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ORIGINAL ARTICLE

Effect of ultrasonic and osmotic dehydration pre-treatments on the colour of freeze dried strawberries

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Abstract The effect of pre-treatments on the colour of freeze-dried strawberries was studied. Strawberries were subjected to different ultrasound and osmotic dehydration conditions followed by freeze-drying. Two concentration levels of sucrose solution (25 and 50 % w/w) and four levels of processing time (from 10 min to 45 min) were studied. Also, ultrasound application without using an osmotic solution was studied. Colour was quantified with a colorimetric analysis (CIE LCh). Sonicated strawberries presented higher lightness (L) and lower hue (h) than fresh and non-treated strawberries (control samples). The sonicated and osmo-sonicated strawberries have presented a more reddish and vivid colour then the control samples.

Keywords · Freeze-drying · Osmotic dehydration · Ultrasound · Colorimetric analysis · Strawberry

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Introduction

Freeze-drying has been used to obtain high quality and high value dehydrated fruits and vegetables. Drying takes place under sublimation conditions promoted by vacuum at low temperature. Freeze-drying offers several advantages compared to other food drying techniques, such as low degradation of shape, colour and size of the product, and higher retention of the fruit's organoleptic properties.

Strawberries became a common target for freeze-drying because they are used in many food products. Among the products currently available in the market containing freeze-dried strawberries are dry breakfast cereals, energy bars and yogurts. Freeze-dried strawberries are found to be of excellent colour and flavour, with high rehydration capacity (Shishehgarha et al. 2002).

Osmotic dehydration is known to improve the colour, flavour and texture of dried fruits (Kumar et al. 2004; Marani et al. 2007; Deng and Zhao 2008; Pani et al. 2008; Falade and Oyedele 2010; Sagar and Kumar 2010); and has already been applied to strawberries, with good results, by Marani et al. (2007) and Taiwo et al. (2003). The use of ultrasound pre-treatment has been applied to strawberries by Garcia-Noguera et al (2010a, b) and resulted in the production of a low calorie strawberry and also in the reduction of drying time. Ultrasound processing has also been applied to strawberries to evaluate its effect on anthocyanin content (Dubrovic et al. 2011).

This aim of this research was to study the effects of the ultrasonic pre-treatment on the colour of strawberries dehydrated by freeze-drying. The effect of different treatment times was evaluated and a comparison with osmotically treated strawberries was performed. Colorimetric analysis

(CIE LCh) was carried out to evaluate the colour differences between the strawberries subjected to the pre-treatments.

Materials and methods

Preparation of samples

Strawberries, *Camarosa* cultivar (mostly conic and long conic shaped), were purchased from a local retail market (Fortaleza, Brazil). Strawberries were cut in half along their long axes. Each half was weighed to select sample halves within a range between 8 g and 12 g. Strawberries were classified based on a relative standard of maturity, shape and colour. Only ripe strawberries characterized by full red colour covering the entire fruit were used in the experiments.

The initial moisture content of berries was determined by heating strawberry halves in a drying oven (Marconi model MA-085, Brazil) at 60 °C for 48 h following AOAC method 934.06 (AOAC 1990). The initial concentration of soluble solids (°Brix) of the berries was calculated reading the refractive index on a refractometer (Atago model AT35, Japan). The mean moisture content of fresh strawberries was 0.914±0.005 g-water/g-fruit. The initial soluble solids content of the fresh strawberry was 5.0±0.2 °Brix.

Pre-treatment

Each experimental unit consisting of 30 g of strawberries was immersed in 400 mL Erlenmeyer flasks filled with 120 mL of the pre-treatment solution. The weight ratio between the fruit and the pre-treatment solution was 1:4 to avoid dilution effects (Oliveira et al. 2006; Teles et al. 2006). The osmotic solutions were prepared mixing food-grade sucrose with distilled water until concentrations (% w/w sucrose in water) of 25 % and 50 % were attained. Samples were removed at 10, 20, 30 and 45 min after immersion. Experiments were carried out at 30 °C. All experiments were carried out in triplicate.

