Influence of cultivar and process conditions on crispness of organic osmo-air-dried apple chips

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Summary The aim of this work was to evaluate the effect of different variables on osmo-air-dried apple chips processing, in order to identify the most influencing factors on final product's characteristics. A 2^3 full-factorial design was selected; cultivar (*Golden Delicious* and *Pink Lady®*), osmosis time (30, 60, 90 minutes) and air drying temperature (70°, 80°, 90°C), were independent variables, crispness coefficient (E, $kNmm^{-2}$), relative relaxation stress (RRS), water activity, colour attribute L*, panel score and moisture content ($gH_2O/100g$ solids) were dependent variables. Apple rings were immersed in sucrose, maltose or a mixture of fructose, glucose and sucrose ($a_w = 0.90$), and air dried to constant weight. Crispness, and physico-chemical properties were affected most by drying temperature, less by cultivar and osmosis time. The screening test discriminated pre-treated apples from non pre-treated ones, the former being evaluated as crispier. Results indicate that the interaction between drying temperature and fruit tissue structure is the key-factor optimizing the osmo-air-drying process, aimed at high level crispy apple chips.

Key words crispness, apple, design of experiment, drying, osmotic dehydration.

Introduction

The consumer market is demanding more healthy and natural, but mainly tastier fruit-based new products. Snack foods such as dried fruit chips could satisfy these requirements as they can be obtained from natural products, and technological pre-treatments can optimize their taste. Moreover, they are ready to eat in a serving size package, and have a good shelf life. For snack foods the sensory attributes that most strongly influence quality evaluation by the consumers are "crispness and "crunchiness", more than taste (Shewfelt, 1999; Roudaut et al., 2002). Water removal from the original matrix has been established as the pivotal step in order to obtain these characteristics and maintain a high quality product. Thus, to reach this purpose, research has been striving towards improving the drying technology (Lewicki, 2006).

In fact convective drying of plant tissue causes numerous modifications in the structure of the material, its composition and the spatial conformation and distribution of biopolymers, mostly caused by solute concentration and temperature gradients (Lewicki and Pawlak, 2003).

Shrinkage is one of the principal physical changes influencing the structure of the plant tissue during drying (Aguilera and Stanley, 1999) and it is almost linearly related to moisture content (Sjöholm and Gekas, 1995; Wang and Brennan, 1995). Changes in cell structure and tissue integrity can also negatively affect quality parameters such as colour and nutritional compounds content (Krokida and Maroulis, 1997; Lewicki and Duszczyk, 1998).

The extent of shrinkage and degree of damage strictly depend on the drying method plus air temperature and velocity (Lewicki and Pawlak, 2003; Wang and Brennan, 1995). As reported by Lewicki and Jakubczyk (2004) mechanical properties of convective dried fruit tissue are strongly related to the hot air temperature. The faster the drying the smaller is the shrinkage, thus yielding an increased porosity of the obtained dry product. The same authors also verified the relation between the state of the water in the dried product and the drying temperature.

Fruit impregnation with sugars through osmotic dehydration, applied before convective drying, is one of the most effective pre-treatments able to decrease shrinkage (Del Valle, Cuadros, & Aguilera, 1998; Lewicki, 1998; Lewicki and Lukaszuk, 2000). Compared to other dehydration processes, the distinctive feature of osmosis, involving placing fruit into solutions of high sugar concentration, is the penetration of solutes into the food material. By modifying the extent of sugar enrichment and syrup composition not only can the end product be diversified but chemical, physical and functional properties can be improved (Torreggiani and Bertolo, 2004; Riva et al., 2005; Pani et al., 2008). In addition, the cellular structure of the fruit, linked to cultivar, was shown to be the key point for optimizing the osmosis process, alone and coupled with convective drying.
The aim of this work was to evaluate the effect of different variables on dried apple chips processing, in order to identify the most influencing factors on final product's characteristics, especially in terms of crispness. In order to accomplish this goal, the influence of apple cultivar, osmosis time, osmotic sugar solution, and air temperature on the end product moisture content and crispness was studied through the Design of Experiment (DoE) technique, which has been widely used in other previous works dealing with the development of new food products (Guha et al., 2003; Calzetta Resio et al., 2006; Affrifah and Chinnan, 2005; Elbert et al., 2001; Farris and Piergiovanni, 2008). In particular, the experimental investigation was carried out by a screening test, using three full factorial designs, one for each osmotic solution. This kind of design allowed separating the vital few factors from the many trivial with a limited number of experiments, minimizing time and cost of the research (Box et al., 2005).

