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Original Article

Quality of ‘Royal Gala’ cut apple during osmotic dehydration
Qualidade de maçã Royal Gala cortada durante a desidratação osmótica

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Abstract

The present work aimed to evaluate quality parameters of ‘Royal Gala’ apple cubes during osmotic dehydration (OD). We investigated the following OD conditions: osmotic agent, pressure, and temperature. The osmotic agent, being lower after OD with sorbitol than sucrose solutions, mainly influenced the water activity of the product. The color changes increased with increased temperature and were higher in vacuum experiments than at atmospheric pressure. In general, we recommend OD at 25 °C and atmospheric pressure for the preservation of the total phenolic content (TPC) and antioxidant activity (AA) of apple cubes during the process. Peleg’s model was found to provide the best fit of TPC and AA data.

Keywords: Osmotic agent; Pressure; Vacuum; Sorbitol; Sucrose; Water activity; Color; Total phenolic content; Antioxidant activity; Mathematical models.

Resumo

O objetivo do presente trabalho foi avaliar parâmetros de qualidade de cubos de maçã Royal Gala durante o processo de desidratação osmótica (DO). As condições de operação da DO investigadas foram: o agente osmótico, a pressão e a temperatura. A atividade de água do produto foi influenciada principalmente pelo agente osmótico, sendo menor após DO com soluções de sorbitol do que de sacarose. Alterações de cor aumentaram com o incremento da temperatura e foram maiores nas experiências a vácuo do que à pressão atmosférica. Em geral, DOs a 25 °C e à pressão atmosférica são recomendadas para a conservação do teor em fenóis totais (TFT) e da atividade antioxidante (AA) dos cubos de maçã durante o processo. O modelo de Peleg permitiu o melhor ajuste dos dados experimentais de TFT e AA.

Palavras-chave: Agente osmótico; Pressão; Vácuo; Sorbitol; Sacarose; Atividade da água; Cor; Teor em fenóis totais; Atividade antioxidante; Modelos matemáticos.

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1 Introduction

In 2015, more than 95% Brazilian apple orchards cultivate Gala apples, including ‘Royal Gala’ (Ávila et al., 2015). In order to present a longer shelf life, sometimes it is beneficial that the fruits undergo pre-treatment, such as osmotic dehydration (OD). This process is becoming popular in the food industry because it improves the sensorial and nutritional properties of foods, with a reduced loss of aroma in semi dried and dried food stuffs (Khan, 2012).

OD consists of immersing a fruit or vegetable in a hypertonic solution of salt, alcohol, starch or concentrate sugar, which has a higher osmotic pressure than the food. The osmotic pressure is the driving force for the mass transfer of water and solutes between the food and the osmotic solution (Ramya & Jain, 2017; Shi & Le Maguer, 2002). Osmotically dehydrated products have a water activity in the range of 0.90-0.95. Normally, these products require further processing in order to extend their shelf life.

Sucrose is the most used osmotic agent in the OD of fruits due to its effectiveness, convenience and desirable flavor (Ahmed et al., 2016; Lenart, 1996). Alternative solutes, such as sorbitol, have also been used (Brochier et al., 2014; Chauhan et al., 2011; Rodriguez et al., 2013). Sorbitol is one of the polyols naturally found in several fruits and it was one of the first to become commercially available (Assis et al., 2017a). Aprea et al. (2017) reported sorbitol contents in some of the apple varieties (except Royal Gala) of a maximum of 12.9 g/kg. Sorbitol is described as non-cariogenic. In the food industry, sorbitol is used as a sweetener (E420). It has 60% of the sweetness of sucrose and it has a lower caloric value: it gives the consumers 2.6 kcal per gram (EU) instead of 4 kcal per gram given by sucrose. Another health property is related to sorbitol producing no significant post-prandial hyperglycemia, which is a positive aspect for a diabetic diet (Ellis & Krantz, 1943). Sorbitol has molecular mass lower than sucrose, and osmotic agents with low molecular mass find less resistance to penetrate into the fruit cell tissues. Assis et al. (2017a) showed that a 60 °Brix sorbitol solution had lower viscosity than a sucrose solution with the same concentration. Sorbitol was found to be a better osmotic agent than fructose to obtain pulsed vacuum osmotically dehydrated bacon slices with a higher fructan retention (Oliveira et al., 2016).