The ultrasonic pre-treatments were carried out using an ultrasonic bath (Unique model USC25, Brazil; internal dimensions: 24×14×9 cm; volume: 2.7 L) with ultrasonic frequency of 25 kHz and 60 W of power. Temperature of the liquid medium was maintained at 30 °C.

After completion of each pre-treatment, strawberry halves were removed from flasks, strained, and blotted with absorbent paper to remove excess solution.

The water loss (WL) and soluble solids gain (SG) were determined using the weight of strawberry halves before and after the pre-treatment trials, as well as the moisture content (wet basis) of strawberries, before and after pre-treatment. WL and SG were calculated according to equations 2 and 3, respectively.

$$WL(\%) = \frac{\left(w_i \cdot X_i - w_f \cdot X_f\right)}{w_i} \cdot 100 \quad (1)$$

$$SG(\%) = \frac{w_f \cdot X_{sf} - w_i \cdot X_{si}}{w_i} \cdot 100 \quad (2)$$

Where, w_i is the initial fruit mass (g) before pre-treatment; w_f is the final fruit mass (g) after pre-treatment; X_i is the initial fruit moisture content on wet basis (g water/g total fruit mass) before pre-treatment; X_f is the final fruit moisture content on wet basis (g water/g total fruit mass) after pre-treatment; X_{si} is the initial fruit dry solid matter content (g dry matter/g total fruit mass) before pre-treatment; X_{sf} is the final fruit dry matter content (g dry matter/g total fruit mass) after pre-treatment.

Freeze drying

The pre-treated strawberries were placed in glass Petri dishes in a single-layer arrangement and stored for 24 h in a freezer (General Electric model GE 360) at −25 °C. The dishes containing the frozen halves were removed from the freezer and placed in a single-layer on the shelf of a freeze dryer (Terroni model Enterprise I, Brazil) with evaporator temperature of −40 °C and an absolute pressure of 160 Pa. Samples were removed and assayed after 24 h of drying. The final moisture of the freeze-dried strawberries was calculated as 0.05±0.01 g-water/g-fruit.

Colorimetric analysis

Colorimetric analysis was performed on fresh strawberries and on freeze-dried strawberries. The CIE LCh system was used to represent colour in a three-dimension space. In the colour space, the L axis represents lightness. It ranges from 0, which has no lightness (absolute black), to 100, which is maximum lightness (absolute white). The C axis represents Chroma (or "saturation"). It ranges from 0, which is completely unsaturated (neutral grey, black or white) to 100 for very high Chroma (saturation) or "colour purity". The hue (h) is the angle that represents the saturated colour in the colour space. It is represented in degrees, which ranges from 0° (red), through 90° (yellow), 180° (green), 270° (blue) to 360° (red).

A colorimeter (Konica-Minolta model MD 400) was used for recording L, C and h values of the strawberries. Measurements were taken following colorimeter manufacturer recommendations. Reference L_{Ref}, C_{Ref} and h_{Ref} values (initial strawberries colour) and samples L, C and h values

were collected in triplicate. The results were presented as ΔL, ΔC and Δh, because each samples had slight differences in colour and the aim of the research was to evaluate the colour changes resulting from the pre-treatments. Five measurements of colour were made for each group of samples.

Experimental design and statistical analysis

An experimental design was used to study the effects of pre-treatment time, ultrasonic frequency and osmotic solution concentration on the colour parameters (L, C and h). The independent variables were osmotic solution concentration (OS) with three levels: 0, 25 and 50 % (w/w), and time (t) with four levels: 10, 20, 30 and 45 min. All experiments were carried out in triplicates and all measurements of colour parameters were also taken in triplicates. Means and standard deviations of the data were reported in the Results section.

