# Optimization of "murtilla" berry drying in an atmospheric freeze dryer

Alejandro Reyes <sup>1</sup>, Ruben Bustos <sup>1</sup> R., Maria Belen Vásquez <sup>1</sup>, Erick Scheuermann <sup>2</sup> Department of Chemical Engineering, Universidad de Santiago de Chile <sup>2</sup> Department of Chemical Engineering, UFRO, Temuco, Chile E-mail: alejandro.reyes@usach.cl

'Murtilla', 'mutilla' or 'murta' (*Ugni molinae* Turcz) is a native Chilean species that produces a small berry fruit with a special aroma and a high antioxidant capacity. It is consumed raw, as jams, juices, canned products, confectioneries and liquors. The short production season demand application of preservation processes in order to enjoy the sensory and nutritional properties of this fruits during the whole year. Freeze drying is the technology that allows keeping such attributes almost indistinguishable from the fresh fruit, as consumers demand, however the high cost of conventional freeze drying (at high vacuum) preclude the mass utilization of this technology. In the present study the optimum conditions for the operation of an atmospheric freeze dryer in a pulsed fluidized bed were determined. Applying a 2<sup>3</sup> experimental design the effect of infrared radiation application, freezing rate and temperature on moisture content, drying time, polyphenols content and antioxidant capacity of dried murtilla fruits were determined.

Drying times were equivalent to those observed for conventional freeze drying and the product obtained kept most of its sensory attributes but equipment and energy costs are significantly reduced.

Keywords: murtilla; *Ugni molinae* Turcz; atmospheric freeze dryer

## 1. Introduction

The convective drying impairs sensory and functional properties of foods, due to protein and vitamin degradation caused by high temperature. Besides, the structure of the solid is heavily damaged which leads to significant texture loss upon rehydration. Freeze-drying represents the best drying alternative for foods (Di Matteo *et al.*, 2003; Venir *et al.*, 2007; Reyes *et al.*, 2008). This process consists of three stages: i) freezing, ii) a primary drying stage, where sublimation of free water occurs (between 65-90% of water content) and iii) a secondary drying stage, where bound water is eliminated by desorption (between 10-35% of the initial moisture content). The negative points are that the freeze drying process is slow and involves high investment and operating costs due to the need of producing and maintaining high vacuum (Ratti, 2001). One

alternative to reduce costs is to use an atmospheric freeze-dryer, since expensive vacuum associated components could be eliminated, although with this technique, the drying time increases (Claussen *et al.*, 2007, Di Matteo *et al.*, 2003; Alves-Filho *et al.*, 2007; Tomova *et al.*, 2004). On the other hand, a way to reduce this drying time is to use a fluidized bed, which has high heat and mass transfer coefficients.

Murtilla is a native Chilean species that produces a small berry fruit with an equatorial diameter of 0.7–1.3cm and with a special and unique aroma and flavour. The fruit is also characterized for having a high antioxidant capacity, equal or superior to other commercial berry fruits. It is consumed raw, as jams, juices, canned products, confectioneries and liquors (Scheuermann et al. 2008).

To reduce the atmospheric freeze-drying time of murtilla particles in a pulsed fluidized bed, the present work studied the influence of freezing rate, air temperature and type of energy supply, on the moisture content reached. The mass transfer gradient was maximized by circulating the air through a fixed bed of silica gel.

# 2. Equipment, materials and methods

Figure 1 shows the scheme of the Atmospheric Freeze Dryer utilized, where air is circulated by a centrifugal blower (A), the temperature is adjusted in a cooling system (B) and dried in a fixed bed of silica gel (C). Then, it passes through a horizontal rotating cylinder with two slots which generate pulsating air flow (D), passing then through murtilla particles bed (E). The system is provided with an IR lamp for runs that require it (F).

Silica gel (2kg) was loaded in the corresponding compartment (Fig. 1-C), the desired temperature value was set on the cooling system (Fig. 1-B) and the pulse generator and centrifugal blower were turned on. After the system reached steady state, 250grams of freezing murtilla fruits were loaded in the drying chamber (a 9.5cm diameter glass tube, with a perforated plate at the bottom to allow air pass through), the initial mass was measured with a BBL62 Boeco balance (Boeco Germany). After 60 minutes, the glass tube was taken out from the freeze- dryer, it was weighed and replaced in the equipment. This procedure was repeated until 13 hours of operation. The moisture content was determined in a vacuum oven until constant weight according to AOAC 920.151 (Anon., 1990). Silica gel was replaced every 2 hours to ensure the fluidization air was dry.

'Murtilla', 'mutilla' or 'murta' (*Ugnimolinae* Turcz), ecotype 14-4 from INIA Carrillanca Genetic Bank, was obtained from an experimental station near to Puerto Saavedra (Chile). Berry fruits with an average diameter of 10 mm ( $\pm$  2 mm) were selected which had 1062 kg/m<sup>3</sup> density and 79.3 % initial moisture (wet basis).

