ARTICULO DE INVESTIGACIÓN: BIOTECNOLOGIA AGRICOLA Y DE ALIMENTOS REVISTA DE CIENCIAS AGRÍCOLAS
Volumen 33(2):55-61 BY-NC ISSN Impreso 0120-0135 e-ISSN 2256-2273 doi:http://dx.doi.org/10.22267/rcia.163302.52

Calidad de productos deshidratados de manzana adicionados con componentes activos.

Quality of dried apple products added with active compounds

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Citar: CORTÉS, M.; CHIRALT, A.; ARANGO, J.C. 2016. Quality of dried apple products added with active compounds. Rev. Cienc. Agr. 33(2): 55-61. doi: http://dx.doi.org/10.22267/rcia.163302.52.

Recibido: marzo 18 de 2016. Aceptado: Septiembre 14 de 2016.

RESUMEN

El objetivo de la presente investigación fue evaluar los atributos de calidad de manzanas secadas por aire convencional y liofilizadas, previo tratamiento de impregnación (IV) con vitamina E. El proceso de secado por aire convencional (PSAC) se realizó en un secador de bandeja a 40ºC, humedad relativa de 59±7% y velocidad del aire de 0,7 m/s; mientras que, el proceso de liofilización (PL) se realizó a presión de vacío de 1.2 x 10^{-3} kPa, temperatura del condensador de -45ºC y temperatura de la bandeja de 25ºC. La cuantificación de vitamina E se realizó por cromatografía de gases con detector de ionización de llama sobre extractos de las muestras en hexano. Las manzanas PSAC y PL presentaron 0,72±0,12 y 1,34±0,14mg dl-α-tocoferol acetato/g; 12,6±1,7 y 7,9±2,0% humedad, respectivamente. Los productos PSAC presentaron pardeamiento, mientras que los productos PL fueron más claros (>L*), verdosos (<a*), menos amarillos (<b*) y menos saturado (<Cab*). Los productos obtenidos por PL presentaron una textura crujiente; mientras que los productos PSAC fueron gomosos. Los procesos integrados IV + PSAC e IV + PL, no implican una alteración notable de la vitamin E incorporada; por lo tanto, es un buen procedimiento para incrementar la vida útil de los productos sin pérdidas del valor nutricional.

Palabras clave: Alimentos funcionales, impregnación al vacío, frutas, vitaminas.

ABSTRACT

The aim of this research was to assess the quality attributes of air dried and lyophilized apples previous a pretreatment of vacuum impregnation (VI) with vitamin E. The air drying process (ADP) was carried out

in a tray dryer at 40ºC, relative humidity of 59±7% and air speed of 0.7m/s. The lyophilization process (LP) was in turn conducted with a vacuum pressure of 1.2 x 10^{-3} kPa, condenser's temperature of -45ºC, and the tray's temperature of 25ºC. The vitamin E was quantified using gas chromatography with a flame ionization detector on the extracts of hexane samples. Apples ADP and LP had 0.72 ± 0.12 and 1.34 ± 0.14 mg dl-α-tocopherol acetate/g; 12.6±1.7 and 7.9±2.0 % moisture, respectively. Products resulting from ADP showed browning, whereas those resulting from LP were lighter in color (>L*), greenish (<a*), less yellow (<b*) and less saturated (<Cab*). Products obtained through LP had a crunchy texture, while those obtained by ADP were rubbery. The integrated processes VI+ADP or VI+LP do not cause a significant alteration of the vitamin E. Therefore, this is a good procedure for extending the shelf life of products without decreasing their nutritional characteristics.

Palabras clave: Functional foods, vacuum impregnation, fruits, vitamins.

