

# Study the Effect of Pre-Treatment of Drying 'Mortño' (*Vaccinium Floribundum Kunth*) with Reference to Drying Rate and Total Content of Soluble Polyphenols and Anthocyanins

García, Almudena<sup>1</sup>; Ruales, Jenny<sup>1</sup>

<sup>1</sup>Escuela Politécnica Nacional, Departamento de Ciencia de Alimentos y Biotecnología, Facultad Ingeniería Química y Agroindustrial, Quito, Ecuador

**Abstract:** The aim of this work was to study the effect of pre-treatments on drying of 'mortño', on the drying rate and total content of soluble polyphenols and anthocyanins. It was applied to fresh fruit pre-treatment of immersion bath of aqueous solution of ethyl oleate and potassium carbonate. It was used a 22 factorial design with axial points, to evaluate its effect on the drying rate and content of phenolic compounds. It was performed a second pre-treatment with SO<sub>2</sub>, it was used a 22 factorial design to study its effect on the content of phenolic compounds. Stability assays were performed with dried fruits stored in a laminated multilayer film and metalized for 2 months under refrigeration (4 °C), freezing (-17 °C) and 25 °C at 90 % RH. It was evaluated the moisture content, color, soluble polyphenols and anthocyanins. It was conducted a sensory analysis of the product. The best conditions to dry 'mortño' are 40 °C, airflow of 0.24 m<sup>3</sup>/s, immersion bath concentration of 1.55 % K<sub>2</sub>CO<sub>3</sub> (w/w) - 1.25 % of ethyl oleate (V/V), concentration of SO<sub>2</sub> 2 000 ppm and sulfidation 2 h. The product was dried to 5 % moisture in the oven in 40 % less time required to dry without pretreatments. The dried 'mortño' retained 98.2 % of anthocyanins and 99.7 % of soluble polyphenols in relation to freeze-dried 'mortño'. Meanwhile the 'mortño' dried by solar dryer hybrid retained 88.5 % of anthocyanins and 99.7 % of soluble polyphenols. The product stored at 18 °C in a multilayer film laminated and metalized, retains 50 % of anthocyanins in relation to its initial content in 4 months and 11 days.

**Keywords:** Mortño, dehydration, pre-treatments, soluble polyphenols, anthocyanins.

## Estudio del Efecto del Pretratamiento de Secado de 'Mortño' (*Vaccinium Floribundum Kunth*) con Referencia a la Velocidad de Secado y el Contenido Total de Polifenoles Solubles y Antocianinas

**Resumen:** El objetivo de este trabajo fue estudiar el efecto de los pretratamientos en el secado de 'mortño', en la tasa de secado y el contenido total de polifenoles solubles y antocianinas. Se aplicó al pretratamiento de fruta fresca un baño de inmersión de una solución acuosa de oleato de etilo y carbonato de potasio. Se utilizó un diseño factorial 22 con puntos axiales, para evaluar su efecto sobre la velocidad de secado y el contenido de compuestos fenólicos. Se realizó un segundo pretratamiento con SO<sub>2</sub>, se utilizó un diseño factorial 22 para estudiar su efecto sobre el contenido de compuestos fenólicos. Los ensayos de estabilidad se realizaron con frutos secos almacenados en una película multicapa laminada y metalizados durante 2 meses bajo refrigeración (4 °C), congelación (-17 °C) y 25 °C a 90% de HR. Se evaluó el contenido de humedad, color, polifenoles solubles y antocianinas. Se realizó un análisis sensorial del producto. Las mejores condiciones para secar 'mortño' son 40 °C, flujo de aire de 0.24 m<sup>3</sup>/s, concentración del baño de inmersión de 1.55% K<sub>2</sub>CO<sub>3</sub> (p/p) - 1.25% de oleato de etilo (V/V), concentración de SO<sub>2</sub> 2 000 ppm y sulfuración 2 h. El producto se secó al 5% de humedad en el horno en un 40% menos de tiempo para secar sin pretratamientos. El 'mortño' seco conservó el 98,2% de antocianinas y el 99,7% de polifenoles solubles en relación con el 'mortño' liofilizado. Mientras tanto, el 'mortño' secado por el híbrido del secador solar retuvo el 88.5% de las antocianinas y el 99.7% de los polifenoles solubles. El producto almacenado a 18 °C en una película multicapa laminada y metalizada, retiene el 50% de antocianinas en relación con su contenido inicial en 4 meses y 11 días.

**Palabras Claves:** Mortño, dehydration, pretreatments, soluble polyphenols, anthocyanins.

### 1. INTRODUCTION

The 'mortño' (*Vaccinium floribundum* Kunth) is a native wild fruit from the Ecuadorian highland moors. In Ecuador, cultivations do not exist to be marketed. It grows in small allotments where the indigenous communities perform the

manual collection twice a year in July-August and October-November (Popenoe et al., 1989, p, 218). Its main use is for the preparation of the "colada morada", (a hot fruit drink with different fruits and spices), which is consumed in November in the "Day of the deceased's". The 'mortño' is a source of polyphenols, anthocyanins,  $\beta$ -carotene, and vitamin C,

[almudena.garcia@epn.edu.ec](mailto:almudena.garcia@epn.edu.ec)

Recibido: 11/05/2016

Aceptado: 16/02/2018

Publicado: 31/01/2018

minerals as potassium, zinc and dietary fiber (Vasco et al., 2009, p. 8275).

These compounds protect to the humans of certain degenerative illnesses as cancer, Alzheimer, diabetes and coronary illnesses. Cardiovascular diseases (Pascual y Sánchez, 2008, p. 282). The blueberries are used in the food industry, due to the phenolic compounds present in these fruits have anti-microbial and antioxidant effects (Heinonen, 2007, p. 688). Based on above-mentioned, it is important to promote the consumption of this fruit as functional food or as an ingredient of drinks and products like dry fruits.

On the other hand, the drying process allows preserving the foods. The conditions of the pretreatments like concentrations of the immersion solutions, exposition time should be determined and selected considering treatments that allows to save energy, the minimum drying time and to have the maximum retention of phenolic compounds.

It has been demonstrated that in fruit processing, the oxygen presence and high temperatures (90 °C) accelerate the destruction of the anthocyanins, meanwhile that if the process was performed in a range from 40 to 60 °C it is not affected its conservation significantly (Macheix et al., 1990, p. 316; Routray y Orsat, 2011, p. 306). In order to increase the drying speed approximately twice as much in the production of grapes happens, cold bath of solution of potassium carbonate and ethyl oleate are used. The ester changes the structure of the wax in the peel of the fruits and it reduces its surface tension; the salt, on the other hand, allows the formation of a stable emulsion (Christensen y Peacock, 2000, p. 213). By means of the sulfidation pre-treatment, it is possible to inactivate the enzymes, especially the polyphenoloxidase which is the causing of the oxidation of the anthocyanins (Macheix et al., 1990, p. 1 990; Clifford, 2000, p. 1 065).