Freezing of samples was carried out in a household freezer for 15 hours at  $-18^{\circ}$ C (slow freezing) or by immersion in liquid nitrogen for 5 minutes (fast freezing). Once frozen, berries were cut in halves in order to reduce drying time.

Antioxidant activity, moisture and polyphenols content of murtilla fruits during atmospheric freeze-drying in a pulsed fluidized bed were studied as a function of freezing rate (slow and fast), air temperature from 7 h (5°C and 15°C) and type of energy supply (convective or convective + IR). The experimentation was carried out

through a factorial design 2<sup>3</sup> and the statistical analysis was carried out using the software Statgraphics Plus 5.1 (Statistical Graphics Corp., USA, 2000).

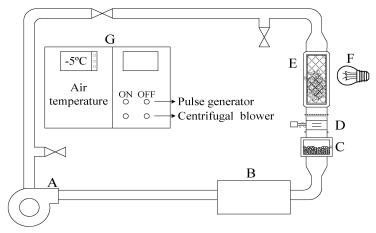


Figure 1. Atmospheric Freeze Dryer. A: centrifugal blower, B: air cooling system, C: silica gel, D: pulse generator, E: freeze drying chamber, F: IR lamp, G: control panel.

**Table 1.** Real and codified level values for the experimental variables

Run	Air Temperature in the second stage <sup>(1)</sup> (°C)	Freezing Rate	Type of energy supply
A1	15 [1]	FF (Fast) [1]	Convective [0]
A2	15 [1]	FF (Fast) [1]	Convective + IR [1]
A3	5 [0]	FF (Fast) [1]	Convective[0]
A4	5 [0]	FF (Fast) [1]	Convective + IR [1]
B1	15 [1]	SF (Slow) [0]	Convective[0]
B2	15 [1]	SF (Slow) [0]	Convective + IR [1]
В3	5 [0]	SF (Slow) [0]	Convective[0]
B4	5 [0]	SF (Slow) [0]	Convective + IR [1]

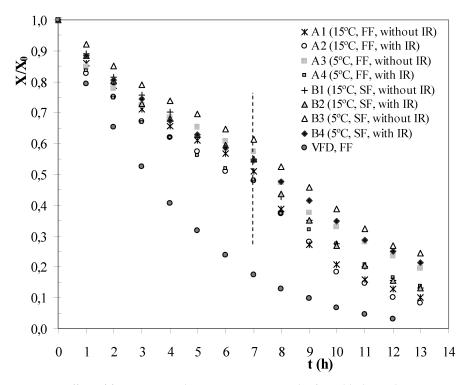
(1) From 7 hours onwards; [Codified Level value]

# 3. Results

Preliminary trials showed that it was necessary to cut the berry fruits in halves in order to get acceptable drying rates. The skin of this berry is a strong natural barrier to dehydration. Even the application of warm alkali treatments that are effective for most other fruits proved to be completely ineffective for murtilla. Additionally, it was verified that better drying results were achieved when two temperature levels were applied during drying: -5 °C during the first 7 hours and 5°C or 15°C during the final 7 hours.

## 3.1 Drying kinetics

Figure 2 shows that increasing the drying temperature from hour 7 onwards the drying rate either increases slightly or, at least, remains the same than during the first 7 hours. Therefore, without applying this temperature increase, drying rate would have significantly decreased since it is at this time that the removal of bound water begins a process normally much more slow than what is observed in these experiments. Figure 2 also shows the drying kinetics for a lyophilisation process under vacuum (VFD), where a drying rate higher than in atmospheric lyophilisation is observed. However, when moisture contents are lower that X/Xo = 0.1, the values come close to those of the run performed at atmospheric pressure (Run A2). It can also be observed that when the applied temperature is the same, application of IR or fast freezing (FF) both increase the drying rate.



**Figure 2.** Effect of freezing rate, drying temperature and infrared light application on the drying kinetics of murtilla fruit halves.

Table 2 shows which experimental variables have significant effects on the final moisture content of murtilla fruits after 13 hours of drying for 90% and 95 % confidence interval. In both analyses temperature had the most significant effect on the reduction of the moisture content, followed by the freezing rate and then IR application. The lowest moisture content after 13 hours of drying were obtained by drying at 15°C samples that were subjected to fast freezing and with the application of IR.

**Table 2.** Estimated effect for the variables Air temperature (A), Freezing rate (B) and Type of energy supply (C) on the final moisture content of murtilla fruit halves dried in an atmospheric pressure fluidized bed freeze dryer.

