OPTIMIZATION OF EXTRUSION PROCESS OF RICE FLOUR ENRICHED WITH PISTACHIO NUT FLOUR

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ABSTRACT

Response surface methodology deriving by superimposing individual contour plots, was used to investigate the optimum operating conditions for extrusion-cooking of rice flour enriched with pistachio nut flour. The highest barrel temperature (128°C) produced a stiff extrudates (high values of breaking strength i.e. 100 N/mm² and bulk density i.e. 2.2 g/mL). However, graphical optimization studies showed that the optimal operating conditions involved values of 16-17% water feed content and 70-95°C barrel temperature. This research points out the importance to study the biopolymer changes that occur during extrusion-cooking processing because of their huge effect on quality characteristics of extrudates.

- Keywords: pistachio nut flour, starch-lipid complexes, optimization response surface methodology, contour plot, breaking strength, bulk density -
1. INTRODUCTION

Nowadays, consumers prefer foods easy and convenient to eat (SCHWARTZ, 2009). Snacks and breakfast cereals are easy to carry, purchase and consume but they are essentially produced from starchy substances such as corn, rice, wheat (YASEEN and SHOUK, 2005) and therefore they could lack some important nutrients. Foods with poor nutritional value, lack in micronutrients such as vitamins, minerals, amino acids, fibers and high content of calories can be considered unhealthy. For this reasons, researches are focused on the improvement of nutritional characteristics by the addition of ingredients such as fruit, nuts, fibers, etc. Among nuts, pistachios could favourably be used thanks to their ability to lower the risk of cardiovascular diseases, to improve to a function of the process variables were also established.

2. MATERIALS AND METHODS

2.1 Raw materials

Rice starch (10.9% moisture) was provided by A.D.E.A. (Bursto Arsizio, Italy); pistachio nut flour was provided by Cartellone (Bronte, Italy); oleic acid was provided by Sigma-Aldrich (Milano, Italy).

The used pistachio nut flour had a moisture content of 4.8±0.2% and the following chemical composition (dry basis): protein (18.1±0.1%); lipid (49±0.5%); starch (3.3 ±1.5%); soluble sugars (4.5±0.2%); fiber (10.6±2%) and ash (9.7±0.1%).

The fat acid composition of lipid fraction of pistachio nut flour, determined according method proposed by RATNAYAKE et al. (2006) was: C14:0 (0.09); C16:0 (9.45); C16:17 (0.86); C17:0 (0.04); C17:1 (0.07); C18:0 (2.12); C18:1 (70.17); C18:2 (15.5); C18:3 (0.32); C20:0 (0.18); C20:1 (0.48); C22:0 (0.09); C24:0 (0.04).

The chemical characteristics of tap water used for extrusion trials was: pH 7.7 ± 0.1, hardness (°f) 25.1±1.5, total dissolved solids dried at 180°C 645±38.5 mg/L and chloride content 54.6±0.4 mg/L.

The content of moisture, ash, protein and fat of flours were determined according to the 44-15A, 08-01, 46-10, 30-25 AACC International Approved Methods (2003).

2.2 Extrusion experiments

According to previous studies (DE PILLI et al., 2011), the formula containing 75% rice starch and 25% pistachio nut flour was used. The extrusion experiments were carried out using a Thermo Prism PTW-24 (Thermo Haake PolyLab System, Germany) co-rotating twin-screw extruder. The screw geometrical features were the following: diameter 24 mm and length 672 mm (L/D = 28:1) and distance between shafts 19 mm. Fig. 1 reports the screw configuration used. During extrusion experiments, the screw speed was kept constant at 140 rpm, as well the flour feed rate was kept constant at 2.8 kg/h (dry weight). The flours were proportioned by volumetric gravity feeder. The extruder was divided into six zones, independent of each other for temperature control and adjustment. For all experiments, the first two zones were kept at 35 and 65°C respectively, whereas the last four zones were adjusted at the same temperature according to experimental plan (Table 1).
The water was pumped to the first zone of the extruder and the delivery capacities of water pump were 7.5; 7; 6; 4.75; 4.25 L/h. These values were chosen to obtain the moisture feed content of dough indicated in the experimental plan (Table 1).

