Optimization of ultrasonic osmotic dehydration of half Tunisian strawberry using response surface methodology/Box–Behnken design

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ABSTRACT:
Strawberries are largely produced in several Mediterranean countries such as Spain, Italia, Algeria and Tunisia. However, its perishability limits large-scale exportation and postharvest shelf life. Osmotic treatments with or without ultrasound is one of the simplest and inexpensive methods used for extending the shelf life of minimally processed strawberries. A suitable optimization of process can be an important tool for improving processes efficiency and quality. A lot of work has been done in the area of osmotic dehydration of strawberry. However, Response Surface Methodology (RSM) has still lacking as a modeling and optimization tool for ultrasound osmotic dehydration of strawberries. The objectives of the present study are to determine the effect of process parameters on sugar gain and water loss during osmotic dehydration of strawberry and to optimize these parameters using RSM. In the first step of the present study, a three factors with three levels of Box-Behnken response surface design were employed to characterize the UOD strawberry process, optimize and investigate the effect of independent variables (ultrasound time [X1, 0–30 min], concentration of osmotic solution (X2, 0–65 °Brix) and temperature [X3, 20–40 °C]) on the target responses, such as the water loss (Y1), solid gain (Y2) and weight reduction (Y3). In the second step, the same Box–Behnken design and the same methodology was used to estimate the main effects of OD without ultrasound treatment.

It was found that concentration of sucrose solution and UOD time were the most significant factors affecting the WL during ultrasound-osmotic dehydration of strawberry followed by temperature. The most important linear effect for SG were the °Brix followed by the UOD time, The efficiency of SG was also affected significantly by all factor interactions such as UOD time x Temperature and UOD time x °Brix.

Response surface methodology with Derringer’s desired function methodology was used to determine the optimum processing conditions that yield maximum water loss and weight reduction and minimum solid gain during osmotic dehydration of Strawberry. The optimum solution by numerical optimization obtained was 47.5°Brix osmotic solution concentration, 31°C osmotic solution temperature, and 20.5 min of UOD duration to get maximal possible WL and WR and minimal SG. At this optimum conditions, WL, WR, and SG were found to be 9.38%, 9.77%, and - 0.387%, respectively. For the osmotic dehydration experiments without ultrasound the Derringer’s desirability function method was employed at obviously optimum conditions to determine the parameters for WL, WR, and SG. This parameters were found to be 10.68%, 10.92 and -0.235%, respectively. Then the use of ultrasound decreases the amount of soluble solids, especially sugar, of the fruit and produces a dried low-sugar fruit.

Keywords : Strawberry . Ultrasound . Osmotic dehydration . Solid gain. Response surface methodology

1. Introduction

Strawberries are largely produced in several countries with a production over 4.3 million tons in 2011 according to FAOSTAT (Food and Agriculture Organization 2013) and are sold both to internal and external markets. Strawberries have the anti-hyperglycemic potential by the capacity to inhibit R-glucosidase concurrently with low inhibition of R-amylase, likely resulting in less unwanted side effects [1].

Consumption of healthy dehydrated strawberry, which can be fresh-cut eaten directly or become part of foodstuffs (e.g., cookies, breakfast cereals, cakes, energy bars and dairy products) has attracted a noticeable increase [2]. Consequently, one of the simplest and inexpensive methods used for extending the shelf life of minimally processed strawberries is to apply osmotic treatments with or without ultrasound in order to reduce water activity and solid gain in the product [3].
Osmotic dehydration (OD) naturally occurs in fruits, following placement in hypertonic solutions presenting a high osmotic pressure and low water activity. A diffusion phenomenon takes place with two simultaneous opposite flows: a water outflow from the food to the solution and a simultaneous inflow of solute from the solution to the food. These mechanisms lead to a water loss (WL) and solids gain (SG) from and into the food. It is often desired in this case, maximum possible WL along the minimum possible SG [4].

Emergent new technologies, ultrasonic waves are often used to strengthen food osmotic dehydration. [3, 5-6] Ultrasonic waves in frequency range of 20–100 kHz can generate in solid media quick series of alternative compressions and expansions in an analogue way to a sponge when it is squeezed and relaxed repeatedly [7].

Varying effects of the ultrasound-assisted osmotic dehydration have been shown on different fruit materials. The influence of US application on the osmotic dehydration rate depends on the magnitude of the process variables involved as well as the product’s structure. Due to the very different results obtained for different fruit, more studies on the effect of ultrasound on osmotic dehydration are still needed [3].

Response Surface Methodology (RSM) is widely used as a statistical technique in process optimization and product quality amelioration in a short time period and minimum experiences [8, 9]. This study has investigated the use of ultrasound as a strengthening treatment to osmotic dehydration of strawberry. Hence, in this research, RSM, Box–Behnken quadratic design (BBD) coupled with Derringer’s desired function methodology was used to optimize and study the induction of the key process variables such as ultrasound time, and concentration and temperature of osmotic solution (independent variables) on WL, WR and SG (dependent variables).

