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Effect of hot air, vacuum and infrared drying methods on quality of rose hip (*Rosa rubiginosa)* leathers

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Summary For the development of fruit leathers, a formulation containing rose hip pulp, sucrose and citric acid was used as initial material for the drying process. Three dehydration techniques were tested: forced hot air, infrared and vacuum, all carried out at 60 and 70 °C. All methods led to flexible, translucent fruit leathers at both temperatures. Colour and water activity were not affected by the dehydration method nor by the temperatures tested. Nutritional parameters such as antioxidant capacity (TEAC) and content of phenolic substances were measured. The best retention was achieved with vacuum drying at 60 °C being of 57.5% and 25.1%, respectively. ESEM observations were conducted to assess the effect of drying methods and conditions on microstructure of leathers. Various extents of sucrose crystallisation were inferred from surface images. Cross-sectional micrographs showed that the size of pores was affected by the drying technique but not by temperature in the range studied.

Keywords Dehydration, infrared drying, rose hip, vacuum drying.

Introduction

Dehydration is a well-known process allowing foodstuffs to be preserved for long periods of time. However, not all drying methods maintain the nutritional characteristics of the fresh product in the dried condition because quality depends on the duration and intensity of the drying process (Cheng & Mujumdar, 2008). Most nutritional compounds present in fruits and vegetables are thermolabile and oxygen sensitive (Damodaran *et al*., 2010). Adults and children have increased in recent times the consumption of fruits and vegetables in search of a healthier lifestyle, so the current trend in food industry focuses more in developing vitamin-rich snack-type products with high content of natural fruit solids (FAO/WHO, 2004). Most snacks are processed by frying, extrusion and baking although, in the case of fruits and vegetables, they are generally transformed by drying (Lusas & Rooney, 2001). Therefore, technologies that reduce drying time and exposure to oxygen may be utilised in industry as quality-driven alternatives for the manufacture of snack products. Fruit leathers were originally known as homemade preparations and have turned into industrial products. They are today a scientific research topic: studies aim at developing and describing several formulations to improve the understanding of their gelation mechanism, drying-related properties and quality, in order to provide variety to diet and optimise the retention of nutritional properties under conditions likely to be applied in industry (Vijayanand *et al*., 2001; Demarchi *et al*., 2013a). On the other hand, rose hip fruits (*Rosa rubiginosa)* cannot be eaten as whole fruits in their fresh state (Mabellini *et al*., 2011). Rose hip pulp possesses a high content of ascorbic acid, phenolic compounds (tocopherols, flavonoids, tannins), pectin and minerals (Ilbay *et al*., 2013). The main objective of this work on rose hip leathers is to comparatively evaluate quality changes produced by three dehydration methods such as forced air, vacuum and infrared drying, in order to find adequate techniques and conditions that would be helpful for research at academia, as well as for applications in industry.

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Materials and methods

Materials

Rose hip pulp was provided by AER El Bolsón INTA (National Institute of Agricultural Technology), El Bolsón, Province of Río Negro, Argentina. This pulp

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was obtained by boiling whole fruits with water during 30 min to soften the pulp, inhibit enzymatic browning, reduce the microbial load and facilitate separation of pulp from seeds and the stiff hairs covering them. This is very important, as hairs are abrasive and cause severe itch in contact with skin and mucosae or a more severe condition such as keratitis (Venkatesh *et al*., 2005). The flesh so obtained was mashed and passed first through a 2-mm sieve and second through a 0.5-mm sieve. If the soluble solid content results lower than 12ºBrix, the pulp is concentrated further to 14ºBrix by evaporation. For proper storage and transportation, the pulp was frozen at −20 °C.

Rose hip leather formulation

The rose hip pulp was characterised in previous work by Mabellini *et al*. (2011). Among their results, a substantial amount of high ester pectic substances, sufficient to allow for the gelation process, was determined. To improve the conditions for the high-methoxyl acid–sugar gelation, sucrose was added and the original pH of the pulp (about 3.75) was reduced to 3.3. The adjustment was made following the pH value through a pH meter after small amounts of citric acid powder were added to the formulation.

