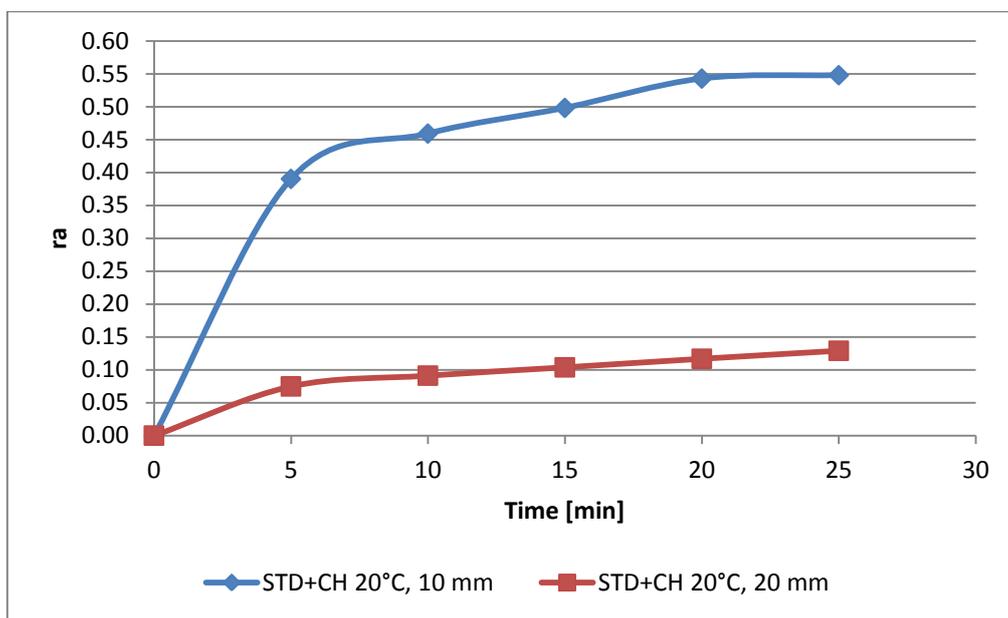


Fig 6.18: Rehydration rates for the standard samples with added carbohydrates

Standard samples containing extra amount of carbohydrates exhibited lower rehydration capacity than boiled samples. As expected, the rehydration ratio for the 20 mm particles dried at 20°C is the lowest among the different recipes considered. The final value for this sample where the rehydration ratio becomes constant is 0.13 whereas the final value for the standard sample subjected to a second drying stage at 35°C (see Figure 6.19) is 0.25. This resulting low rehydration is in agreement with bulk density data since samples with larger particle size were strongly affected by shrinkage and high temperature drying. It is apparent that low shrinkage and more porous structure leads to higher capacity to absorb water during reconstitution.

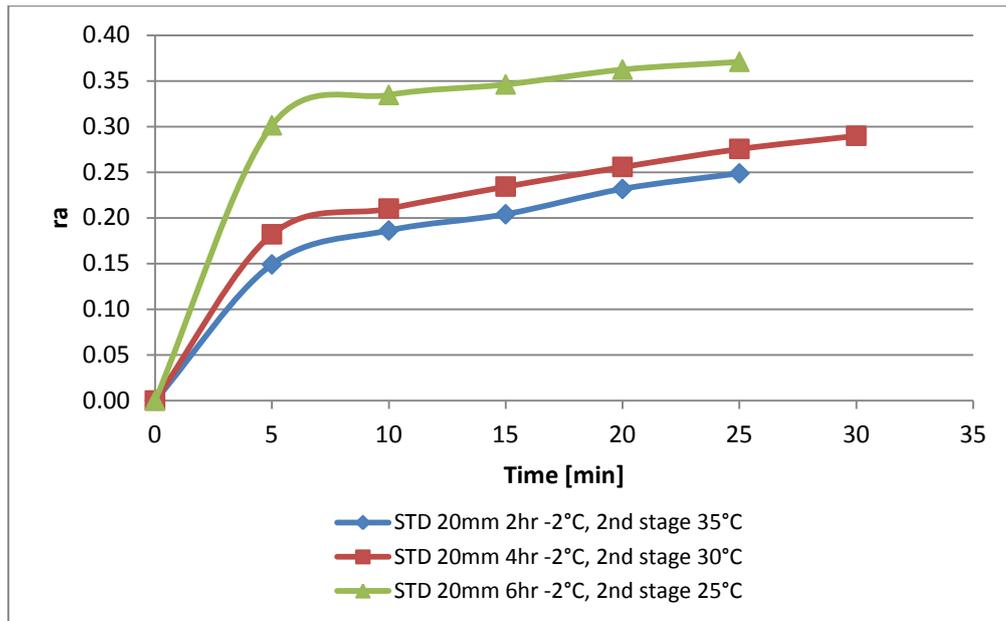


Fig 6.19: Rehydration rates for the standard samples subjected at a second drying stage

It is clearly observed that rehydration capacity is higher for two-stage dried samples at lower temperatures because material is less affected by shrinkage and the product is more porous. Thus, water gain in these samples is higher indicating that product quality is better. Accordingly, drying at lower temperatures enhances the rehydration and quality of the final product.