% confidence	Average	Air temperature (A)	Freezing rate (B)	Type of energy supply (C)	AB	AC	ВС	$s_{ m ef}$
90	0.1543	-0.0860*	-0.0500	-0.0265	0.0115	0.0180	-0.0120	0.0025
95	0.1543	-0.0860	-0.0500	-0.0265	0	0	0	0.0025

<sup>\*</sup>Significant effects in bold

Equation 1, where the codified levels of the variables [0 or +1], are utilized, allows to estimate the final moisture content of murtilla fruits for a 90% confidence interval. At a 95% confidence level, only the three first terms of this equation remains.

$$\left(\frac{X}{X_0}\right) = 0.24425 - 0.1155 \cdot [A] - 0.0495 \cdot [B] - 0.0325 \cdot [C] + 0.036 \cdot [AC] \tag{1}$$

#### 3.2 Antioxidant activity and polyphenols content of murtilla

Whatever the processing condition applied, there is a decrease in both, polyphenols content and antioxidant activity with regard to the fresh fruit. However, under the conditions of Run A4, antioxidant activity remains almost identical to fresh murtilla fruits (Table 3). On the other hand, under the conditions of Run A2, the experiment that showed the highest drying rate, the greater decrease in polyphenols content among all the experiments is observed. Curiously, the antioxidant activity shows a tendency to increase with the application of IR. Probably such behavior could be due to the fact that this runs had shorter drying times and, therefore, the time of contact of murtilla with air (an oxidant agent) was shorter.

**Table 3:** Effect of the drying process conditions on the final polyphenols content and antioxidant activity of murtilla fruit halves

Type of drying	Drying process conditions	Polyphenoles (mg/100g dry weight)	Antioxidant activity EC50 (mg/L)	
	Fresh fruit	1460	108	
Vacuum	Fast rate freezing	875	82	
freeze drying (VFD)	Slow rate freezing	950	58	
Atmospheric	Run B3 [5°C, SF, without IR]	868	61	
freeze drying (AFD)	Run B4 [5°C, SF, with IR]	760	77	
	Run A2 [15°C, FF, with IR]	605	71	
	Run A4 [5°C, FF, with IR]	680	98	

With the goal of keeping the antioxidant capacity of fresh murtilla in the final product, it can be stated that berries dried at atmospheric pressure have a greater antioxidant capacity than the vacuum freeze dried fruits. Under optimum operation conditions (Drying at 5°C, fast freezing and IR application), a large percentage of the antioxidant capacity presented in the fresh fruit remains in the dry fruit. The dry fruit showed a slight darkening that made the red-purplish color of the fresh fruit to change to a more intense red color that might be equally attractive. The texture also kept adequate to what is expected when eating a dry fruit which also kept a pleasant flavor when consumed directly or with yogurt.

#### 4. Conclusions

The size of the murtilla semi-spheres is adequate to fluidize them in a pulsed fluidized bed. The drying times achieved by the atmospheric freeze drying configuration proposed are equivalent to those observed for conventional freeze drying and the product obtained kept most of its sensory attributes. The atmospheric freeze drying configuration proposed allows for significant reduction in equipment and energy costs.

**ACKNOWLEDGEMENTS**. The authors thank the financial support made by Project Fondecyt 1070019 and DICYT - Usach.

#### 5. References

- AOAC, 1990, Official methods of analysis. In: Association of Official Analytical, 15<sup>th</sup> Ed..; K. Heldrich (Ed.); Washington, DC, USA, 1298.
- Alves-Filho, O., T.M. Eikevik, A. Mullet, C. Garau and C. Rossello, 2007. Kinetics and Mass Transfer during Atmospheric Freeze-drying of red Pepper. Drying Technology, 25, 1155-1161.
- Claussen, I.C., T.S. Ustad, I. Strømmen and P.M. Walde, 2007. Atmospheric Freezedrying A review. Drying Technology, 25, 947-957.
- Di Matteo, P., G. Donsì and G. Ferrari, 2003. The role of heat and mass transfer phenomena in atmospheric freeze-drying of foods in a fluidized bed. Journal of Food Engineering, 59 (2-3), 267-276.
- Ratti, C., 2001. Hot air and freeze-drying of high-value foods: a review. Journal of Food Engineering, 49, 311-319.
- Reyes A., Vega R., Bustos R. and Araneda C., 2008. Effect of Processing Conditions on drying Kinetics and Particle Microstructure of Carrot, Drying Technology 26, 1272-1285
- Scheuermann E.; I. Seguel; Montenegro A.; Bustos R.; Hormazabal E.; Quiroz A., 2008; Evolution of Aroma Compounds of Murtilla Fruits (Ugni Molinae Turcz) During Storage, Journal of the Science of Food and Agriculture, 88 (3), 485-492
- Tomova, P., W. Behns, H. Haida, M. Ihlow and L. Mörl, 2004. Experimental analysis of fluidized bed freeze drying, In 14<sup>th</sup> International Drying Symposium, São Paulo, Brazil, August 22<sup>nd</sup>-25<sup>th</sup>, 526-532.
- Venir, E., M. Del Torre, M.L. Stecchini, E. Maltini and P. Di Nardo, 2007. Preparation of freeze-dried yoghurt as a space food. Journal of Food Engineering, 80 (2), 402-407.