The present work had as objective to study the effect of pre-treatments of drying 'mortiño' as regards to the drying speed and the retention of total content of soluble polyphenols and anthocyanins. A stability test was performed on the dried 'mortiño', stored to different storage conditions, humidity and temperature. The fruits were also dried in a hybrid solar dryer to the best conditions of the pre-treatments.

## 2. MATERIALS AND METHODS

### 2.1 Materials

In this work was used fresh 'mortiño' (*Vaccinium floribundum* Kunth) at organoleptic ripeness stage. It was acquired in Pedregal located in Machachi in Mejía County. The fruits were disinfected in a water solution chlorinated with 100 ppm. The disinfected fruits were washed in a continuous rotational washer for fruits with bars, brand Sinclair Scott Co. The washed 'mortiño' was packed in polyethylene cases in portions of approximately 3 kg and stored at 5 °C.

### 2.2 Physical-chemical characterization of the raw material and of processed

For the physical-chemical characterization of the raw material, fruits were freeze dried in batches of 2 kg.

#### 2.2.1 Methods of physical-chemical characterization of the raw material

In the fresh fruit, it was determined: pH, ashes, humidity and acidity titratable by the method AOAC (2007). In the freeze-dried 'mortiño' samples was determined a total content of soluble polyphenols by means of the method of Folin-Ciocalteu (Slinkard y Singleton, 1997). Total anthocyanins were determined by applying the method described by Giusti (Giusti y Wrolstad, 2001). Proteins and ether extract by the method, (AOAC (2007), galacturonic acid according to (Theander et al., (1991)), minerals (K, Ca, Mg, Cu, Zn, Fe) by the method (DECAB (2001)), total dietetic fiber by the method (Asp et al. (1983)), sugar contents (DECAB, 2004a), vitamin C (DECAB, 2004b) and the contents of carotenoids (Pettersson y Jonsson, 1990) by HPLC.

#### 2.2.2 Physical-chemical characterization of the processed product

In the dried 'mortiño' samples, soluble polyphenols were quantified, total anthocyanins, humidity, minerals, total dietary fiber and profile of sugars, vitamin C, and organic acids. In order to quantify the percentage of retention of phenolic compounds in the dried 'mortiño' in a stove and in a hybrid solar dryer, it was considered as 100 % the quantity in the freeze-dried 'mortiño'.

#### 2.3 Temperature effect and airflow in the drying process in a tray dryer

The 'mortiño' was dried in a stove (Freas model 625-A) with a variation of airflow, provided of four perforated aluminum trays of dimensions 45 x 42.5 cm. In order to study the effect of the temperature and airflow on the content of soluble polyphenols, anthocyanins content and drying time a factorial design was applied 2<sup>2</sup>. The temperature was varied between 40 and 50 °C and the flow rate between 0.24 and 0.40 m<sup>3</sup>/s of air in 2 levels. Each experiment was carried out for duplicated. The statistical analysis was carried out in the program STATGRAPHICS CENTURION.

#### 2.4 Effect of the pre-treatment of the immersion bath on the drying process

The pre-treatment was applied to the fresh 'mortiño', to determine the effect of the composition of the immersion bath and the exposure time on the content of soluble polyphenols, anthocyanins content and time of drying. It was applied for this purpose an experimental design 2<sup>2</sup> with axial points; in the Figure 1, the sorted corresponding pairs to the samples are presented. The conditions are shown for the nine samples carried out in the Table 1.

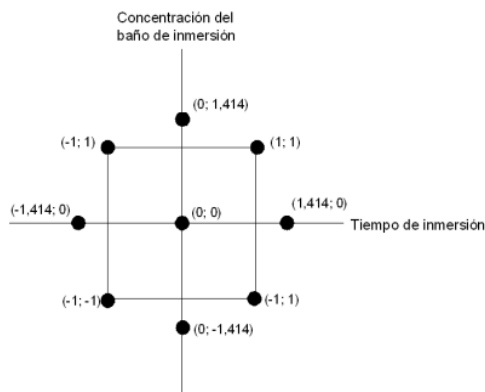


Figure 1. Experimental Model  $2^2$  with axial points

Table 1. Conditions of concentration of the bath and time of immersion for each sample

Coded value	Concentration of $K_2CO_3$ Concentration of ethyl oleate (P/P)-(V/V)	Time of immersion (min)
(1;1)	2.22-1.78	4.6
(1;-1)	2.22-1.78	2.4
(-1;-1)	0.88-0.72	2.4
(-1;1)	0.88-0.72	4.6
(0; 0)	1.55-1.25	3.5
(0;1.414)	1.55-1.25	5.0
(1.414; 0)	2.50-2.00	3.5
(0;-1.414)	1.55-1.25	2.0
(1.414; 0)	2.50-2.00	3.5

The composition of the immersion bath was fixed to 2.5 %  $K_2CO_3$  (W/W) – 2 % of ethyl oleate (V/V) and 0.6 %  $K_2CO_3$  (W/W) and 0.5% of ethyl oleate (V/V); the time of exposition was between 2 and 5 minutes. The treatment at the central point (0; 0) was repeated three times. The results were analyzed in the STATGRAPHICS CENTURION program.

### 2.5 Effect of the pre-treatment with sulfur dioxide on the drying process

The pre-treatment 'mortiño' was placed in a stainless steel chamber with a base of 60 cm and a height of 50 cm, the corresponding quantity of sulfur was burned. To analyze the influence of the sulfidation on the anthocyanins content and soluble polyphenols in the dehydrated 'mortiño', a factorial design was applied 22. The dioxide concentration of sulfur (1 000 and 2 000 ppm) and time of sulfidation (1 and 2 h) as variables of the process according to bibliographical information were selected. (Mujumdar, 1995, p. 647; Christensen y Peacock, 2000, p. 214)

### 2.6 Drying 'mortiño' in a hybrid solar dryer

The 'mortiño' with the best pre-treatments became dehydrated in a hybrid solar dryer. This consists of a solar collector of dimensions 180 x 70 cm, a chamber of drying of 60 x 70 x 100 cm, an automatic controller for the temperature, an electric resistance and two porous trays of plastic of dimensions 40 x 60 cm. The temperature in the chamber was 50 °C and the airflow was provided with a fan that was applied to the equipment.

### 2.7 Tests of stability

The dried 'mortiño' with the best conditions of temperature, airflow and parameters of pre-treatments, underwent tests of stability. The dried fruits were stored during 2 months in a laminated and metalized multilayer film, to -17 °C, 4 °C and 25 °C to 90% HR. The color was evaluated and the soluble anthocyanins, polyphenols and humidity were quantified.