Analysis of perturbation of factors was carried out with the data to evaluate the effect of time and osmotic concentration on colour difference. The analysis was also evaluated at a 90 % confidence level.

Differences in changes in colour as a function of time were analysed using Tukey HSD test at 90 % confidence level, using five replicates. All values in the figures were reported as mean±standard deviation.

Results and discussion

The L, C and h mean values for fresh strawberry were 50.2±0.9, 32.1±4.6 and 29.9±2.9, respectively. Freeze-dried strawberries (without any kind of pre-treatment) have presented ΔL, ΔC and Δh mean values of 2.98±1.27, −3.41±0.69 and −0.10±1.51, respectively. This change, in terms of colour, has produced a strawberry that is very similar to the fresh strawberry, since the h value changed very little and the higher lightness was compensated by a similar reduction in chroma.

Figure 1a presents the ΔL, ΔC and Δh mean values for freeze-dried strawberries subjected to ultrasonic pre-treatment at different immersion times. Two main changes were observed: an increase in lightness (L) and a decrease in hue (h). The change in chroma did not present any consistent tendency, and remained within the range of −1.1 and −5.4. The variations observed in chroma during immersion time were only cased by experimental errors.

Hue decreased with increasing immersion time, which is a positive and desirable result in strawberries, because the

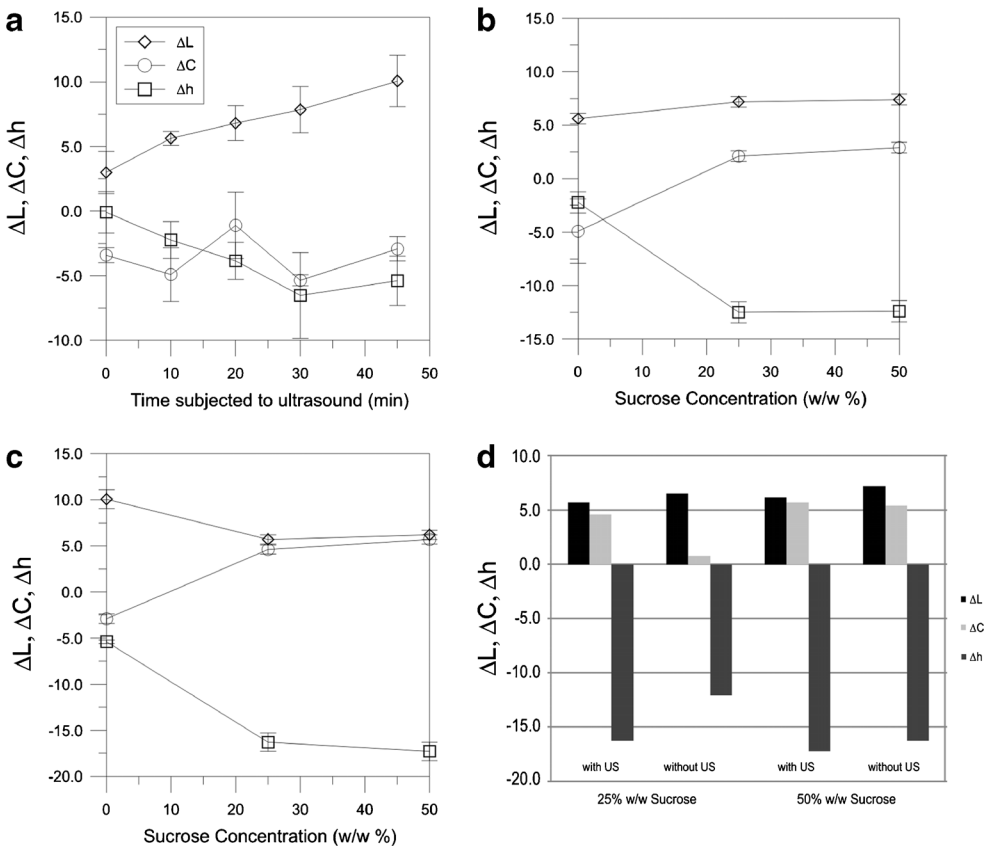

Fig. 1 Colour change (ΔL, ΔC and Δh) of freeze-dried strawberries subjected to ultrasound as a function of (**a**) processing times (Time t=0 min refers to the colour change of freeze-dried strawberries without any pre-treatment in relation to fresh strawberries - control group); (**b**) concentration of the osmotic solution (Processing time=10 min); (**c**) concentration of the osmotic solution (Processing time=45 min); (**d**) ultrasound application and concentrations of the osmotic solution (Processing time=45 min). Number of samples for each experiment (n)=5

decrease in hue produced a more reddish strawberry. The increase in lightness is also a positive contribution, because it will produce a brighter strawberry.

The use of an osmotic solution during sonication influenced the chroma and hue of the strawberries (Fig. 1b). Increasing sucrose concentration in the osmotic solution has increased the chroma value and decreased significantly the hue. Both changes are positive for the strawberry final colour, because the use of the osmotic solution has produced a more reddish fruit with a more vivid colour in freeze-dried strawberries. The hue of the strawberries treated for 10 min subjected to ultrasound and immersed in a 50 % w/w sucrose solution presented a value ranging from 8 to 10, which represents an almost pure red colour (h=0).