2. Materials and methods

2.1. Raw material

Fresh strawberries were purchased from a local market in Tunis (Tunisia) and stored in a refrigerator at 4°C for maximum 4 days until use. The fruits were selected according to their appearance and physical damage. The strawberries were removed from refrigeration and left to equilibrate at the room temperature before experimentations. Then, the fruits were washed and dried with absorbent tissue paper. After that, the peduncle was removed and the strawberries were cut they were manually cut with a stainless steel knife parallel to the main axis into semi-spherical halves. From each fruit, 2 halves were obtained. Then, they were weighed individually. The first half untreated strawberry (blank) was used directly to characterize the fresh product by measuring its °Brix and the moisture content.

2.2 Osmotic Treatment

The osmotic solutions were prepared using commercial sugar and distilled water to give a concentration of 32.5 and 65 °Brix. The osmotic dehydration was carried out in separate 100-ml Erlenmeyer flasks.

2.3 Ultrasound-assisted Osmotic Dehydration

The second weighted strawberry half was immersed in distilled water or in the osmotic solution and was subjected to ultrasonic waves. The 100-ml Erlenmeyer device used to avoid interference between the samples and runs was covered with a plastic plate to reduce moisture loss from osmotic solution during experiments then was placed on an ultrasound cleaner bath (frequency 40 kHz, power 120 W and internal dimensions: 15/14/24cm; volume: 3 L) during a period of 10 to 30 min. The solution/fruit ratio was set at 4 : 1 (w/w). This ratio was desirable in considering the small volume of the ultrasonic bath [3, 6].

The ultrasonic bath is filled with a known amount of water (2 L) and the water is let resting in the bath until it reaches equilibrium with desired temperature. The experiments were carried out at 20, 30 and 40°C. The ultrasound equipment is turned on and the water temperature is measured. The temperature increase during the experiments was not significant (less than 2 °C at 40°C) after 30 min of ultrasonic treatment.

To evaluate the effect of ultrasound, the same procedure was realized in the ultrasound cleaner but without application of ultrasound. The experiments were carried out in triplicates and the mean values were used. When the immersion time was reached (respectively 10, 20, and 30 min), sample was withdrawn from the glass beakers and quickly rinsed with fresh running water to withdraw excess solution. It was subsequently slightly wiped with an absorbent paper and weighted using an analytical balance with an accuracy of 0.0001 g.
[11]. Moisture content of the samples was determined by a gravimetric method, according to AOAC 930.04 (AOAC, 1990) [10]. Weighed Sample was placed in a drying oven at 105 °C for 24 h until a constant weight was reached. The samples were cooled down in desiccators and weighed. Moisture content and solid content of the samples were then calculated from the sample weights before and after drying. The °Brix of osmotic solution was measured simultaneously with a digital refractometer (Reichert Leica AR200). The water loss and solid gain were calculated according to the method proposed by [12]. In this method, WR, WL and SG are calculated from the weight of samples and moisture content.

2.4 Statistical analysis

2.4.1 Experimental Design and Statistical Analysis

In the first step of the present study, three factors with three levels of Box-Behnken response surface design were employed to characterize the UOD strawberry process, optimize and investigate the effect of independent variables on the target responses, such as the water loss (Y1), solid gain (Y2) and weight reduction (Y3). The uncoded (actual) and coded levels of the independent variables, which were selected based on the results from literature experimentation, are given in Table 1. This generated 15 experiments with three replications at the center point (Table 1). In the second step, the same Box–Behnken design and the same methodology was used to estimate the main effects of OD without ultrasound treatment.

Table 1: Box-Behnken design matrix of real and coded values used for ultrasound OD process.

<table>
<thead>
<tr>
<th>Factors</th>
<th>Coded values</th>
<th>Uncoded values</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Low (-1)</td>
<td>Central (0)</td>
</tr>
<tr>
<td>X1: Ultrasound-Osmotic</td>
<td>10</td>
<td>20</td>
</tr>
<tr>
<td>time (min)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>X2: °Brix (%)</td>
<td>0</td>
<td>32.5</td>
</tr>
<tr>
<td>X3: Temperature (°C)</td>
<td>20</td>
<td>30</td>
</tr>
</tbody>
</table>

Run 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15
X1 10 30 20 20 10 30 20 20 20 20 10 30 10 30
X2 65 65 65 65 0 0 0 32.5 32.5 32.5 32.5 32.5 32.5 32.5
X3 30 30 20 40 30 30 20 40 30 30 20 20 40 40