### 2.8 Sensorial analysis

It was carried out the sensorial analysis to establish the existent differences in the color, flavor, texture and strange flavors, among the product dried in stove without pre-treatments and with pre-treatments.

## 3. RESULTS AND DISCUSSIONS

### 3.1 Physical-chemical characterization of the raw material and the finished product

In the Table 2 the characterization of the fresh fruit is presented (freeze-dried) and of the dried fruit as much in the stove as in the hybrid solar dryer. By means of the statistical study, it was determined that there was not found any statistically significant differs in the concentration of soluble polyphenols, anthocyanins and potassium in the dehydrated 'mortiño' with pre-treatments by means of the techniques studied in comparison to the freeze-drying.

### 3.2 Effect of the temperature and flow of air in a dryer of trays in the drying process

#### 3.2.1 Effect of the temperature and airflow regards to the time of drying

In the Figure 2 the curves of dehydration are shown. The three periods of dehydration can be observed; for the curve of 40 °C and 0.24 m<sup>3</sup>/s, 0.24 m<sup>3</sup>/s, the period of warm-up lasted around 5 h, the period of constant speed 20 h and the period of falling speed 5 h. One can observe that the process is extensive since the diffusion of the vapor of water is hindered by the nature of the peel of the fruits, which does not have enough capillary that allow the exit of the water.

**Table 2.** Chemical characterization of the fresh fruit and the dried 'mortiño'.

	Units	Fresh fruit	Dried fruit, dehydrated en heating stove*	Dehydrated fruit en hybrid solar dryer *
Humidity <sup>2</sup>	g/100 g PF	83.47 ± 3.02	5.01 ± 0.71	5.66 ± 0.13
Proteins <sup>3</sup>	g/100 g PF	0.60 - 0.64	-	-
Ashis <sup>3</sup>	g/100 g PF	0.33 - 0.34	-	-
Dietetic fiber, total	g/100 g PF	2.39	3.83	4.38
Extract of ethereal <sup>3</sup>	g/100g PF	0.79 ± 0.04	-	-
pH <sup>4</sup>		2.61 ± 0.06	-	-
Titrateable acidity	g Citric acids /100 g PF	7.72 ± 0.08	-	-
Galacturonic <sup>4</sup> acids	g/100 g PF	0.52 ± 0.04	-	-
Soluble <sup>3</sup> sugars				
Fructose	g/100 g PF	4.55 ± 0.06	4.12 ± 0.13	3.83 ± 0.21
Glucose	g/100 g PF	2.96 ± 0.04	2.97 ± 0.11	2.73 ± 0.13
Organic <sup>3</sup> acids				
Citric acid	mg/100 g PF	1 185.99 ± 74.53	1 113.79 ± 38.58	949.57 ± 79.06
Malic acid	mg/100 g PF	451.52 ± 14.96	436.45 ± 31.45	417.10 ± 9.80
Minerals <sup>3</sup>				
Ca	mg/100 g PF	26.74 ± 9.37	27.53 ± 3.14	26.64 ± 0.41
K	mg/100 g PF	58.49 ± 6.74	62.30 ± 1.12	52.03 ± 4.83
Mg	mg/100 g PF	15.13 ± 1.73	17.90 ± 0.64	16.99 ± 0.89
Cu	mg/100 g PF	0.06 ± 0.01	0.01 ± 0.00	0.02 ± 0.00
Zn	mg/100 g PF	0.84 ± 0.13	0.20 ± 0.00	0.18 ± 0.01
Fe	mg/100 g PF	0.49 ± 0.08	0.40 ± 0.10	0.26 ± 0.04
Na	mg/100 g PF	1.66 ± 0.05	1.74 ± 0.17	1.69 ± 0.14
Antioxidant components				
Vitamin C <sup>3</sup>	mg/100 g PF	1.27 ± 0.02	0.96 ± 0.01	1.19 ± 0.04
β-caroten <sup>3</sup>	mg/100 g PF	0.13 ± 0.00	-	-
Soluble polyphenols	mg/100 g PF	729.49 ± 112.85 <sup>1</sup>	960.72 ± 8.69 <sup>4</sup>	960.69 ± 33.74 <sup>4</sup>
Anthocyanins	mg/100 g PF	268.67 ± 33.65 <sup>2</sup>	439.56 ± 10.93 <sup>4</sup>	396.21 ± 6.25 <sup>4</sup>

<sup>1</sup>Media ± DE (n=9), <sup>2</sup>Media ± DE (n=10), <sup>3</sup>Media ± DE (n=2), Media ± DE (n=3) \* Characterization of 'mortiño' from Cotacachi province in Imbabura; PF= Peso Fresco (=Fresh weight)

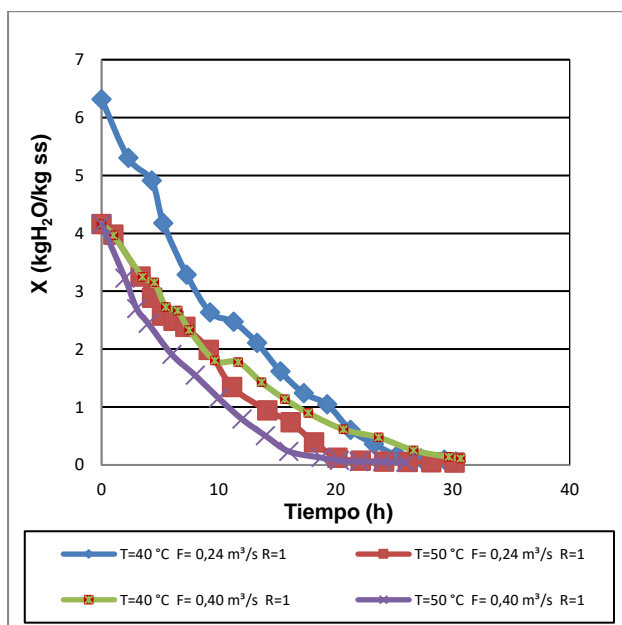


Figure 2. Graphs of drying for different established conditions when drying in dry base. a: warm-up, b: period of constant speed and c: period of falling speed

By means of the graphs of drying, the time of drying was determined in the one that the product has 5% of humidity approximately, these values are presented in the Table 3.