Materials and Methods

Apples

Organic apples of two cultivars were used: Golden Delicious and Pink Lady®, both cultivated in the Research Centre for Agriculture and Forestry (Laimburg, BZ, Italy). Apples, harvested at the same maturity stage (Golden Delicious: dry matter 14.84%, total titratable acidity 10.73 meqNaOH/100 g, refractive index 13.05 °Bx; Pink Lady®: dry matter 15.96%, total titratable acidity 9.70 meqNaOH/100 g, refractive index 14.10 °Bx), were washed, wiped dry and cored by a spoon soil auger (25.0 mm diameter), and mechanically cut (LT INOX, Kronen, Germany) into 5.0 mm thick rings.

Treatments

Osmotic dehydration. Apple rings were immersed for different times (30, 60, 90 minutes), at 25°C in three different sugar solutions at the same water activity (a_w = 0.90): sucrose (59% wt/wt), maltose (62% wt/wt) and a mixture (53% wt/wt) of fructose, glucose and sucrose, which had the same composition as apple (Golden Delicious: fruct 49%, glu 13%, sucr 38%; Pink Lady®: fruct 47%, glu 4%, sucr 49%). The sugar solutions were continuously recirculated through a peristaltic pump. The ratio fruit/solution was 1/3. Soluble solids gain (SG) and water loss (WL) were calculated according to Giangiacomo et al. (1987), and expressed as g/100g of initial fresh fruit weight.

Air drying. Air drying was performed at different temperatures (70°, 80°, 90°C) up to constant weight, using a pilot alternate upward-downward air circulated drier (Thermolab, Codogno, Italy) operating at an air speed of 1.5 m/s. For all samples, the equilibrium weight (i.e. the constant weight) was achieved when the difference in weight was less than 1 mg/g solids after 90 additional minutes of drying.

Chemical analyses

Dry matter of raw apples and total titratable acidity were determined according to AOAC methods (1995); refractive index (°Bx) using a multiscale automatic refractometer (mod. RFM91, BS, UK). Sugars were quantified on aqueous extract by HPLC according to Forni et al. (1992). Water activity (a_w) of osmo-air-dried apple rings was measured by an electronic hygrometer (Aqua Lab. CX-2 –

Decagon Devices, Pullman, USA), based on the determination of the dew point and previously calibrated with a standard solution of LiCl of known activity (prepared by High-Purity Standards for Decagon Devices). Results are the mean of 6 determinations. Moisture content (m) was determined according to Karl Fischer method after extraction in anhydrous methanol (ASTM D 6304-2004 a, 1-procedure A). Results are the mean of 3 determinations and are expressed as g H_2O/100 g solids.

Colour

Colour analysis was carried out on dried apple rings using a D65 illuminant/10° observer reflection colorimeter (Minolta Chroma Meter CR 200 - Minolta Camera Co. Ltd., Japan). Chips were placed on a white standard plate (L^* = 100) and the *CIE* $L^*a^*b^*$ co-ordinates were simultaneously measured, although only the L^* parameter was considered in the experimental design as a measure of lightness. Reported data are the mean of 20 readings, made on five different apple chips.