In the formulation intended for leathers, constituents such as rose hip pulp, sucrose and citric acid (Química Anedra) were combined in mass proportions of 78:21:1, respectively. The amount of each component was proportioned to obtain a pH of 3.3 and a solids concentration above 65% at the drying endpoint.

Drying methods

Hot air drying was carried out in a bench-scale tray dryer with *in situ* weighing at two temperatures, 60 and 70 °C, using an air velocity of 2.5 m s^{-1}. Both the temperature and air velocity were automatically controlled with corresponding microprocessor-based devices (Quintero Ruiz *et al*., 2012).

Vacuum drying was conducted in an airtight cabinet equipped with a digital temperature control (Arcano, China), which was connected to a vacuum pump (Dosivac DV95, Buenos Aires, Argentina). The absolute pressures for the drying process were of 21.0 kPa for 60 °C and 35.0 kPa for 70 °C. Both pressures were selected so that oven temperature remains below the boiling point of water, thus allowing liquid diffusion to be the prevailing mass transfer mechanism within the solid, improving the textural appearance of the product.

Infrared drying was conducted in a digital moisture analyser (Mettler LP16, Greifensee, Switzerland), being 60 and 70 °C the experimental temperatures chosen, as in the methods described above.

The amount of formulation needed to carry out the drying experiments was calculated theoretically by considering first a final thickness of 2 mm, for this value was found earlier to provide high sensory acceptability scores (Leiva Díaz *et al*., 2009), and second, that shrinkage during dehydration is only moisture content dependent, at least in the range of temperatures covered (Ochoa *et al*., 2002). To conduct this calculation, the following data were used: moisture content of formulation (W_i) and of final leather (W_f), both on a dry basis, 2.125 and 0.25 kg water kg^{-1} dry matter, respectively, leather density, 745 kg m^{-3}, final thickness, 2 mm as mentioned above, sample holder diameter and height 0.060 and 0.015 m, respectively. Assuming that the dry matter is conservative during the drying process, the final mass of leather (m_f) was estimated first with the data provided above, whereas Equation. (1) was used to reach a calculated initial formulation mass (m_i), which was found to be 0.01 kg.

$$m_i = m_f \times \frac{(1 + W_i)}{(1 + W_f)}. \quad (1)$$

Determination of total dry matter

Samples of 7 g were placed in a temperature-controlled mechanical convection oven set at 105 °C until reaching constant weight as determined by analytical balance (precision 0.1 mg). This procedure follows the AOAC method 925.45 (AOAC, 1998).

Water activity determination

Values of a_w were measured at 25 °C by the hygrometric method AOAC 978.18 (AOAC, 1998) with an AquaLab 3TE water activity meter (Decagon Devices Inc., Pullmann, WA, USA). A sample is introduced in a sealed chamber, and the technique is based on bringing the headspace air relative humidity to equilibrium with the product a_w, being this equilibrium attained without modifications in the a_w of the sample due to the large ratio of sample mass to headspace air mass. Therefore, by measuring the air equilibrium relative humidity (h_{re}) with an accurate chilled-mirror dew point sensor and a noncontact infrared thermometer, the water activity of the product was determined as $a_w = h_{re}$.

Colour changes

A Konica-Minolta CR-400 tristimulus colorimeter (Osaka, Japan) was employed. Measured values were read in the L* a* b* CIELAB scale and utilised to calculate parameters such as hue angle $h^* = \tan^{-1}(b^*/a^*)$ and Chroma $C^* = (a^* + b^*)^{1/2}$, which provide

information on colour and colour saturation, respectively (MacDougall, 2002). The optical sensor was placed at the upper side of the sample (i.e. the side that was not in contact with the drying tray bottom), and measures were made after the sample was brought to room temperature.