2.4.2 Optimization

The RSM was applied to the experimental data using the software STATISTICA (Trial Version Statsoft, Inc, 2010). A regression polynomial model with 10 coefficients, including quadratic and linear effects of factors and linear effect of interactions, was supposed to depict relationships between response (Y) and the independent variables (X1,X2,X3) as follows:

\[ Y = \beta_0 + \beta_1X_1 + \beta_2X_2 + \beta_3X_3 + \beta_{12}X_1X_2 + \beta_{13}X_1X_3 + \beta_{23}X_2X_3 + \beta_{11}X_1^2 + \beta_{22}X_2^2 + \beta_{33}X_3^2 \]  

(1)

Where Y is a response variable of removal efficiency (%), \( \beta_0 \) is the estimated coefficient of regression of the fitted response at the center point, \( \beta_1, \beta_2 \) and \( \beta_3 \) are linear coefficients, \( \beta_{12}, \beta_{13} \) and \( \beta_{23} \) are cross product coefficients, \( \beta_{11}, \beta_{22} \) and \( \beta_{33} \) are quadratic coefficients. The \( \beta \) coefficient is that the amplitude of these values gives ideas to the contribution of each factor in the prediction of the response. Plus the positive value of \( \beta \) of a parameter, higher would be the impact of that parameter, while negative term indicates an antagonistic effect upon the response.
3. Results and Discussion

In order to evaluate the combined effect of different levels of ultrasound-osmotic dehydration time (X1), °Brix of osmotic solution (X2) and Temperature of medium (X3) on SG (Y1), WL (Y2) and WR (Y3) and to determine the maximum WL and minimum SG transfer correspondent to the optimum levels, a quadratic polynomial model (Eq. 1) was suggested to compute the optimum levels of factors. By carrying out the multiple regression testing on trial data, a quadratic model in coded unit shows the role of every variable and their interactions in independent variables. Estimated regression coefficients of the second-order polynomial for ultrasound assisted osmotic dehydration of strawberry on SG, WL and WR model are shown in Table 2.

Table 2: Parameters estimation for BBD design experiments during ultrasound assisted OD of half strawberry.

<table>
<thead>
<tr>
<th>Source</th>
<th>Solid Gain SG (%)</th>
<th>Water Loss WL (%)</th>
<th>Weight reduction WR (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mean/Intercet.</td>
<td>-0.555</td>
<td>0.073</td>
<td>0.00065</td>
</tr>
<tr>
<td>(1) UOD time (min)(L)</td>
<td>0.245</td>
<td>0.045</td>
<td>0.0285</td>
</tr>
<tr>
<td>UOD time (min)(Q)</td>
<td>0.148*</td>
<td>0.066</td>
<td>0.07596</td>
</tr>
<tr>
<td>(2) °Brix (%)(L)</td>
<td>0.312</td>
<td>0.045</td>
<td>0.00097</td>
</tr>
<tr>
<td>°Brix (%)(Q)</td>
<td>-0.007*</td>
<td>0.066</td>
<td>0.91019</td>
</tr>
<tr>
<td>(3) Temperature (°C)(L)</td>
<td>-0.115*</td>
<td>0.045</td>
<td>0.05097</td>
</tr>
<tr>
<td>Temperature (°C)(Q)</td>
<td>0.156*</td>
<td>0.066</td>
<td>0.06502</td>
</tr>
<tr>
<td>1L by 2L</td>
<td>0.267</td>
<td>0.063</td>
<td>0.00856</td>
</tr>
<tr>
<td>1L by 3L</td>
<td>0.389</td>
<td>0.063</td>
<td>0.00172</td>
</tr>
<tr>
<td>2L by 3L</td>
<td>0.296</td>
<td>0.063</td>
<td>0.00563</td>
</tr>
</tbody>
</table>

L: linear; Q: quadratic; * Non-significant (p value >5%).

The sufficiency of the model was evaluated through analysis of variance (ANOVA). Moreover, the variation of the data around the fitted model designed by the Lack of Fit was also checked. In the present study with regards to WL and WR, the Lack of Fit is not significant relative to the pure error, indicating good response to the model (results not shown). ANOVA for SG, WL and WR, respectively indicated that the second-order polynomial model (Eq. (1)) was adequate to represent the actual relationship between the response and the variables, with a high value of coefficient of determination (R^2 = 0.9714 for SG, R^2 = 0.989 for WL and R^2 = 0.9897 for WR).

It can be seen from the magnitude of P-value from Table 2 that all linear terms have significant effect at 5% level of significance except for the process temperature no significantly affect SG. The main effects of UOD time and °Brix on all independent variables were highly significant as was evident from their respective P-values (0.00005< p <0.00285 ). Interaction term of X12 and X13 were significant at 5% level. Only the quadratic term of ultrasound time and temperature has significant effect on WL and WR at 5% level of significance (P < 0.05). The Temperature has significant negative effect on WL and WR in quadratic term. The result also indicated that Temperature could act as limiting factors, and increase in their values will considerably alter either growth rate. The interaction between UOD time and Temperature had significant effects on the SG (P<1X3 < 0.00172).