Mechanical tests

Apple chips mechanical properties were determined using two compression tests, as described by Farris et al., (2008). The former was a stress-relaxation test, carried out on a 40 ± 2.0 mm pile of apple slices which was compressed at a speed of 10 mm/min between a lower (150 mm Ø) and an upper plate (80 mm Ø), connected with a Zwick Machine (mod. Z005, Zwick Roell, Germany) fitted with a 100 N load cell. At 20% deformation, the crosshead surface was stopped and the pile was allowed to relax for 20 s. The relative relaxation stress (rrs), calculated by the software (TestXpert V10.11 Master), was considered. The latter was a bending-snapping test: one apple ring at a time was placed on two supports and a third compressing bar was driven down at a speed of 10 mm/min, bending each specimen until it snapped. The slope (i.e. the elastic modulus, *E*) before the first fracturability peak of highest magnitude, was considered as the index of crispness of the dried apple rings. Final results are the mean of 3 and 10 replicates, respectively.

Sensory analysis

For sensory analysis a descriptive test was applied. Ten panelists expressed a judgment on crispness intensity on a free scale from 0 (low) to 10 (very high). Each session was repeated on two subsequent days. Results were elaborated with FIZZ, Software Solutions for Sensory Analysis and Consumer Test, Biosystemes, France.

Statistical analysis

Data were subjected to one-way anova using Statgraphics plus 4.0 software, followed by the least significant difference multiple range test ($P \leq 0.05$) for comparison of the mean values. Modde software package (Modde 2006, version 8.0; UMETRICS AB, Umea, Sweden) was used for evaluation of raw data and regression analysis in the screening design, according to the least squares analysis technique.

Light microscopy

Photographs were taken on apple slices (300 μm thickness) obtained cutting a section of a ring by means of a vibrating blade slicer (Vibroslice NVSL, World Precision Instruments, Inc., USA). Reported images refers to the central part of the section. Samples were examined under an optical microscope (Olympus, BX50) equipped with a digital camera (Q IMAGING, Retiga 2000R-fast 1394).

Experimental design

Within the screening test four different designs were built: three for the different sugar solutions, and one for not pre-treated apple chips. In each of such designs, a qualitative factor (cultivar, X_1) and two quantitative factors (osmosis time, X_2, and air-drying temperature, X_3) were considered. Each of these independent variables was assessed at two equidistant levels (-1 and +1) from the centre point (0), except for the qualitative one, which assumes only discrete values. Factors and their levels are shown in Table 1. In addition, six dependent variables (responses) were selected to evaluate apple chips quality: crispness coefficient (Y_1), relative relaxation stress (Y_2), water activity (Y_3), lightness (Y_4), panel score (Y_5) and moisture content (Y_6). Taking into account the available resources, a 2^3 full factorial design of resolution V (eight corner points and four centre-point replicates) was selected for the osmo-air-dried apple rings, whereas the choice for the not pre-treated rings resulted in a 2^2 full factorial design, which included only four corner points due to the absence of the factor "osmosis time". As an example, the worksheet obtained from the design concerning the maltose solution is reported in Table 2. All designs support an interaction model expressed by the general polynomial equation:

$$Y_z = b_{z0} + \sum_{k=1}^{l=5} b_{zk} X_k + \sum_{k \neq j}^{l=5} b_{zkj} X_k X_j + \varepsilon$$

where:

- Y_z = dependent variable;
- b_{z0} = response value when all factors are set at medium level (centre point);
- b_{zk} = linear regression coefficient;
- b_{zkj} = interaction regression coefficient;
- ε = residual response variation not explained by the model.

By estimating the numerical values of the model terms, i.e. the regression coefficients, it has been possible to establish the correlation between each factor and the different responses.