Preparation of extracts from samples

For the determination of Trolox-equivalent antioxidant capacity (TEAC) and phenolic substances, ethanolic extracts of 0.2 g mL^{-1} were prepared from a 2 g fruit leather sample, which was suspended in 10 mL ethanol 96º (Química Anedra) under stirring for 40 min at 4 °C. The extract was then centrifuged for 10 min at 10 000 ***g*** at 4 °C to retain the supernatant. Liquid extracts were kept in Eppendorf micro test tubes and stored at −80 °C until using.

Trolox-equivalent antioxidant capacity (TEAC) by ABTS*$^{+}$ assay

Determinations followed the ABTS*$^{+}$ radical cation decolorisation method (Re *et al*., 1999) as described in a previous work on apple leathers (Quintero Ruiz *et al*., 2012). The 2,2′-azino-bis(3-ethylbenzothiazoline-6-sulphonic acid) (ABTS) and potassium persulfate ($K_2S_2O_8$) were provided by Sigma-Aldrich LLC Co. (St. Louis, MO, USA). ABTS was dissolved in water to a 7 mM concentration, then the radical cation (ABTS*$^{+}$) was produced by reacting that ABTS solution with 2.45 mM potassium persulfate (final concentration) and allowing the mixture to remain in the dark at room temperature for 12–16 h before use. From this concentrated solution, an ethanolic ABTS*$^{+}$ solution was prepared to read an absorbance of 0.7 at 734 nm, which was considered as the base value. For a sample measurement to be valid, the final value of absorbance after a reaction time of 6 min must decrease by 20–80% from the base value. Determinations were carried out before and after the drying process, and triplicate ethanolic extracts were prepared from each sample. All extracts were placed in a freezer at −80 °C as soon as they were prepared at the time of the drying experiment and remained there until the entire experimental programme was completed. Then, the frozen samples so accumulated were analysed in the same laboratory session. A 10-point calibration curve was prepared with a Trolox standard solution (2.22 mM) (Sigma-Aldrich LLC Co.) to record absorbance readings for known concentrations (Absorbance = 4623.6* (μmol trolox) + 2.6027). Using this calibration curve, absorbance readings of the samples were expressed as Trolox equivalents (μmol trolox g^{-1} sample dry matter), also called as Trolox-equivalent antioxidant capacity or TEAC.

Total phenolic compounds by Folin–Ciocalteu colorimetry

A colorimetric assay using the Folin–Ciocalteu reagent was employed (Waterhouse, 2001; Massolo *et al*., 2011). A volume of 50 μL of the diluted liquid extract, prepared as explained in 2.7, was added to a reaction tube containing 2300 μL of water and 50 μL of aqueous Folin–Ciocalteu solution (1:1). After 5 min of reaction, 100 μL of sodium carbonate solution 20% w/v in NaOH 0.1 N was added to the reaction tube. After mixing each of all tubes in a vortex device, reaction took place in the dark at room conditions during 90 min; the absorbance was read at 765 nm. To quantify the phenolic substances in the samples, a 10-point calibration curve was measured starting from a 0.24 μg mL^{-1} gallic acid solution. Therefore, the results were expressed as mg gallic acid equivalents per 100 g of sample dry matter.

Environmental scanning electron microscopy (ESEM)

The analysis was performed in a FEI QUANTA 200 scanning electron microscope (Hillsboro, OR, USA), operating in environmental mode (ESEM), using a gaseous secondary electron detector (GSED). Fruit leathers were cut in 12 mm^3 sections and mounted directly on a metal support dish, to be scanned at 8 °C and 1.8 torr, at 20 kV acceleration voltage. The surface and cross sections of the samples were analysed.

Statistical analysis

Triplicate determinations were carried out in all experiments. The statistical software OriginPro v 8.1 (Origin, 2009) was used for data analysis and model fitting. The statistical significance was assessed by one-way analysis of variance (ANOVA), as well as by the Tukey′s test ($P < 0.05$) (Bower, 2009),to detect significant differences between treatments.