The final mathematical equation in terms of significant actual factors determined by STATISTICA software for SG, WL and WR and are given in Eq. (2), 3 and 4, respectively.

\[
SG = 4.74 - 0.178x_1 - 0.33x_2 - 0.213x_3 + 8.23 \cdot 10^{-4}x_1x_2 + 3.89 \cdot 10^{-3}x_1x_3 + 9.11 \cdot 10^{-4}x_2x_3 \quad (2)
\]

\[
WL = -16.59 - 0.018x_1^2 + 1.43x_1 - 0.026x_2^2 + 7.7 \cdot 10^{-3}x_1x_2 + 1.33 \cdot 10^{-2}x_1x_3 \quad (3)
\]
\[ WR = -21.33 - 0.011X_1^2 + 1.64X_3 - 0.0274X_1^2 + 6.8 \cdot 10^{-3} x_1 x_2 + 9.4 \cdot 10^{-3} x_1 x_3 - 2.1 \cdot 10^{-3} x_2 x_3 \] (4)

The result also could be explained by the frequency histogram Pareto chart of effects (Figure 1). Pareto analysis is a technique helps to prioritize and focus resources visually, that shows the amount of influence each factor has on the response in decreasing order. As it can be seen (Fig. 1 (A)), the most important linear effect for SG were the °Brix followed by the UOD time, The efficiency of SG was also affected significantly by all factor interactions such as UOD time x Temperature and UOD time x °Brix. Nevertheless, an excess of °Brix producing negative influence on the SG but is it statically non-significant.

![Figure 1](image_url)

<table>
<thead>
<tr>
<th>X2</th>
<th>X1*X3</th>
<th>X2*X3</th>
<th>X1*X2</th>
<th>X3</th>
<th>X3*X2</th>
<th>X1*X2</th>
<th>X2*X3</th>
</tr>
</thead>
</table>

Figure 1. Pareto chart of standardized effect of the main effects for SG: (A) of UOD strawberry and (B) of NUOD strawberry. The vertical dashed line indicated the level of significance at p=0.05.

A Pareto chart of the main effects for SG of experiments carried out without application of ultrasound is shown in figure 1 (B). It was observed the same tendency as obviously and the most important linear effect for SG were the °Brix followed by the OD time, meanwhile with slightly inferior coefficients. This difference may be explained by the effect of ultrasound treatment. The use of ultrasound also decreases the amount of soluble solids, especially sugar, of the fruit and produces a dried low-sugar fruit. Same tendency have been shown in the UOD of pineapple and papaya [13, 14].

The Derringer’s desirability function method was employed to optimize the process variables, which would donate maximum WL, WR and minimum SG. As shown in Figure 2 the optimum conditions were found to be UOD time of 20.5 min, osmotic solution concentration of 47.5 °Brix, and osmotic temperature of 31 °C, respectively. At this optimum conditions, WL, WR, and SG were found to be 9.38%, 9.77%, and -0.387%. For the osmotic dehydration experiments without ultrasound the Derringer’s desirability function method was employed at this optimum conditions to determine the parameters for WL, WR, and SG. This parameters were found to be 10.68%, 10.92 and -0.235%, respectively. The small high obtained WL for NUOD can be explained as reported by [6] that the effect of the ultrasound-assisted osmotic dehydration on the water diffusivity depended on the degree of breakdown of cells in the fruit tissue.

4. Conclusion

This work evaluated the production of osmotic-dehydrated strawberry with low sugar content. The produce was obtained with applying ultrasound to decrease the sugar uptake from the fruit. The influences of operation parameters such as ultrasound and OD time, solution concentration and system temperature on water loss and solid gain are discussed. The effects of ultrasonic osmosis on weight reduction are also studied. Response surface methodology was used to establish the optimum operating conditions that yield maximum water loss and weight reduction and minimum solid gain in osmotic dehydration of strawberry. The second-order polynomial models for all the response variables were found to be statistically significant. The optimal conditions for maximum water loss and weight reduction and minimum solid gain, were 20.5 min for UOD time, 47.5 °Brix osmotic solution concentration and 31°C medium temperature in order to obtain water loss of 9.38 (g/100 g fresh sample), weight reduction of 9.77 (g/100 g fresh sample), and solid gain of -0.387 (g/100 g fresh sample). OD concentration, US duration and Temperature increase the amount of water removed during OD.
However, the ultrasound waves decreases the intake of solids by the product. Then ultrasound OD is better appropriated if the WL should be increased and the solids (sugar) uptake should be limited (dietetic products).

**Figure 2:** Desirability charts of variables for maximum response for UOD strawberry.

### 5. References