Results and discussion

The first step in data processing was the study of responses values' distribution in order to evaluate the opportunity of a transformation to obtain a normal data distribution, thus improving model validity. In order to refine the models, both main linear and interaction regression coefficients were calculated for each design. Those coefficients outside the confidence level of 95% were removed and the model refitted to the data. Arising from this data analysis, the term “osmosis time”, and consequently the linked interactions, was significant only for the DoE ‘mixture’, thus it was removed from all the responses of the other designs, being the related coefficients not significant in defining the factors-responses relationship. The lack of correlation between time and responses could be due to the trend of the solid-liquid exchanges during the osmotic treatment; this hypothesis seems to be strengthened by the fact that the rates of both water loss and solid gain reach their highest values at the beginning of the osmotic process (Torreggiani and Bertolo, 2004; Kowalaska and Lenart, 2001), especially for samples with a wide exchange surface such as apple rings. Solid gain and water loss values of the two cultivar apple rings are shown in Table 3, while sugar content during osmosis of Golden Delicious are reported in Figure 1. The same trend was observed in cv Pink Lady® (data not reported). In both apple cultivar rings treated in maltose and sucrose the most important changes in all the sugars contents occurred in the first 30 minutes, although maltose content increased up to 90 minutes of osmosis. On the other hand the increase in all the sugar contents in apple rings treated in mixture, was smaller but steady over the entire osmosis treatment, with a similar change observed in the three time intervals: 0-30, 30-60 and 60-90 min. These small but regular changes could be responsible for the influence of time on the responses, however this hypothesis should be confirmed in a further study. It can also be noticed, as expected, that SG values are higher in apple treated in maltose and sucrose than in mixture, being these sugars absent (maltose) and present in limited amount (sucrose) in the fruit, while mixture having the same sugar composition as apple. The power of fitting of the refined models was then assessed taking into consideration the power of fit (R^2), the power of prediction (Q^2), the model validity and the reproducibility parameters, as defined elsewhere (Farris and Piergiovanni, 2008). Values of the above-mentioned parameters are reported for each DoE in Table 4. The high Q^2 values calculated for all the responses show that good models, in some cases even excellent (Q^2 >0.9), have been obtained, with the only exception of water activity. The low Q^2 values for this response can be explained by the slight differences in a_w observed among samples; these differences are not significant because they are within the experimental error, being a_w values in the narrow range 0-1 (Farris and Piergiovanni, 2008). Since the model cannot explain a_w data variation, this response cannot be used for prediction purposes, so it will no longer be taken into account. Figure 2 shows, as an example, the influence of each factor (as main linear and as interaction effect) on the responses for DoE 'maltose'. It can be observed that temperature is the most influencing factor on all the responses and, in particular, it influences positively crispness coefficient, relative relaxation stress and score, i.e., with an increase of temperature, these responses increased as well. On the other hand, increasing temperature, moisture content and lightness of apple chips decreased. In the same way temperature is the most important factor for 'sucrose', 'mixture' and 'not pre-treated' DoE, confirming the highest influence on all the responses. The utmost importance of temperature is also highlighted by the main effect plot, which is a graphical representation of the effect of each individual independent variable at a time on a pre-selected response. Figure 3 depicts the main effect of temperature on the crispness attribute (expressed as panel score) of apple rings not pre-treated and dipped into the three different sugar solutions. It can be observed that the increase in temperature from 70°C to 90°C caused a different variation of the score given by the panellists, depending on the specific treatment undergone by the apple rings. In particular, considering the Δ values (given by the difference between the scores recorded at the highest and lowest air drying temperature), it was observed that the effect of temperature was significantly greater for maltose-treated rings (Δ = 5438.84), when compared with those sucrose-treated (Δ = 3619.57), mixture-treated (Δ = 3582.22) and not-treated (Δ = 3170.66). Similar results were also obtained for the response crispness coefficient E (data not reported). Furthermore, it has to be noticed the role of the cultivar, which can be inferred from the coefficient overview plots reported in Figures 2 and 4 for the 'maltose' and 'mixture' designs, respectively. In such plots the bars related to the two cultivars have the same height, since the cultivar is a qualitative variable. However, the bars are always counter-oriented, indicating which cultivar influenced most one specific response, thus providing only qualitative information. So, from the ‘maltose’ design it can be asserted that Pink Lady® cultivar appears more suitable when the mechanical attributes have to be enhanced; conversely, the Golden cultivar is more convenient when an improvement in colour and consumers’ acceptance, and a reduction of moisture content is aimed at. On the contrary, cv Golden Delicious exhibited a positive effect on all the responses as shown in the ‘mixture’ design (Figure 4). The differences between the cultivar can be observed in the main effects plot reported in Figure 5, which shows the influence of the two cultivars on the panel score. For the four different pre-treatments (maltose, sucrose, mixture, not pre-treated), the highest values were recorded for cv Golden Delicious. With respect to the different pre-treatments, maltose samples showed the highest Δ value (3292.09), whereas mixture and not pre-treated curves had the same slope. This suggests that the particular composition of the mixture does not modify the original apple sugar composition, since the mixture contains fructose, sucrose and glucose in the same proportion as in the raw apples. Pre-treated samples were discriminated from apple rings air-dried without a previous osmosis step, the former being evaluated as crispier from both mechanical and sensorial tests. This difference could be attributed to the microstructural differences of the specimens, which can be clearly visible in the light microscopy images shown in Figure 7. The images show the section of two apple chips air-dried at 80°C after 90 minutes of osmosis in the mixture (Fig. 7a), and without the osmotic pre-treatment (Fig. 7b). In particular, such an important shrinkage took place in the control sample that the whole section of the ring could be observed through the microscope lens. Moreover far fewer voids were present and their shape was not as round as can be seen in Figure 7a. With regards to the cultivar, panellists perceived Golden Delicious chips crispier than Pink Lady® ones, regardless of the treatment, but this result is not in total accordance with the instrumental data; in fact, as can be seen in Figure 6, only Golden Delicious rings pre-treated in sucrose solution and in the mixture obtained E values higher than Pink Lady® in the bending-snapping test. This apparently contradictory result can be explained comparing Figure 5 and Figure 6. In fact, for E values higher than 11 kN mm^{-2}, as can be observed for maltose treated rings, panellists perceived samples no longer crispy and brittle but hard and stiff, thus giving a lower score to cv Pink Lady® samples. This result could account for the different porosity of the dried apples, which in turn relies on the tissue structure of the two cultivars and consequently to different solid-liquid exchanges during osmosis. For this reason, it was ascertained that the bending-snapping test, in spite of its ability to discriminate samples with different moisture contents, was not able to detect textural changes due to microstructural differences. Hence the necessity to set up a more exhaustive technique that could provide information about tissues' structural changes during the process.