Results and discussion

Drying conditions

Triplicate drying experiments were carried out at 60 and 70 °C for each of the three methods utilised, to give a total of 18 drying tests. The final appearance of rose hip leather for dried by hot air at 70 °C is shown in Fig. 1. For hot air (convective heat transfer), vacuum (predominantly conductive heat transfer) and infrared drying (radiation heat transfer), the drying time, that is, the period of time elapsed from drying start to the attainment of a final moisture content of 0.25 kg water kg^{-1} dry matter, varied in the range of 165 to 460 min, being the shortest drying time found

Figure 1 Rose hip leather produced by hot air drying at 70 °C.

for vacuum drying at 70 °C and the longest, for hot air at 60 °C (Table 1). Hence, higher temperatures lead to shorter drying times in fruit leathers as found by Bains *et al.* (1989), and the method providing the largest driving force for mass transfer was vacuum drying, in agreement with authors that compared mass transfer mechanisms in similar foods (Drouzas *et al.*, 1999; Jaturonglumlert & Kiatsiriroat, 2010).

Physicochemical parameters such as the final water activity and an organoleptic indicator such as the surface colour did not statistically differ among the temperatures nor between the drying methods tested, as observed in Table 1. As a complementary evaluation, water activities were compared to values calculated by desorption isotherm model for the rose hip-based formulation (Demarchi *et al.*, 2013b) at the same moisture content. Both sets of values were similar, leading us to conclude that effect of the drying method on the sorption behaviour was negligible at least in the low moisture zone.

Trolox-equivalent antioxidant capacity (TEAC)

The value of TEAC in the formulation before drying was 59.19 μmol Trolox g^{-1} dry matter. In Fig. 2,

Table 1 Drying-related parameters of the rose hip leathers developed by three drying methods

Drying method (heat transfer mechanism)	Temperature (°C)	Time (min)	a_w^+	Chroma(C*)$^+$	Hue (h*)$^+$
Hot air (convective)	60	460	0.58 ± 0.02^c	15.95 ± 1.26^a	29.37 ± 0.10^b
	70	280	0.56 ± 0.02^c	15.45 ± 1.24^a	28.80 ± 0.94^b
Vacuum (conductive)	60	260	0.56 ± 0.02^c	15.59 ± 1.67^a	31.28 ± 1.12^b
	70	165	0.56 ± 0.01^c	15.71 ± 1.99^a	31.17 ± 0.94^b
Infrared (radiative)	60	346	0.56 ± 0.06^c	17.29 ± 1.15^a	26.88 ± 1.30^b
	70	270	0.57 ± 0.05^c	14.07 ± 2.21^a	27.62 ± 0.85^b

Values are the mean of triplicates ± standard deviation.
Equal lower case letter in column means no significant difference in Tukey's test ($P < 0.05$).

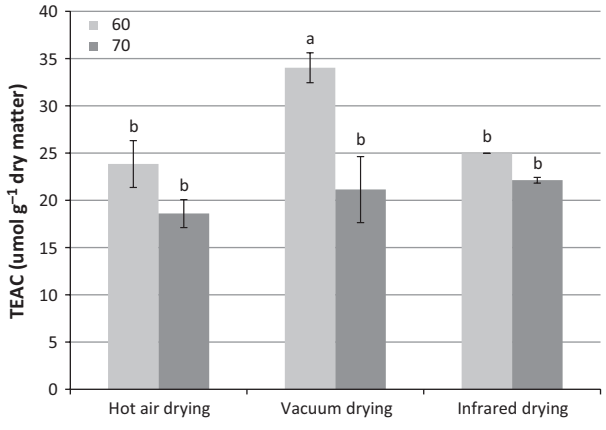

Figure 2 Average content of Trolox-equivalent antioxidant capacity in rose hip leathers dehydrated at 60 and 70 °C by three drying methods (initial value in sample 59.19 μmol Trolox g^{-1} dry matter). Bars with equal letters imply no significant differences (Tukey, $P < 0.05$).