Table 3. Dehydration time of 'mortño' with the established conditions of temperature and airflow

Temperatura (°C)	Flow (m3/s)	Time of drying Repetition 1 (h)	Time of drying Repetition 2 (h)
40	0.24	30	30
50	0.24	24	20
40	0.40	28	31
50	0.40	24	24

Generally, to dehydrate blueberries lingering time is required, due to the waxy cuticle of these fruits, because the wax controls the speed of diffusion of water during the dehydration. It is analyzed that the restrictive factor in the dehydration is the mass transfer (Casp and April 2003, p. 337; (Ibarz et to the one, 2000, p. 99; Bhat et to the one, 2009, p. 441).

According to the ANOVA the temperature has statistically significant influence ( $p < 0.05$ ) on the time of drying. The airflow in the studied levels did not influence significantly on the same one. The higher temperature the less time of drying since the diffusion of vapor of water is directly proportional to the temperature (Ibarz et to the one., 2000, p. 95).

### 3.2.2 Temperature effect and airflow on the retention percentage of anthocyanins and soluble polyphenols in the dehydrated 'mortño'

In Table 4 can be observed that there is bigger percentage retention of anthocyanins on low temperature and airflow. This is due to that the anthocyanins are destroyed logarithmically with the increment of the temperature

(Macheix et al., 1990, p. 314; Routray y Orsat, 2011, p. 306) and when there is oxygen presence in the system, the anthocyanins demean due to the enzymatic browning (Macheix et al., 1990, p. 295).

Table 4. Percentage of anthocyanins retention to the different conditions of temperature and airflow

Temperature (°C)	Flow (m³/s)	Retention Iteration 1 (%)	Retention Iteration 2 (%)
40	0.24	54.2	45.4
50	0.24	39.1	39.9
40	0.40	34.6	35.0
50	0.40	34.2	36.6

According to the ANOVA the airflow had statistically significant influence ( $p < 0.05$ ) on the anthocyanins content. Less temperature and airflow will result in higher retention percentage.

In the Table 5, the obtained results of the percentage of retention of soluble polyphenols are presented in the dehydrated 'mortño'. It is observed that these values are relatively high, near to 100% in relation to the content in the freeze-dried 'mortño'.

Table 5. Percentage of retention soluble polyphenols in dehydrated 'mortño' under different temperature conditions and airflow

Temperature (°C)	Flow (m³/s)	Retention Replica 1 (%)	Retention Replica 2 (%)
40	2	97.8	97.7
50	2	94.1	93.9
40	4	99.9	80.9
50	4	92.2	98.0

According to the statistical analysis the flow of air and the temperature do not have it influences statistically significant ( $p < 0.05$ ) on the percentage of retention of soluble polyphenols. It could be due to that in comparison to the anthocyanins, the polyphenols is composed whose structure is more resistant to the temperature and the oxygen (Joshi et al., 2009, p. 456).

Statistically drying conditions of 40 °C and 0.24 m³/s airflow were selected in the dryer of trays.

### 3.3 Effect of the pre-treatment of immersion bath on the drying process

#### 3.3.1 Effect of the pre-treatment of immersion bath about the time of drying

The Figure 3 (to and b) it is shown the curves of 'mortño' dehydration to the different concentrations of the immersion bath.

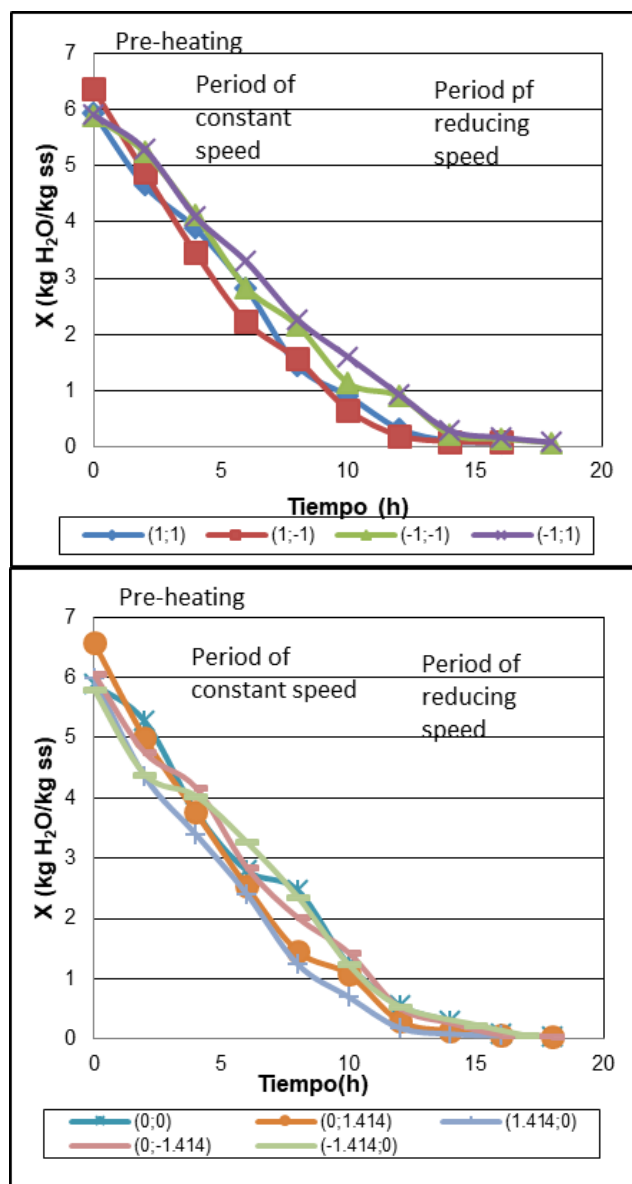


Figure 3. Curves of dehydration for the different established conditions of drying (a and b)

It can be observed that the time when it arrives to the established humidity (5 to 6%) is around 50% less than the drying without pre-treatment according to the curves of dehydration that are presented in the Figure 2. The  $K_2CO_3$  and the ethyl oleate modify the structure of the present waxes in the shell of the fruit and it facilitates the diffusion of the

vapor of the water (Christensen and Peacock, 2000, p. 213). According to the ANOVA the concentration of the bath is a variable that has statistically significant influence ( $p < 0.05$ ) about the drying time, in such a way that the more concentration results in less time of drying. In Figure 4 illustrates the response surface of maximum time of drying. Inside this area is the coordinate point (0; 0) that corresponds to the conditions of concentration of the bath of 1.55%  $K_2CO_3$ , 1.25% ethyl oleate and 3.5 min immersion. The shortest drying time can be found near the point code (1; 1), that corresponds to the conditions of concentration of the bath of 2.22%  $K_2CO_3$ , 1.78% ethyl oleate, and 4.56 min immersion.