The present work aimed to evaluate quality parameters, such as water activity, color, total phenolic content (TPC) and antioxidant activity (AA) of ‘Royal Gala’ apple cubes during the OD at different conditions of osmotic agent, pressure, and temperature using sucrose and sorbitol.

2 Material and methods

2.1 Samples

Apples (Malus spp., variety Royal Gala) were graciously supplied by Campotec, Portugal, and stored at 4 °C. The apple cubes (12 mm) were prepared according to Assis et al. (2017a). The soluble solids content of the apple was 16.2 ± 1.2 °Bx (hand refractometer, Atago, China).

2.2 Osmotic dehydration process

The OD process was carried out with sucrose or sorbitol solutions at 60 °Brix and a mass ratio of sample to solution of 1:4 and at a constant agitation of 50 rpm (Assis et al., 2017a). The experiments with the sucrose solution were performed first at 25, 40 and 60 °C, at atmospheric pressure and in vacuum (hermetic containers at 150 mbar). After analysis of the results, the experiments with the sorbitol solution were set up at 25 and 60 °C.

The apples samples were removed from the solution at different times and selected quality parameters were evaluated in the apple cubes.

The experiments were performed in duplicate.
2.3 Moisture content (M_d)

The samples moisture content in dry basis was determined in an oven (FP115, Binder, Tuttlingen, Germany) and calculated through the Equation 1:

\[ M_d = \frac{w_i - w_d}{w_d} \]  

where \( w_i \) (g) is the initial weight of the apple cubes; \( w_d \) (g) is the weight of the apple cubes after 24 hours at 105 °C in the oven.

The moisture content was evaluated in triplicate. Afterwards, \( M_d \) was normalized by dividing it by \( M_{d,0} \), because the initial value of the apple sample was different for each experiment.

2.4 Quality evaluation

2.4.1 Water activity (a_w)

The \( a_w \) of the samples was determined during the process, using a hygrometer (Aqualab Series 3, Decagon Devices Inc., Pullman, Washington, USA) at 22 °C. Each determination was performed in duplicate.

2.4.2 Color

Samples color was determined in the CIE \( L^*a^*b^* \) color space (Commission Internationale de L’éclairage, 1978), using a Minolta CR-300 colorimeter (Minolta Camera Co., Ltd., Osaka, Japan). Three replicates were used for each sample. The total color difference (\( \Delta E \)) was calculated through the Equation 2:

\[ \Delta E = \sqrt{(L_0 - L)^2 + (a_0 - a)^2 + (b_0 - b)^2} \]  

where the index “0” indicating the sample before OD.

\[ \Delta E = \sqrt{(L_0 - L)^2 + (a_0 - a)^2 + (b_0 - b)^2} \]

2.4.3 Total Phenolic Content (TPC)

The apples samples were ground in liquid nitrogen with a mortar. Subsequently, we weighed 2 g of each sample, and added 10 mL of methanol (Sigma Aldrich, Steinheim, Switzerland); the mixture was then homogenized using an ultra-turrax (T25, IKA, Breisgau, Germany) for 2 min. The supernatant was used after centrifugation at 5000 rpm and 4 °C for 15 min (Chong et al., 2013).

The total phenolic content was determined by the Folin-Ciocalteu method (Singleton & Rossi, 1965). The reaction was performed by adding 0.5 mL of apple extract, 0.5 mL of Folin-Ciocalteu reagent, 1 mL of sodium carbonate 75 (g L^{-1}) (Sigma-Aldrich) and 1.4 mL of deionized water. After 1 h in the dark at room temperature, the TPC was determined at 750 nm in a spectrophotometer (Shimadzu UV-vis 1240, Japan). Quantification was performed with respect to the standard curve of the gallic acid. The determinations were carried out in triplicate.