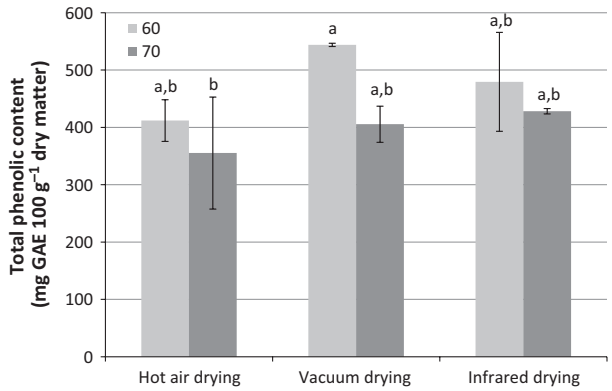

Figure 3 Average content of phenolic compounds in rose hip leathers dehydrated at a 60 and 70 °C (initial value in sample 2167.66 mg per GAE per 100 g dry matter). Bars with equal letters imply no significant differences (Tukey, $P < 0.05$).

there is an interacting effect of temperature and drying method, being TEAC retention of 57.5% in vacuum drying at 60 °C, significantly higher (Tukey, $P < 0.05$) than for the remaining combinations. Therefore, no significant differences were found among TEAC values after hot air at 60 °C and 70 °C, vacuum at 70 °C and infrared drying at 60 and 70 °C.

Total phenolic compounds

The initial value for the formulation before drying was 2167.66 mg of GAE per 100 g dry matter. The contents after drying, ranging from about 350 to 550 mg of GAE per 100 g dry matter, are presented in Fig. 3. Hence, reductions were substantial, varying between 74.5 and 83.3%, denoting that all methods used produced considerable losses. The only significant difference occurred between hot air drying at 70 °C (355.378 mg of GAE of 100 g dry matter) and vacuum drying at 60 °C (544.016 mg of GAE per 100 g dry matter), being the latter method and experimental condition that which leads to the highest retention of phenolic compounds (25.1%). As for TEAC, differences were caused only by interacting effects.

ESEM images

Given that the formation of fruit leathers implies the restructuring of a pulp-based formulation by pectic gelation induced by dehydration, a porous and disordered microstructure was observed. When comparing images of samples dried at 60 and 70 °C, the effect of temperature was negligible. Concerning the effect of drying method, Fig. 4 shows the micrographs taken at 60 °C in the cross-sectional view of the sample, that is, where the sample thickness is magnified; micrographs (a) hot air, (b) vacuum and (c) infrared show small pores in all of them, possibly denoting the places where water was present in the formulation. Restructuring by gelation led to an amorphous, irregular arrangement determined by a formulation that is partly a solution and partly a well-mixed suspension. Larger pores were observed in the vacuum-dehydrated leathers.

With respect to the surface view of samples, formation of rectangular or square-shaped sugar crystals was observed. This was the result of the initially high content of soluble solids, notably sucrose. Concerning the size of sugar crystals in the hot air-dried sample