### 3.3.2 Effect of the pre-treatment in immersion bath on the percentage of retention of anthocyanins in the freeze-dried 'mortiño' with the pre-treatment

The percentages of retention of anthocyanins in the freeze-dried fruits with the pre-treatment are presented in Table 6. Less percentages than 100% are observed, is due to that the anthocyanin is more stable between acid ( $pH < 3$ ) than between neutral or alkaline (Pascual and Sanchez, 2008, p. 282)

Table 6. Content of anthocyanins in the freeze-dried 'mortiño' with pre-treatment in immersion bath

Valor Codified	Anthocyanins freeze-dried 'mortiño' with treatment (mg/100 g PF <sup>1</sup> )	Retention of anthocyanins freeze-dried 'mortiño' with treatment (%)
(1;1)	161.9	63.0
(1;-1)	158.4	61.6
(-1;-1)	124.6	48.4
(-1;1)	129.1	50.2
(0;0) <sub>1</sub>	256.2	99.2
(0;0) <sub>2</sub>	221.7	85.8
(0;0) <sub>3</sub>	223.0	86.3
(0; 1.414)	115.0	44.7
(1.414; 0)	140.8	54.8
(0; -1.414)	230.6	89.7
(-1.414; 0)	124.8	48.5

<sup>1</sup>PF = Peso Fresco (Fresh weight)

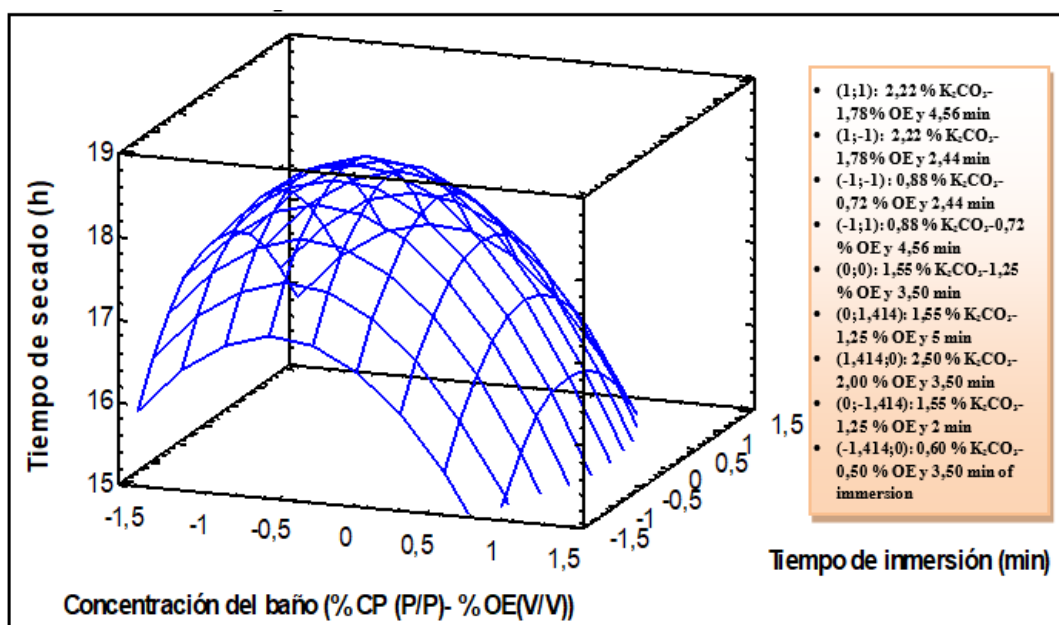


Figure 4. Response surface to study the influence of concentration of the immersion bath and time regards to the time of drying. CP= Potassium Carbonate; OE= Ethyl Oleate

According to the ANOVA the quadratic term of the concentration of the immersion bath influences statistically ( $p < 0.05$ ) on the percentage of retention of anthocyanins in the freeze-dried 'mortiño' with the pre-treatment. In the Figure 5, the response surface is shown. One can observe an area of maximum retention percentage, in which is the point of coordinated (0;0) that corresponds to concentration of the bath of 1.55% of K<sub>2</sub>CO<sub>3</sub>-1.25% of ethyl oleate and 3.5

immersion min, the same one that is the central point of the outlined experimental design. The Figure 7 corresponds to the graph of contours. It is evidenced that the maximum percentage of anthocyanins retention is located in the region among the coordinates -0.2 (2.09% of K<sub>2</sub>CO<sub>3</sub>-1.67% of ethyl oleate) and 0.4 (1.82% of K<sub>2</sub>CO<sub>3</sub>-1.46% of ethyl oleate) for the concentration of the bath, for the time of drying among the coordinates -0.7 (4.3 min) and 0.1 (3.6 min).

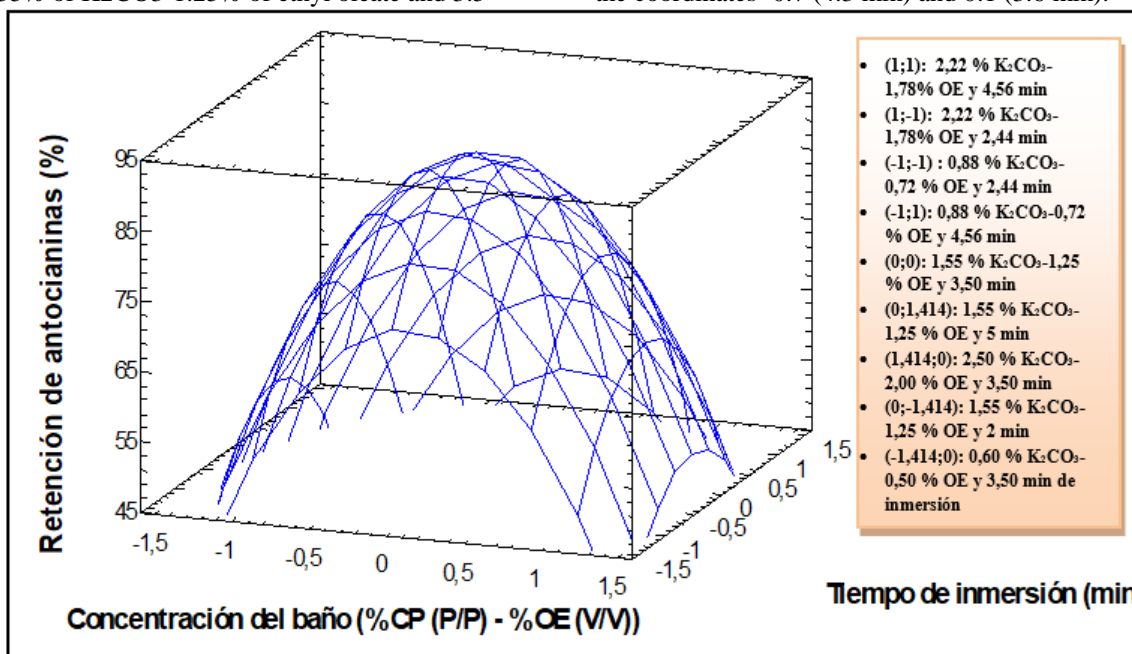
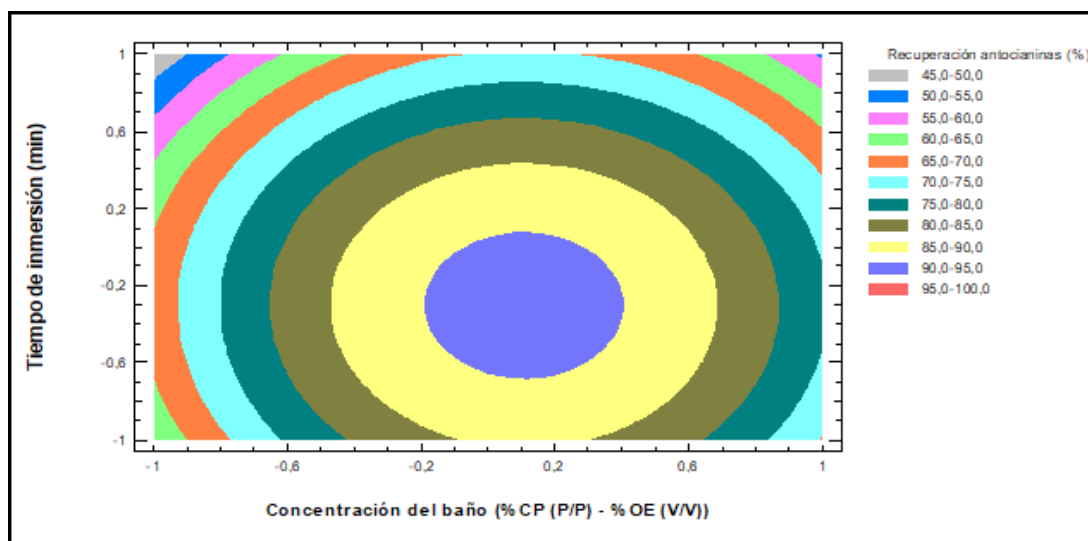