2.4.4 Antioxidant Activity (AA)

The ABTS method was used to determine the AA (Gião et al., 2007). After addition of 1 mL of the ABTS+ solution (absorbance = 0.700 ± 0.02) to 0.2 mL of extract, the analysis was performed after 6 min at 734 nm and expressed as mg ascorbic acid/g dry matter. The determinations were performed in triplicate.
2.5 Mathematical models

The following models were used to fit the TPC and AA data.

Peleg’s model
\[
\frac{TPC}{TPC_0} = \frac{AA}{AA_0} = 1 - \frac{t}{k_1 + k_2 \cdot t}
\]  
(3)

Page’s model
\[
\frac{TPC}{TPC_0} = \exp\left(-A \cdot t^B\right)
\]  
(4)

\(A\) and \(B\), and \(k_1\) and \(k_2\) being Page’s and Peleg’s model parameters, respectively; the index “0” indicating the sample before OD.

2.6 Statistical analysis

The statistical analysis was performed using Microsoft Excel 2000 (Microsoft Corporation, Washington, USA) (mean and standard deviation calculations) and IBM SPSS® Statistics 20.0 for Windows® (2012, SPSS Inc., Chicago, USA). The model parameters of the experimental data fit were estimated by non-linear regression procedures, and the estimates margin of error was calculated at 95% (the margin of error is the half width of the confidence interval at 95%). The regressions were also assessed by ANOVA approaches, assuming a significance level of 5%.

The adequacy of the models fit was evaluated by the determination coefficient (R²) and by the residual analysis. The residual analysis was performed in order to check the assumptions of independence, randomness and normality. The normality of the residuals was evaluated by Kolmogorov-Smirnov test.

3 Results and discussion

The \(a_w\) decreased during OD (results not shown). This is important for safety issues. This decrease in \(a_w\) was confirmed by Assis et al. (2017a) during OD of apple cubes. OD with sorbitol solutions produced apple cubes with significantly lower \(a_w\) than with sucrose solutions, which is in agreement with Assis et al. (2017a). The \(a_w\) of the apple cubes osmotically dehydrated with sorbitol was 0.892-0.914, while the samples osmotically dehydrated with sucrose presented a \(a_w\) of 0.933-0.944. No effect of the pressure on \(a_w\) was found in the present work. Vieira et al. (2012) found that \(a_w\) was mainly affected by temperature. They also found that a higher vacuum pulse time (at least 20 min) also favored a \(a_w\) decrease.

When the OD is carried out at atmospheric pressure, \(\Delta E\) was lower than when the apple cubes were osmotically dehydrated in vacuum (Figure 1). Color differences between samples osmotically dehydrated in vacuum and at atmospheric pressure were visually perceived. The highest \(\Delta E\) corresponds to the highest changes in L* (results not shown), both at atmospheric pressure and in vacuum. Neri et al. (2016) and Moreno et al. (2017) explained that the L* parameter decreased more in OD in vacuum than at atmospheric pressure, because of the total or partial air-to-solution replacement in the sample pores that occurred with vacuum impregnation, at pressures below 857 mbar.
There was an increase in $\Delta E$ at 60 °C, probably due to Maillard reactions, which are more intense at higher temperatures and lower $a_w$ (until approximately 0.8) (Fennema, 1996). Enzymatic browning by polyphenol oxidase could have occurred at room temperature within 15 minutes after cutting (Tuccillo, 2018). However, a proper pre-treatment was performed to avoid this browning process: immediately after cutting, the cubes were immersed in a solution with 0.9% sodium chloride for 3 min (Assis et al., 2017a).

The highest color change ($\Delta E$) was, therefore, observed in cubes osmotically dehydrated at 60 °C in vacuum and the smallest one at 25 °C and atmospheric pressure. The use of sorbitol showed a tendency to induce a higher $\Delta E$ than sucrose, especially at high temperatures.