Rehydration capacity of the single-stage dried samples at a temperature of -2°C could not be measured because the total amounts of these samples were used in the second stage drying process. Therefore, conclusions about rehydration rates differences between single and two-stage dried samples could not be drawn.

Conclusions and Recommendations

7.1. Conclusions

There has been a significant growth in the last years in using heat pumps in combination with drying operations. Heat pump drying satisfies important requirements in industrial drying as it is known as an energy efficient and environmentally friendly technology which produces high quality drying products. A diversity of lines of research and development has been established at NTNU and SINTEF in this field.

Based on experiments carried out using protein mixtures and applying a fixed bed heat pump dryer (see Chapter 6) it was found that the higher drying rates were achieved for the protein mixtures subjected to a boiling pre-treatment and they also have higher rehydration rate due to the more porous structure of the samples. On the other hand, single-stage drying of the standard 20 mm samples below the initial freezing point at a temperature of -2°C requires much longer drying times to achieve the same moisture removal which leads to an increase in the energy consumption. However, this drying condition causes less damage to the product and its final quality is better accepted in the market.

Temperature and sample size are the characteristics strongly affecting quality parameters. Color components of the samples are well-preserved after heat pump drying although lower brightness and slightly higher redness values are normally achieved for the different recipes and drying conditions tested.

Slightly better color values close to fresh protein mixtures are achieved for standard recipes comparing to recipes containing added carbohydrates and also samples drying at lower temperatures but color differences are not significantly different to decide which recipe has the best quality at the end of the drying process.

As far as bulk density concerned, the highest difference between the initial and final dried product is achieved for the standard recipe dried at -2°C and also for the standard 10 mm sample with added carbohydrate. Bulk densities are reduced about 19% and 10.3%, respectively. This clearly shows that smaller sample and lower drying temperature produce better product quality in terms of final density. The reason is that mass reduction in these samples is higher than volume reduction due to shrinkage so a more porous sample is obtained which is desirable. In addition, standard 20mm samples dried at -2°C and followed by a second drying stage at a temperature of 25 and 30°C presented lower final densities. However, higher final density values from the initial ones are achieved in samples dried at 35°C as well as in 20 mm samples with added carbohydrate. Therefore, both samples exhibited shrinkage values that cannot be accepted in the market and drying under these conditions cannot be validated.

As for rehydration capacity, higher values are presented at lower temperatures. Standard 20 mm samples subjected to a second drying stage at 25°C have reasonable good rehydration capacity as well as 10 mm samples containing added carbohydrate and dried at 20°C which achieved the highest values. Nevertheless, 20 mm samples for this recipe have the lowest rehydration rate what is in agreement with bulk densities values because they are strongly affected by shrinkage.

Thus heat pump drying allows handling of heat sensitive materials either under non or atmospheric freeze drying conditions. The possibility of sequencing these two operations is specially advantageous when drying foods because drying under frozen conditions minimizes shrinkage although at lower drying rates. Furthermore, splitting of

the drying process into atmospheric freeze drying at temperatures around -2°C followed much faster finish drying at 25 or 30°C making it possible to control quality parameters such as porosity, rehydration rates and color.

The recipes containing added carbohydrates achieved good quality properties with optimum drying rates but only for small samples of 10 mm sides. Moisture removal ratio was about 22% during a 390 minutes drying process at 20°C . Furthermore, 20 mm standard samples dried at a temperature below the freezing point at -2°C and then at a temperature of 25°C produced acceptable quality with moisture removal rates about 25% during a drying process of 510 minutes. Therefore, samples containing added carbohydrates and dried at 20°C are recommended but only for production of samples with 10 mm side. The reason is that drying capacity is higher leading to lower cost of production since more product can be dried to a desired moisture content during the same time. This leads to lower dried product prices for the customer. However, drying capacity can be improved for standard recipes by reducing the sample size from 20 mm to 10 mm.

7.2. Recommendations for Further Work

So far, a lot of improvements have been done in HPD technology in order to reduce energy consumption in industrial drying. But higher energy efficiencies and better control of quality parameters can be achieved in the future.

Further research is necessary and it is of great interest to conduct more experiments with a wider variety of settings. Special interest could be a single-stage drying of the standard recipe for smaller samples thickness to compare with the results obtained for the standard recipe with added carbohydrate. In addition, more experiments on the two-stage drying for the standard recipe with added carbohydrate and also for the boiled recipe need to be carried out to be able to make predictions.

Further experiments should also include measurements of total energy consumption which is related to production cost and important for scale up the dryer

system from laboratory to industrial size. Furthermore, comparison of the energy consumption to other drying forms could be an interesting task.

Research other methods, processes and parameters for intensification of the drying process is recommended. The effect of the bed height could also be increased to influence capacity and compare drying rates.

Finally, it should be considered that industrial operations are made in continuous mode instead of laboratory test doing in batch mode. This should be considered in future experiments and a great challenge would be to develop an industrial scale plant working on this mode.

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