Figure 5. Response surface to the study of the influence of the concentration of the bath and time of immersion on the percentage of retention of anthocyanins CP = Carbonate of potassium; OE = ethyl Oleate



**Figure 6.** Graph of contours for the concentration of the bath and time of immersion on the percentage of anthocyanins retention in the 'mortiño' freeze-dried with the pre-treatment

### 3.3.3 Effect of the pre-treatment of immersion bath on the percentage of retention of anthocyanins and soluble polyphenols in the dehydrated 'mortiño'

The results of the percentage of anthocyanins retention and soluble polyphenols are presented in the Table 7. The percentages of retention of anthocyanins are around 50% for all the experimental conditions. According to the statistical analysis, the concentration of the bath and the time of immersion do not influence ( $p < 0.05$ ) on the concentration of anthocyanins in the dehydrated fruit.

It is also observed that there is difference in the percentage of retention of soluble polyphenols depending on the concentration of the immersion bath. This indicates that these bioactive compounds are affected by the components of the immersion bath.

**Table 7.** Percentage of retention of anthocyanins and soluble polyphenols in the 'Mortiño', dehydrated in stove for each concentration of the bath and time of immersion

Codified Value	Retention of Anthocyanins 'mortiño' dehydrated (%)	Retention of polyphenols 'mortiño' dehydrated (%)
(1;1)	52.2	73.2
(1;-1)	52.9	74.8
(-1;-1)	44.4	71.3
(-1;1)	51.8	72.8
(0;0)1	50.9	82.4
(0;0)2	46.7	81.4
(0;0)3	46.0	76.6
(0;1.414)	39.3	74.0
(1.414; 0)	42.4	68.0
(0;-1.414)	44.6	77.3
(-1.414; 0)	50.8	70.9

According to the ANOVA the quadratic term of the concentration of the immersion bath influences statistically ( $p < 0.05$ ) on the percentage of retention of soluble polyphenols in the dehydrated 'mortiño'.

In the Figure 7 the estimated response surface is presented, one can observe an area of maximum percentage of retention of soluble polyphenols. Inside this area is found the point (0; 0), that is the central point of the outlined experimental design (22 with axial points) and it corresponds to the conditions of concentration of the bath of 1.55% of  $K_2CO_3$ – 1.25% of ethyl oleate and 3.5 immersion min. Statistically the conditions of the bath and time of immersion of 1.55%  $K_2CO_3$  was chosen (P/P) – 1.25% of ethyl oleate (V/V) and time of immersion of 3.5 min.

### 3.4 Effect of the pre-treatment with $SO_2$ on the drying process

#### 3.4.1 Effect of the pre-treatment of the sulfidation regards to the percentage of retention of anthocyanins

In the Table 8, the percentage of anthocyanins retention is presented in the dehydrated 'mortiño'. One can observe that the retention percentage increased to 45% in relation to the dehydrated 'mortiño' without pre-treatment. This could be explained because the  $SO_2$  inactivates the enzyme polyphenoloxidasas that is the causing of the oxidation of anthocyanins (Macheix et al., 1990, p. 311; Clifford, 2000, p. 1065).



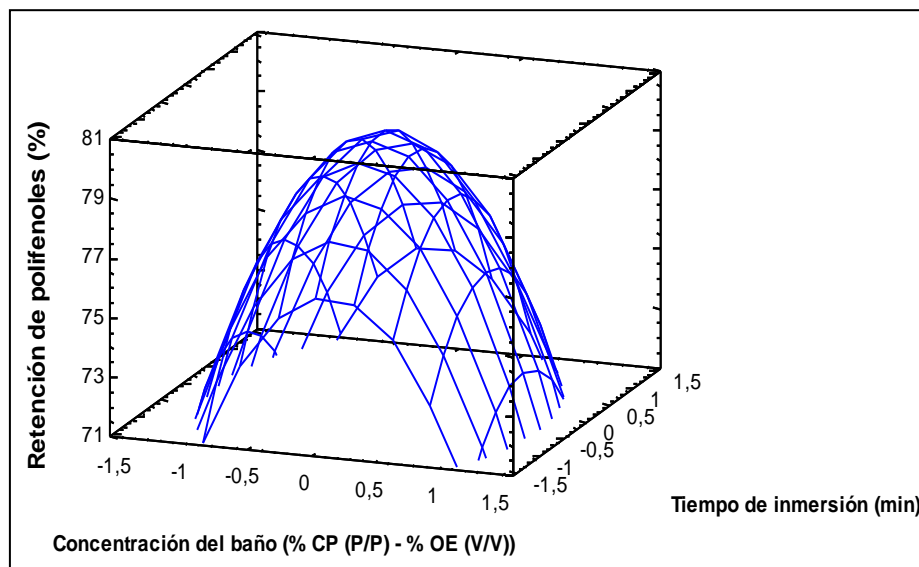


Figure 7. Response surface to study the influence of the concentration of the bath and time of immersion on the percentage of retention of soluble polyphenols

By means of the statistical study it was determined that the concentration of sulfur has statistically significant effect ( $p < 0.05$ ) on the percentage of the retention of anthocyanins. Higher concentration of sulfur will result in more retention percentage.