The sonication time did not significantly influence the final colour of the strawberries when an osmotic solution was used. Figure 1c shows that the changes in lightness, chroma and hue were similar whether a sonication time of 10 or 45 min were applied. This result may be explained by the similar amount of sugar incorporated during the process. At both processing times the sugar gain was 20±3 % and 31±2 %, respectively for the process carried out with a 25 and a 50 % w/w sucrose solution. Thus, the incorporated sucrose plays an important role in changing the lightness, chroma and hue values of freeze-dried strawberries.

The colour of the freeze-dried strawberries produced by sonication and by conventional osmotic dehydration is presented in Fig. 1d. The results show the tendency of a higher increase in chroma and hue when ultrasound is applied. Thus, in a general tendency, the sonicated strawberries will have a more reddish and vivid colour than strawberries subjected to conventional osmotic dehydration. The colour difference was more pronounced when a less concentrated sucrose solution was used. At higher concentrations of the sucrose solution, the colour differences between the processes were lower, but at the expense of a higher incorporation of sugar in the fruit.

The results showed an increase in water loss with increasing osmotic solution concentration, which is already expected because of the increase in the gradient between the soluble solids concentration in the fruit and in the osmotic solution. For strawberry treated in the 50 % sucrose solution, greater positive water loss was found as water diffused out of the strawberry tissue. The water loss was 2.6±0.3 % and 5.1±0.7 %, respectively for the process carried out with a 25 and a 50 % w/w sucrose solution for 45 min of immersion time. Water loss could not be directly correlated to changes in colour, especially because the amount of water lost during ultrasound-assisted osmotic dehydration was not expressive. Its effect on colour is more related to the concentration of solids in the fruit, which may impact the luminosity and chroma of the fruit. From this point of view, the slight increase in water loss that was observed has increased the solid concentration in the fruit, which lead to a slight increase in luminosity and chroma (Fig. 1b, c).

Table 1 presents the analysis of perturbation of factors for the application of ultrasound and osmotic dehydration pretreatments in freeze-drying of strawberries. The results show a negative effect of the concentration of the osmotic solution and the immersion time on lightness difference. The lightness showed to increase with increasing time at low osmotic gradient and to decrease with increasing time at high osmotic gradient.

Chroma difference was mainly influenced by the concentration of the osmotic solution, while the immersion time was not statistically significant. Hue difference was influenced by immersion time and by the concentration of the osmotic solution.