Conclusions

Design of Experiment (DoE) technique was successfully applied to evaluate the effect of different variables on osmo-air-dried apple rings processing, allowing a better understanding of the factors that influence to a greater extent the responses. Temperature is the most important process parameter, as it mainly influences apple chips quality characteristics, in terms of crispness (evaluated using both mechanical and sensorial tests), and physico-chemical properties (colour and moisture content); on the other hand osmosis time, over the range considered, does not have a significant influence on the responses.

Shrinkage was substantially reduced in osmo-air-dried apple chips and this fact was reflected in enhanced mechanical properties and in a greater appreciation by panellists. This difference could be attributed to the different porosity of the specimens, linked to a better preservation of the apple tissue structure, which in turn can be ascribed to the partial concentration achieved during the osmotic pre-treatment. Comparing the sugar solutions used for the osmosis pre-treatment, a different trend was observed on the responses, but it was not possible to detect meaningful differences. Accordingly, a future perspective could be the optimization of the osmosis pre-treatment in the sugar mixture, since it does not modify the apple sugar composition. This choice can be seen as the first step towards using a concentrated apple juice, leading to an end product completely fruit-based in the near future. Moreover, although maltose pre-treatments yielded crispier products, some hurdles such as high costs and taste modification hamper its potential use at an industrial scale. The results evidenced also that cultivar Golden Delicious was preferred compared to cv Pink Lady® regardless to the treatment. Finally, compression tests used in this work were able to describe apple chips' texture, discriminating between the two cultivar and the different process conditions. However, crispness coefficient values (E), obtained from the bending-snapping test, were not in total accordance with sensory scores. Indeed, at high E values panellists perceived apples hard and stiff, so they did not assign high crispness scores. For this reason, further studies should be carried out to fix up an index or an instrumental technique that better reflects the brittleness of the specimen in agreement with panellists' judgement.

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