Table 8. Percentage of retention of anthocyanins in the dehydrated 'mortiño' with pre-treatment

SO <sub>2</sub> (ppm)	Time of sulfidation (h)	Retention de Repetition 1 (%)	Retention de Repetition 2 (%)
1 000	1	63.2	47.7
2 000	1	93.3	99.5
1 000	2	77.0	98.1
2 000	2	99.2	98.9

### 3.4.2. Effect of the sulfidation pre-treatment on the percentage of retention of soluble polyphenols

The percentage of retention of soluble polyphenols is presented in the Table 9. One can observe that these percentages are near to 100%, this indicates that the sulfidation doesn't have effect on these compounds.

Table 9. Percentage of polyphenols retention in dehydrated 'mortiño' with pre-treatments

SO <sub>2</sub> (ppm)	Time of sulfidation (h)	Retention repetition 1 (%)	Retention repetition 2 (%)
1 000	1	92.0	99.4
2 000	1	97.4	98.6
1 000	2	84.2	99.2
2 000	2	98.8	98.1

Statistically the concentration of SO<sub>2</sub> of 2 000 ppm and 2 h were chosen to expose the fruit.

### 3.5. Comparison between processes

In the Table 10, the final results of the concentration of soluble polyphenols and total anthocyanins are presented in

the dehydrated 'mortiño' and time of drying, for each one of the carried out processes. As you, one can observe in the dehydrated product in stove without pre-treatments the percentage of retention of anthocyanins in relation to the freeze-dried is of 54.2%. While in the drying of 'mortiño' with pre-treatments to the same conditions, 98.2% of retention was reached. This was achieved because due to the sulfidation pre-treatment the enzymes polyphenols oxidizers could have been deactivated (Macheix et al., 1990, p. 311; Clifford, 2000, p. 1065). In the hybrid solar dryer, it was obtained dehydrated 'mortiño' with 88.5% of retention. With pre-treatment of immersion bath, it was possible to diminish 12 hours of drying in relation to the process without pre-treatment.

Table 10. Comparison results among the processes of 'mortiño' dehydration

Characteristics	DESPT	DECPT	DSCPT
Anthocyanins (mg/100 g PF)	150.4	439.6	396.2
Polyphenols (mg/100 g PF)	573.9	960.7	960.7
Time of drying	30 h	18 h	5 days
Retention Anthocyanins (%)	54.2	98.2	88.5
Retention Polyphenols (%)	97.8	99.7	99.7

DESPT = Dehydrated in oven sin pre-treatment  
 DECPT = Dehydrated in oven con pre-treatment  
 DSCPT = Dehydrated en hybrid solar dryer with pre-treatment

### 3.6. Test of stability

The change of the concentration of the soluble polyphenols with the storing time is presented in the Figure 8. As it is observed so much for the conditions of freezing storage (-17 °C), refrigeration (4 °C) and atmosphere (25 °C; 90% HR), the concentration of polyphenols oscillates between 751 and 791 mg/100 g PF. Decrease of concentration of polyphenols is not evidenced in the storage time, in this study.

In the Figure 9, the variation of the concentration of anthocyanins is shown with the storage time. It can be observed that the anthocyanins gets lost with the time, because the presence of bioactive component in the

'mortiño', which is the most sensitive to the time and storage, conditions (temperature and humidity) (Macheix et al., 1990, p. 313).

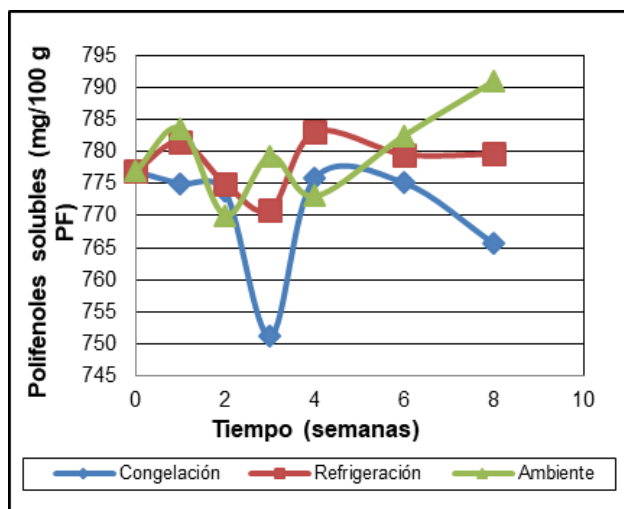


Figure 8. Concentration of soluble polyphenols vs. time storage in the dehydrated 'mortiño'

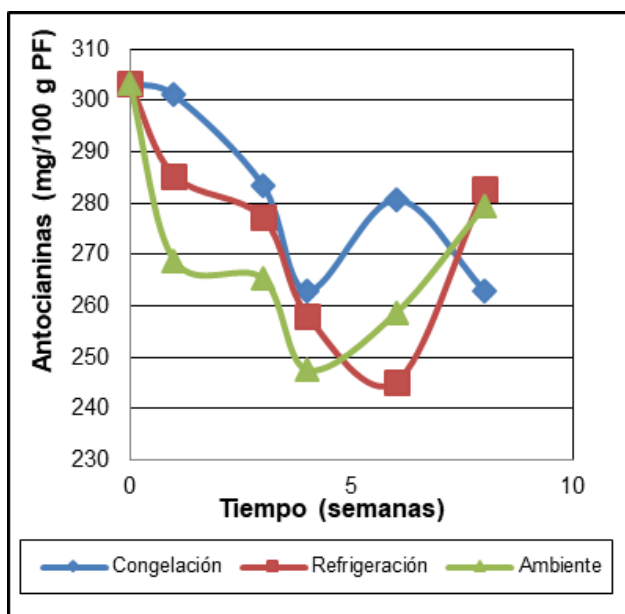


Figure 9. Concentration of soluble anthocyanins vs. time in dehydrated 'mortiño'

Table 11 shows, the kinetic parameters of the stability of dehydrated 'mortiño'.