Tukey HSD test showed that the colour of the pre-treated strawberry is statistically different from the non pre-treated strawberry. Significant differences between the pre-treated and non pre-treated strawberries were observed especially in lightness and hue.

According to Meschter (1953), the most important factor in changing the kinetics of the degradation of colour in strawberry products is temperature. The rate of colour deterioration increases in proportion to the log of the temperature, which

Table 1 Analysis of perturbation of factors for freeze drying of strawberries

Variable	Effect	Standard Error	*p*
Effect on ΔL			
Mean	6.78	1.08	0.008
t	1.05	0.74	0.249
t^2	−0.40	0.48	0.460
OS	0.79	1.05	0.504
OS^2	1.25	1.97	0.571
OS×t [a]	−1.79	0.63	0.066
Effect on ΔC			
Mean	3.03	2.13	0.250
t	1.07	1.46	0.517
t^2	−0.04	0.94	0.966
OS [a]	7.01	2.06	0.042
OS^2	−5.72	3.87	0.236
OS×t	0.81	1.24	0.560
Effect on Δh			
Mean	−14.59	0.67	0.000
t [a]	−3.50	0.46	0.005
t^2 [a]	0.83	0.29	0.066
OS [a]	−10.37	0.64	0.001
OS^2 [a]	9.84	1.21	0.004
OS×t	−0.50	0.39	0.286

[a] significant factors. OS is the osmotic solution concentration and t is the processing time.

explains why colour degradation on freeze-dried strawberries is not observed.

Several sugars and sugar degradation products are capable of reacting with the pigment components of strawberries to increase their rate of loss (Francis 1989). Substances formed during the browning of sugars, such as furfural and hydroxymethylfurfural (HMF), may cause colour loss in strawberry products. These compounds are capable of reacting with pelargonidin 3-glucoside forming yellow chalcone species, which would result in an increase in the hue value (Yang et al. 2008). Sondheimer and Kertesz (1948a, b) also reported that colour degradation is proportional to the amount of sugars present in the strawberries.

The use of freeze-drying and thus, low processing temperatures prevents browning and the formation of yellow coloured species, which would compromise the final colour of the dried fruit. The low temperature may have prevented the formation of furfural and HMF. Consequently, the amount of sugar incorporated by the fruit during osmo-sonication and conventional osmotic dehydration where not degraded into forming dark coloured compounds.

Pelagonidin-3-glucoside degradation is slower when glucose, sucrose or maltose is added to the fruit. Sucrose also increases the stability of anthocyanins (Francis 1989). The absorbance of anthocyanins, such as pelagonidin-3-glucoside, increases when glucose, sucrose or maltose is added (Lewis et al. 1995). Anthocyanins colour increases upon the removal of water by displacement of the hydration/dehydration equilibrium toward coloured species (Brouillard 1983). This observation explains why the parameter L (lightness) have increased and why the parameter h (hue) have decreased during the osmotic and osmo-sonication processes.

Dubrovic et al. (2011) observed high stability of strawberries' anthocyanins after ultrasound processing. Only 4.4 % of the anthocyanins present in strawberry juice were degraded during ultrasound application, whereas a loss of 7.1 % was observed for the thermal processing of strawberry juice. As reported earlier, the red colour of strawberries is due to its main anthocyanin (pelagonidin-3-glucoside), which is not significantly affected by ultrasound processing. Thus, the results observed by Dubrovic et al. (2011) corroborates with the results presented herein that shows that hue values do not change significantly. Similar results were observed by Tiwari et al. (2010), which showed that pelagonidin-3-glucoside was only slightly affected by ultrasound application.

Conclusion

Ultrasound pre-treatment and osmotic dehydration enhanced the colour in strawberry samples, especially after prolonged ultrasonic exposure and applying high sucrose concentrations with the pre-treatment. Freeze-dried strawberry subjected to ultrasound presented a more reddish and vivid colour then the fresh and non-treated strawberries.

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