INTRODUCTION

The vacuum impregnation technique (VI) has been described (Fito, 1994) through the hydrodynamic mechanism (HDM). It is presented as an applicable alternative in the food industry for the production of new functional foods by the incorporation of physiologically active components (PAC) (Fito *et al*., 2001). The fast changes in composition caused by VI, allow ensuring a better stability of the product (decrease of pH and a_w, incorporation of anti-microbial o anti-brown) (Betoret *et al*., 2015; Neri *et al*., 2016) or improvement in some quality attributes (global product flavor, enrichment with specific nutrients) (Peña *et al*., 2015). The aim of this work was to evaluate the quality of dehydrated apple products with functional properties, by incorporation of vitamin E, by using the VI technique, the determination of vitamin level in function of the reference daily value (RDV) and the evaluation of parameter color and texture.

MATERIALS AND METHODS

Apples (Granny Smith var.) purchased in local markets in Valencia, España, were used. Homogeneity in size, shape, and apparent ripeness were important for its selection; they were stored at 4ºC before being used. Slices of apples (f_{int}: 22.4mm, $f_{ext:}$ 66,7mm, thickness: 10mm), with a weight of 25g approximately, were used for the air drying process (ADP) and quarter slices apples, but 5mm thick and a weight of 3g approximately, were used for the lyophilization process (LP). Moisture content (X_w) was determined according to AOAC (1980) method 20.013; the water activity (a_w) was determined with a dew point hygrometer AquaLab Decagon CX-3; soluble solids (ºBrix) were measured with an Atago refract meter NAR-3T, thermostat 20ºC; The density of emulsions (r^E) was measured to 25ºC with a digital densimeter DA - 110M Mettler Toledo.

The color was tested with a spectrocolorimeter Minolta SpectraMagic CM-3600D, illuminant D65 for the 10º observer. The color parameter values of lightness (L*), chromaticity greenness/redness (a*), chromaticity blueness/yellowness (b*), saturation (C_{ab}*) and hue (h_{ab}*) were recorded for each product. The translucency of the products ADP were evaluated using the Kubelka–Munk Theory (Lana *et al*., 2006). Thirty-six samples were evaluated for each product and in the case of the products ADP, it was determined at four points (90º) per sample.

The mechanical tests were carried out at 25ºC with a Universal Texture Analyser TA.XT2 (Stable Micro Systems). The samples ADP were subjected to a penetration test (metal probe 4mm diameter, deformation rate 2mm/s, deformation distance 10mm). From force-distance curves obtained, distance (D_r) and force fracture peak (F_r), and more initial slope (j) to relate with the product

extensibility module. The mechanical test on the products LP was carried out with plastic probe 10mm diameter, strain 95%, deformation rate 2mm/s. From the force–% relative deformation curves obtained, force deformation at 95% ($F_{95\%}$), force deformation constant (F_f) and the % initial relative deformation (g_F^o) and end (g_F^f), keeping F_f. Six samples were evaluated for each product and in the case of ADP, it was determined at three points (120º) for the sample.

For the products LP, the dl-a-tocopherol acetate (0.065 %) was emulsified in an isotonic glucose solution (9ºBrix) containing: Tween 80 (0.051%), Span 60 (0.049%), Arabic gum (0.1%) and $CaCl_2$ (1.7%). For the products ADP, the dl-a-tocopherol acetate was 0.132% and changed the $CaCl_2$ for NaCl (0.051%) to help the charge stabilization (Sarkar *et al*. 2016). The emulsions were prepared in a vacuum homogenizer (Ultraturrax T25 - Janke & Kunkel IKA - Labortechnik) for 20 minutes to 24000rpm. A glass container (250mL) with recirculation and jacket refrigeration was adapted.