After 8 h of osmotic treatment in the sucrose solution at 60 °C and atmospheric pressure, the losses of TPC and AA of apple cubes were around 75% (Figures 2 and 3), in agreement with Assis et al. (2017b). These losses were around 60% at 40 °C and 40% at 25 °C.
In general, both Peleg’s and Page’s models fitted well the experimental data of the antioxidant activity (AA) and total phenolic content (TPC) of apple cubes during OD, as high determination coefficients ($R^2$) were obtained (Table 1) and the residual analysis showed that the residuals are normally distributed at a 5% significance level. The visual analysis of the scatterplot of residuals versus predicted values allowed noting that the residuals were distributed randomly around zero with no systematic pattern, satisfying the assumption of homoscedasticity and zero mean errors. Therefore, we selected the best fit based on the precision of parameter estimation, calculated though the Standard Half Width (SHW = margin of error/ parameter value) at 95% confidence. The average SHW was 20% and 27% for AA and 21% and 30% for TPC, for Peleg’s and Page’s models, respectively. Thus, Peleg's model described better the evolution of AA and TPC of apples cubes during OD, with the highest precision of parameter estimates (lowest SHW) for all conditions tested (Figures 2-5, Table 1). None of these models fitted well the TPC of apple cubes during the OD with sucrose solution at 40 °C and vacuum (Figure 2). Therefore, we did not take this condition into account in the comparison of the process variables. In addition, none of the models fitted $\Delta E^*$ data well, because they did not follow one or more of the assumptions described above.

Table 1. Parameters of the fit of Peleg’s and Page’s models of the antioxidant activity (AA) and total phenolic content (TPC) of apple cubes during OD.

| SOLUTE  | PRESSURE (MBAR) | AA          | TPC          |
|---------|----------------|-------------|--------------|
|         |                | $K_1 \pm$ MARGIN OF ERROR | $A \pm$ MARGIN OF ERROR | $K_2 \pm$ MARGIN OF ERROR | $B \pm$ MARGIN OF ERROR | $K_1 \pm$ MARGIN OF ERROR | $A \pm$ MARGIN OF ERROR | $K_2 \pm$ MARGIN OF ERROR | $B \pm$ MARGIN OF ERROR | $R^2$ |
| SUCCINE | 25 1013        | 2.309 ± 0.835 | 1.780 ± 0.130 | 0.940 | 0.422 ± 0.071 | 0.194 ± 0.075 | 0.931 | 2.406 ± 1.007 | 1.759 ± 0.166 | 0.955 | 0.412 ± 0.087 | 0.206 ± 0.096 | 0.945 |
| SORBITOL 25 1013 | 1.945 ± 0.529 | 1.499 ± 0.088 | 0.962 | 0.508 ± 0.068 | 0.227 ± 0.063 | 0.959 | 3.712 ± 1.326 | 1.505 ± 0.184 | 0.936 | 0.326 ± 0.085 | 0.343 ± 0.117 | 0.931 |
| SORBITOL 40 1013 | 1.836 ± 0.557 | 1.470 ± 0.104 | 0.958 | 0.479 ± 0.062 | 0.275 ± 0.064 | 0.968 | 1.929 ± 0.721 | 1.496 ± 0.133 | 0.936 | 0.453 ± 0.070 | 0.286 ± 0.076 | 0.954 |
| SORBITOL 60 1013 | 1.721 ± 0.996 | 1.390 ± 0.112 | 0.982 | 0.675 ± 0.163 | 0.171 ± 0.101 | 0.982 | 1x10^4 ± 1.515 | 1.000 ± 0.081 | 0.933 | 1.012 ± 0.032 | 0.031 ± 0.150 | 0.933 |
| SORBITOL 60 150 | 1.415 ± 0.409 | 1.305 ± 0.087 | 0.967 | 0.546 ± 0.082 | 0.358 ± 0.090 | 0.968 | 2.253 ± 0.561 | 1.071 ± 0.119 | 0.975 | 0.427 ± 0.071 | 0.557 ± 0.106 | 0.982 |
| SORBITOL 60 150 | 2.711 ± 0.329 | 0.961 ± 0.046 | 0.984 | 0.410 ± 0.066 | 0.606 ± 0.088 | 0.984 | 2.359 ± 0.427 | 0.940 ± 0.057 | 0.978 | 0.413 ± 0.111 | 0.674 ± 0.147 | 0.977 |
| SORBITOL 60 150 | 3.122 ± 0.209 | 2.061 ± 0.315 | 0.854 | 0.326 ± 0.099 | 0.208 ± 0.135 | 0.849 | 3.237 ± 1.866 | 1.779 ± 0.269 | 0.863 | 0.346 ± 0.108 | 0.252 ± 0.140 | 0.857 |
| SORBITOL 60 150 | 5.096 ± 0.746 | 0.864 ± 0.075 | 0.973 | 0.190 ± 0.055 | 0.849 ± 0.149 | 0.973 | 6.683 ± 1.125 | 0.750 ± 0.107 | 0.960 | 0.073 ± 0.036 | 1.325 ± 0.274 | 0.973 |
| SORBITOL 60 150 | 3.191 ± 0.637 | 0.945 ± 0.075 | 0.971 | 0.340 ± 0.100 | 0.664 ± 0.158 | 0.970 | 3.320 ± 0.711 | 0.884 ± 0.074 | 0.975 | 0.254 ± 0.109 | 0.867 ± 0.239 | 0.977 |