Table 11. Kinetic parameters of the stability of the 'mortiño' dehydrated in stove with pre-treatments

Parameter	Value
<b>Constant of velocity k (week<sup>-1</sup>)</b>	
Freezing (-17 °C)	0.0343
Refrigeration (4 °C)	0.0345
Environment (25 °C y 90 % HR)	0.0419
<b>Frequency factor k' (week<sup>-1</sup>)</b>	0.1440
<b>Activation energy de E (KJ/mol)</b>	31.200
<b>Time of usefulness of the product by storing in 18 °C</b>	4 months y 11 days

In the three storage conditions the humidity increases. This can be due to the characteristic properties of permeability of the used packing.

It was determined that the initial color of the 'mortiño' dehydrated in a stove with pre-treatments was N99C40M50 (N = black, M = Magenta y C = Cyan), what means that the dehydrated product presented a black-brown color. No change of color of the product was detected during the 2 months of storage.

### 3.7 Sensorial analysis

It was found that the pre-treatments do not influence statistically ( $p < 0.05$ ) in the color, flavor, texture and presence of strange flavors in the dehydrated 'mortiño', not from the hybrid solar dryer nor the product from the stove.

## 4. CONCLUSIONS

In the dehydrated 'mortiño' in stove with 40 °C and flow of air of 0.24 m<sup>3</sup>/s, showed the maximum percentage of retention of phenolics compound (54.2% anthocyanins and 97.8% of soluble polyphenols) in relation to the freeze-dried fruits.

The immersion of the 'mortiño' for 3.5 min in the bath with a concentration of 1.55% K<sub>2</sub>CO<sub>3</sub> (P/P) – 1.25% of ethyl oleate (V/V), was possible to diminish the dehydration time 12 hours in the stove.

The pre-treatment of immersion bath evidenced that statistically has a determining effect on the percentage of retention of soluble polyphenols.

The sulfidation to the fresh fruits with 2 000 ppm of concentration of SO<sub>2</sub> and 2 h of exhibition allowed to increase the percentage of retention of anthocyanins from 54.2% to 98.2%. The dehydrated 'mortiño' in the hybrid solar dryer conserved 88.5% anthocyanins in relation to the freeze-dried fruit.

The dehydrated 'mortiño' in stove and in the hybrid solar dryer, had a hard texture, but it does not present any strange flavor due to the pre-treatments.

The dehydrated 'mortiño' has a time of useful life of 4 months and 11 days.

## REFERENCIAS

AOAC, 2007, "Official Methods of Analysis of AOAC International", 18 edition, Maryland, USA.

Asp, N., Johansson, C., Hallmer, H., y Siljestroem, M. (1983). Rapid Enzymatic Assay of Insoluble and Soluble Dietary Fiber. *Journal of Agricultural and Food Chemistry*, 31 (3), pp. 476 - 482.

Bhat, N., Desai, B. y Suleiman, M. (2006). Grapes and Raisins. En Barta, J., Cano, M., Gusek, T., Sidhu, J. y Sinha N. *Handbook of fruits and fruit processing*, (pp. 439-452). USA: Blacwell Publishing.

Casp, A. y Abril, J. (2003). *Proceso de conservación de alimentos*. (2da. ed.). Madrid, España: Mundi-Prensa.

Clifford, M. (2000). Anthocyanins – nature, occurrence and dietary burden. *Journal of the Science of Food and Agriculture Review*, 80, pp.1063-1072.

Christensen, L. y Peacock, L. (2000). *Raisin Production Manual*. California.

DECAB. (2004a). Método modificado y validado por el DECAB del paper: Macrae, R. (1988), "HPLC in food analysis", Academic Press, Segunda Edición, Gran Bretaña.

DECAB. (2004b). Método modificado del Manual de la Columna ASTEC NH2 series de Advanced Separation Technologies Inc. USA.

Giusti, M. y Wrolstad, R. (2001). Characterization and measurement with UV-visible spectroscopy. *Current protocols in food analytical chemistry*, John Wiley & Sons, pp. 1-13.

Heinonen, M. (2007). Antioxidant activity and antimicrobial effect of berry phenolics a Finnish perspective. *Journal of Nutritional Food*, 51, pp. 684-691.

Ibarz, A., Barbosa, G., Garza, S. y Gimeno, V. (2000). *Métodos experimentales de la ingeniería alimentaria*. Zaragoza, España: Acribia S.A.

Joshi, A., Rupasinghe, H. y Khanizadeh, S. (2009). Impact of drying processes on bioactive phenolics, vitamin C and antioxidant capacity of red-fleshed apple slices. *Journal of Food Processing and Preservation*, 35, pp. 453-457.

Macheix, J., Fleuriot, A. y Billot, J. (1990). *Fruit phenolics*. Florida, United States: Boca Raton.

Mujumdar, A. (1995). *Handbook of industrial drying*. (2da. ed.). New York, United States of América: Marcel Dekker.

Pascual, S. y Sánchez, M. (2008). Anthocyanins: from plant to health. *Phytochem Review*, 7, pp. 281-299.

Popenoe, H., King, S., León, J., Kalinowski, L. (1989). *Lost Crops of the Incas*. Washington, USA: National Academy Press.

Routray, W. y Orsat, V. (2011). Blueberries and Their Anthocyanins: Factors Affecting Biosynthesis and Properties. *Comprehensive Reviews in Food Science and Food Safety*, 10, pp. 303-320.

Vasco, C., Kaisu Riihinen, Ruales, J. y Afaf Kamal-Eldin. (2009). Chemical Composition and Phenolic Compound Profile of 'mortiño' (*Vaccinium floribundum Kunth*). *Journal of Agricultural and Food Chemistry*, 57(18), pp. 8 274 – 8 281.



**García Ruiz Almudena.** Dra. en Ciencia y Tecnología de los Alimentos, Departamento de Ciencias de los Alimentos y Biotecnología, Facultad de Ingeniería Química y Agroindustrial, Escuela Politécnica Nacional, Quito (Ecuador) Prometeo.



**Ruales Nájera, Jenny Cumanda.** Profesor principal del Departamento de Ciencia de Alimentos y Biotecnología de la Escuela Politécnica Nacional. Ingeniera Química de la Escuela Politécnica Nacional. Licenciada en Ingeniería de Alimentos y Ph.D. de la Universidad de Lund-Suecia. Las líneas de investigación de interés son la valorización de materiales nativos, aislamiento de principios activos y desarrollo de alimentos funcionales. Los polifenoles y carotenoides son de especial interés.

Aplica en sus investigaciones tecnologías convencionales y tecnologías emergentes. Otra línea de interés es toxicología de alimentos, investiga la presencia de metales pesados (As, Cd) en materias primas y alimentos. Posee un RG de 23.64 y un h-index de 15 al 13 de junio del 2016.