VI experiments were carried out in specially designed equipment (Salvatori *et al*., 1998). Sample impregnation was carried out by its immersion in each emulsion for 20min, applying vacuum (50mbar) for the first 10min. The volume fraction of the emulsion, X ($m^3_{emulsion}/m^3_{fruit}$) was evaluated using equations one. The before (Mº) and after (M^{IV}) weights of the impregnation were quantified. The volume of fresh apple (Vº) was evaluated from apparent density (ρ_{apm} = 802kg/m^3). The effective porosity of the sample (e) available to the HDM action was evaluated according to equations two and three, under the considerations that in the impregnation process, the vacuum step deformation is insignificant and it doesn't have atmospheric pressure (P_{atm}) (Fito *et al*., 1996).

$$X = \frac{M^{IV} - M^0}{\rho^E V^0} \quad \text{(Eq. 1)} \qquad X = \varepsilon\left(1 - \frac{1}{r}\right) \quad \text{(Eq. 2)} \qquad r = \frac{P_{atm}}{P_{vacuum}} \quad \text{(Eq. 3)}$$

The criterion for the fortification of vitamin E was to add enough quantity of the component to ensure 33mg dl-α-tocopherol acetate in 200g of fresh apple. The method used for determination vitamin E is a modification of the method of Kmostak and Kurd (1993), by incorporation of ultrasound treatment instead of agitation. The quantitative determination was carried out by a gas chromatography (CG-FID) - Fisons Instrument model NPD 800 and it is described in Restrepo *et al*. (2010).

Impregnated apple samples were dehydrated in: a) laboratory lyophilize Lioalfa 6-80, Telstar for 48h, condenser temperature: -45ºC, hot plate temperature: 25ºC and vacuum:1.2 x 10^{-3}kPa; final moisture: 7.9 ± 2.0 %; b) Tray Dryer for 24 h at 40ºC, final moisture: 12.6±1.7 %.

Cryo-Sem Technique was used for the structural analysis of the impregnated samples. A transversal section from a slice (2mm thickness) is placed in the holder and frozen in liquid nitrogen slush (-210ºC), fractured and sublimated to -94.5ºC and 10^{-5} torr. for 15 min. Finally, the sample is gold coated and observed at -150ºC by SEM using JEOL microscope, JSM-5410.

RESULTS AND DISCUSSION

Mean values and standard deviations of the physicochemical properties and characterization of VI in the studied apples were shown in Table 1. The samples were homogeneous in terms of $a_{w,}$ X_w and soluble solids. The values of the density of emulsions were 1031±1 (n=11) and 1045±1 (n=5) kg/m^3 for the products ADP and LP, respectively. Due to the variability of the samples themselves in relation to their shape and dimensions, as well as the impregnation with different emulsions, they were considered as two separate treatments.

Table 1. Physicochemical properties and characterization of IV in apple samples.

Process	Shape	a_w	X_w kg_{water}/kg_{fruit}	ºBrix	X $m^3_{emulsion}/m^3_{fresc\ fruit}$	ε m^3/m^3_{fruit}
ADP		0.988±0.002[(a)]	0.857±0.010[(a)]	12.7±1,0[(a)]	0.20±0,03[(c)]	0.21±0.03[(c)]
LP		0.991±0.003[(b)]	0.862±0.009[(b)]	12.5±1.3[(b)]	0.09 ± 0.03[(d)]	0.10±0.03[(d)]

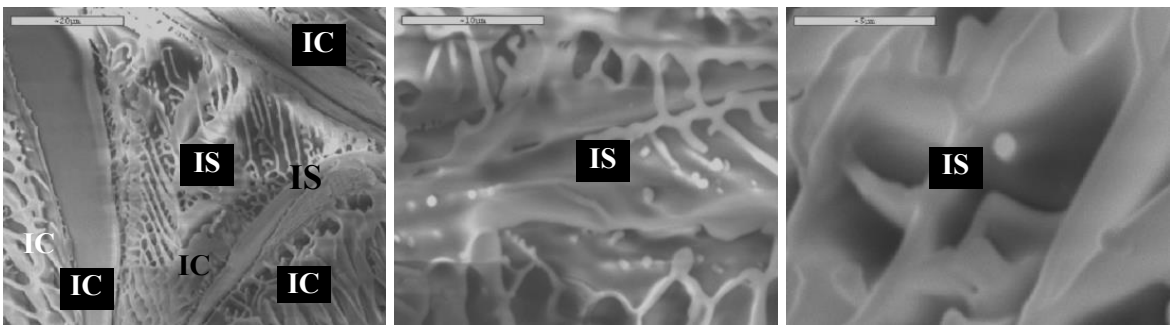
(a): n = 31; (b): n = 9; (c): n = 93; (d): n = 75. (n: samples number)