Note: $K_1$ and $K_2$ are the parameters of Peleg’s model; $A$ and $B$ are the parameters of Page’s model; $R^2$ is the determination coefficient.
Figure 3. Experimental data and the fit of Peleg’s model for normalized AA of apple cubes during the osmotic dehydration in a 60 °Brix sucrose solution at atmospheric and vacuum (150 mbar) pressures and at 25, 40 and 60 °C (AA₀ = 7.42 ± 1.21 mg ascorbic acid/g DM).

Figure 4. Experimental data and the fit of Peleg’s model for normalized total phenolic content of apple cubes during the osmotic dehydration in a 60 °Brix sorbitol solution at atmospheric and vacuum (150 mbar) pressures and at 25 and 60 °C (TPC₀ = 4.77 ± 1.04 mg gallic acid/g DM).

Figure 5. Experimental data and the fit of Peleg’s model for normalized antioxidant activity of apple cubes during the osmotic dehydration in a 60 °Brix sorbitol solution at atmospheric and vacuum (150 mbar) pressures and at 25 and 60 °C (AA₀ = 7.42 ± 1.21 mg ascorbic acid/g DM).

The value of -1/k₁ (Peleg’s model) gives the initial rate of the kinetics of the TPC and AA. Based on this, it could be observed that the process variables (type of solute, pressure and temperature) did not affect the kinetics of TPC. The value of 1-1/k₂ is the equilibrium value given by Peleg’s model (Equation 3). The TPC at equilibrium did not follow a clear pattern and, in most conditions, the process variables did not affect the
final TPC. The same trend was observed with respect to the parameters of Page’s model (Equation 4). Concerning the AA, $k_1$ and $A$ parameters tend to present the same behavior. Based on Figures 2-5, OD condition of 25 °C and atmospheric pressure seems to preserve TPC and AA of apple cubes better during the process. These results on the quality of osmotically dehydrated apple cubes go along with the findings on the OD process rate, which increases when temperature increases (Assis et al., 2017a). As in the present work, Assis et al. (2017b, 2018) did not find a significant difference neither in TPC nor in AA between OD in sucrose and sorbitol solutions after 8 h at the same conditions.

4 Conclusions

The water activity of apple cubes was lower after osmotic dehydration (OD) with sorbitol solutions. The highest color changes were found in the vacuum experiments.

Peleg’s model was the most adequate to fit the total phenolic content (TPC) and antioxidant activity (AA) during the OD process.

In order to prevent color changes and preserve TPC and AA of apple cubes, we recommend OD at atmospheric pressure and 25 °C. Sorbitol did not seem to affect these parameters in relation to sucrose.

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