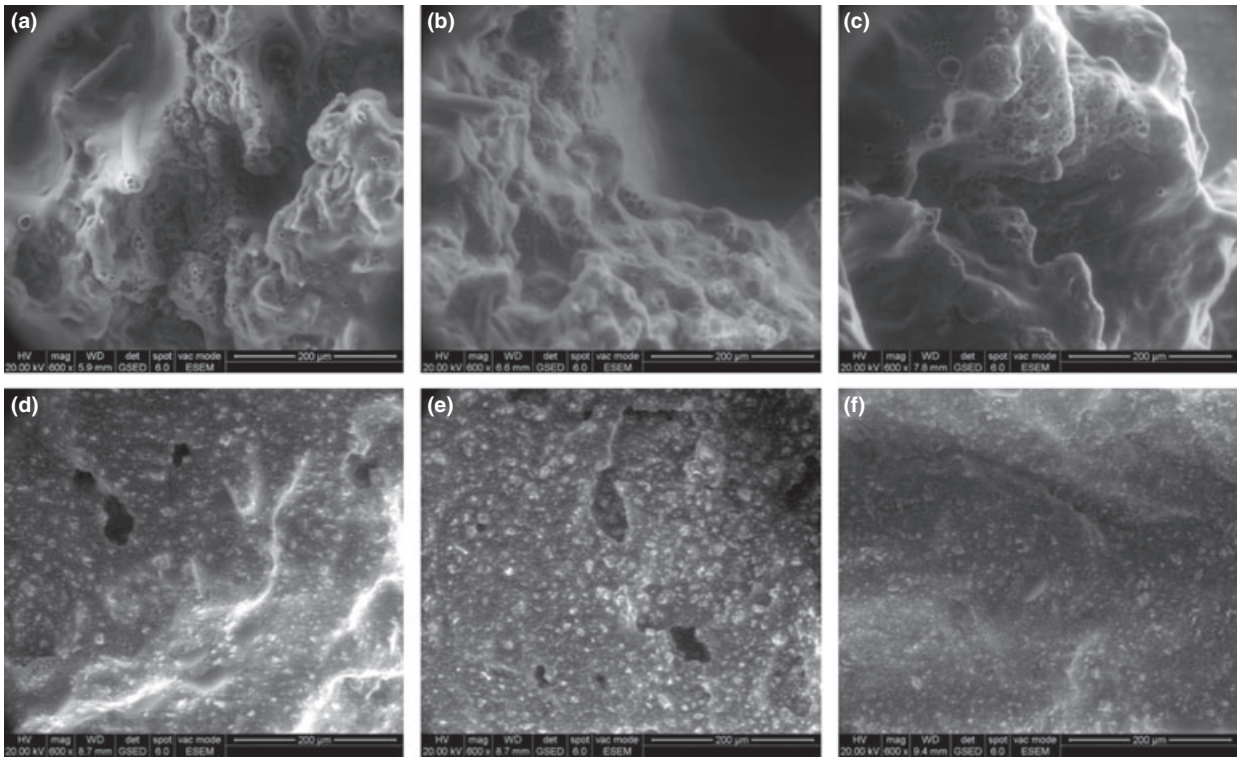

Figure 4 ESEM micrographs at 600×. Cross-sectional view of samples: (a) hot air (b) vacuum (c) infrared. Surface view: (d) hot air (e) vacuum and (f) infrared.

(micrograph (d), it was comparatively higher than in the samples produced by the other drying methods, and the surface appeared rougher, possibly as a result of the airflow passing over the sample at high velocity. Voids (dark zones) were also observed, which constitute the pores through which the water was diffusing out the matrix (e). In the infrared-dried sample, the surface was smoother and the identification of crystals was more difficult (f).

Conclusions

Rose hip leathers were produced by three drying methods differing in the predominant heat transfer mechanism, that is, conduction, convection and radiation.

Physical and organoleptic characteristics such as water activity and surface colour are dependent on the leather moisture content and not, in the range studied, on drying temperature (60–70 °C) nor on the drying method.

All drying methods reduced the content of phenolic compounds and antioxidant capacity.

For the nutrition-related characteristics studied in this work, an interacting effect of method and temperature allowed to select vacuum drying at 60 °C as the combined condition leading to the highest retention.

A study of the microstructure by environmental scanning electron microscopy allowed to observe the formation of sugar crystals with variable size and amount, on the surface. Concerning the cross-sectional views, some variation in pore size was detected. Both aspects are influenced by drying method and not, in the range studied, by drying temperature.

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