The analysis of variance showed significant differences between the treatments ($p<0.05$). The response of IV in the products ADP turned out to be the best impregnation treatment carried out, yielding data very similar to those obtained with cylindrical samples impregnated with an isotonic solution (Fito *et al*., 2001). Because of the interactions of pectin in the apples with Ca^{+2} ions present in the emulsion and the microstructural collapse consequence of changes in pressure imposed in process, LP showed a lower impregnation level. On the other hand, the lower porosity achieved was the result of coupling of hydrodynamic mechanism and deformation-relaxation phenomena during vacuum impregnation process of the solid porous (Fito *et al*., 1996).

The structural analysis for Cryo-SEM allowed the observation of impregnated apples tissues (Figure 1). The micrograph (a) shows the intercellular spaces (IS) filled with the impregnation emulsion used in the ADP products and by effects of the sublimation occurred with dendrític shape in the internal cellular (IC). The cells did not show relevant changes in the structure by effects of the IV, keeping their oval characteristic. In the micrographs (b) and (c) it was observed the oil phase in spherical form, in the intercellular space of impregnated apples parenchymatic tissue. The diameter of the drop was between 1.0 - 1.5mm.

The % recovery tocopherol acetate and squalane, the levels of vitamin E ($C_{Vit.E}$) expressed in mg dl-α-tocopherol acetate /g dry apple and the % RDV are shown in Table 2.

No significant differences were found in the process of extraction of the emulsions, and given the similarity between the results obtained with the ADP, it was concluded that the vacuum homogenization process used in the emulsification and drying did not result in loss of the active component with vitaminic activity. Losses in the extraction of squalene (»9%) were similar in all the samples and were considered when correcting the real content of dl-a-tocopherol acetate.



(a) (b) (c)

Figure 1. Cryo-SEM micrographs of Intercellular space in impregnated apples tissues.

Table 2. Concentration and recovery tocopherol acetate and squalane in the products ADP and LP.

Product	a_w	Recovery Tocopherol acetate (%)	Recovery Squalane (%)	$C_{Vit.E}$ (*)	%RDV
ADP	0.55	91.1 ± 2.5	91.5 ± 2.8	0.72 ± 0.12	86.0 ± 16.8
LP	0.11 - 0.22	113.0 ± 7.8	90.9 ± 13.0	1.38 ± 0.14	134.6 ± 16.3
Emulsions	.....	91.2 ± 3.3	91.3 ± 3.3	.....	.....

(*) mg dl-a-tocopherol acetate / g dry apple

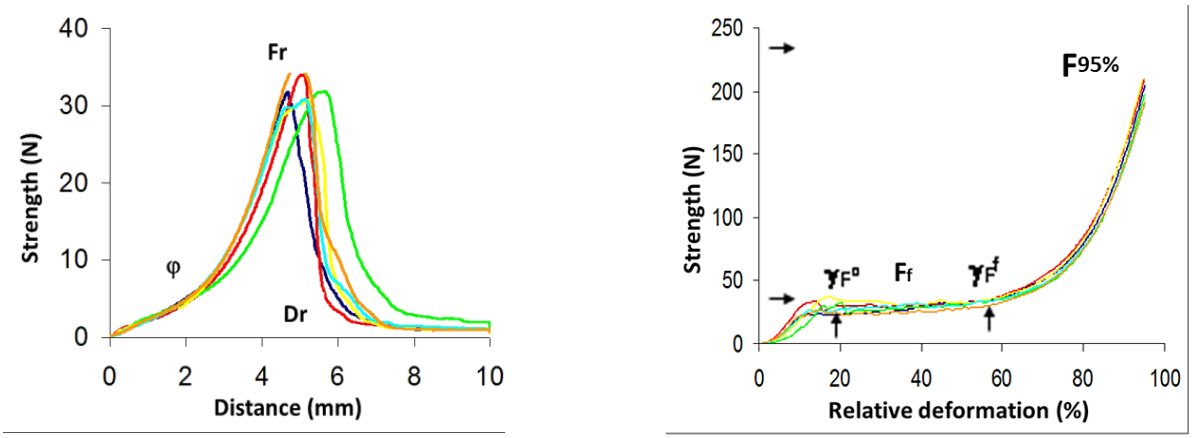
For the LP, the recovery and the RDV were > 100%, owing to exit of intracellular in the vacuum stage, which is replaced by an amount of emulsion that has not been quantified in theoretical calculations. These high values equally evidence a better extractability of the component with vitamin E activity probably associated to the greater porosity of the samples and the easier diffusion of the solvent through the pores. The a_w conditions of the products guarantee stability to microbial damage, although other decay processes such as non-enzymatic browning and solute crystallization can occur, especially in the products ADP with higher a_w values (Troller, 1987).

The color parameters for the ADP and LP products are shown in Table 3. Every parameter shows significant statistical differences. The darkening of the ADP products was generated by both enzymatic and non-enzymatic browning reactions (Acevedo *et al*., 2008), producing a decrease in luminosity (L*); that behavior could be explained by the low water activity on the Maillard reactions (Udomkun *et al*., 2015), enzymatic activity that is still present after the impregnation treatment and the conversion of polyphenols into polycarboniles (Acevedo *et al*., 2008). The increased value of a* was attributed to a greater chlorophyll degradation of the ADP products. The LP products were more clarity (>L*), more greenish (<a*) and less intense (< Cab*); besides, they presented a higher opacity and more reflection of light, due to the increased amount of gas occluded in the pores.

The results of the texture of ADP and LP products are shown in Figure 2. The strength-distance curves obtained, allow the observation of the point where strength is maximum (F_r), corresponding to the point in which the gummy sample breaks at a rupture distance (D_r). For most materials, the relation stress-deformation remains linear only at small deformations in which the material had an elastic behavior (Rosenthal, 2001). This linearity is observed in the first segment of the curve, whose slope (j) is related to the product extensibility module. For the LP products the behavior was typical of porous products, where a plateau of constant strength (F_f) were observed. A deforming without increasing in resistance, could be associated with the progressive fracture of dry tissue layers inside the matrix, while the volume of internal air is decreasing. When the volume of air disappears, the material is compacted by compression element, with an exponential increase of the resistance to deformation. It is possible to observe the interval of relative deformation, initial ($g_F{}^o$) and final ($g_F{}^f$), during the time in which the value of F^f is maintained.

Table 3. Color parameter of the ADP and LP products.

Product	L*	a*	b*	C_{ab}*	h_{ab}*
ADP	66.30 ± 6.20	0.61 ± 1.01	20.44 ± 4.07	20.47 ± 4.06	88.08 ± 2.93
LP	84.78 ± 0.51	-3.12 ± 0.47	14.02 ± 0.82	14.37 ± 0.86	102.52 ± 1.54



(a) (b)

Figure 2. Strength (N)-Distance (mm) curve in ADP products (a) and Strength (N)-Relative Deformation (%) curve in LP products (b).

CONCLUSIONS

The incorporation of vitamin E inside the porous structure of the apple contributed generating value to the fruit. The process VI is an adequate technique for the development of new products with functional properties, it has a lot of applications in other fruits with similar structures and/or with other PACs that could favor the prevention of some diseases. The response to VI is affected by the shape and thickness of the samples, as well as by the characteristics of the composition of the emulsion and its interactions with fresh apple. The effect of dehydration was not a critical factor in vitamin E content; in addition, the color and texture were acceptable for the moisture values reached in each of the products.

ACKNOWLEDGMENT

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