The die used had a spherical shape (diameter 300 mm) in which there was one circular hole with a diameter of 5 mm. At the exit of the die, the extrudates were manually cut into sticks (about 50 mm in length) using a knife. The extrudates were dried over night at 40°C in a vacuum dryer.

Table 1 - Coded and actual values of variables (A) and the arrangement and responses of factorial design (B).

<table>
<thead>
<tr>
<th>A)</th>
<th>Coded Level</th>
<th>Uncoded</th>
<th>Barrel temperatures of last four zones (X1) (°C)**</th>
<th>Water feed content (X2) (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>128</td>
<td>21.8</td>
</tr>
<tr>
<td></td>
<td>+1.4</td>
<td></td>
<td>120</td>
<td>21.0</td>
</tr>
<tr>
<td></td>
<td>+1</td>
<td></td>
<td>100</td>
<td>19.0</td>
</tr>
<tr>
<td></td>
<td>0</td>
<td></td>
<td>80</td>
<td>17.0</td>
</tr>
<tr>
<td></td>
<td>-1</td>
<td></td>
<td>72</td>
<td>16.2</td>
</tr>
<tr>
<td></td>
<td>-1.4</td>
<td></td>
<td>72</td>
<td>16.2</td>
</tr>
</tbody>
</table>

**The first two zones were kept at 35° and 65°C respectively, whereas the last four zones were adjusted according to experimental plan.

<table>
<thead>
<tr>
<th>B)</th>
<th>Coded Level</th>
<th>Processing variables (X1) (°C); (X2) (%)</th>
<th>Responses</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>X1</td>
<td>X2</td>
<td>Y1 (%)</td>
</tr>
<tr>
<td>1</td>
<td>-1</td>
<td>1</td>
<td>23.67±0.02</td>
</tr>
<tr>
<td>2</td>
<td>-1</td>
<td>1</td>
<td>23.00±0.06</td>
</tr>
<tr>
<td>3</td>
<td>1</td>
<td>-1</td>
<td>60.00±0.03</td>
</tr>
<tr>
<td>4</td>
<td>1</td>
<td>1</td>
<td>60.00±0.01</td>
</tr>
<tr>
<td>5</td>
<td>-1.4</td>
<td>0</td>
<td>20.00±0.07</td>
</tr>
<tr>
<td>6</td>
<td>-1.4</td>
<td>0</td>
<td>70.00±0.01</td>
</tr>
<tr>
<td>7</td>
<td>0</td>
<td>-1.4</td>
<td>52.45±0.01</td>
</tr>
<tr>
<td>8</td>
<td>0</td>
<td>1.4</td>
<td>52.00±0.03</td>
</tr>
<tr>
<td>9</td>
<td>0</td>
<td>1</td>
<td>51.50±0.02</td>
</tr>
<tr>
<td>10</td>
<td>0</td>
<td>0</td>
<td>52.00±0.03</td>
</tr>
<tr>
<td>11</td>
<td>0</td>
<td>0</td>
<td>50.80±0.01</td>
</tr>
</tbody>
</table>

X1: barrel temperatures of last four zones (°C); X2: water feed content (%); Y1: complex index; Y2: \(\lambda_{\text{max}}\); Y3: breaking strength; Y4: bulk density.
uum oven Salvis Vacucenter VC 50 (Salvis AG, Reussbühl/Lucerne, Switzerland). Samples used to determine complexing index and iodine spectrum of the soluble fractions of the extrudates were finely ground (particles < 300 microns) using a Bühler ML 1204 mill (Germany). All the ground samples were defatted in a Soxtec fat-extractor with petroleum ether at 37°C (bp 34.6°C) for 155 min to remove uncomplexed lipids before submit samples to chemical analyses (BHATNAGAR and HANNA, 1994).

2.3 Experimental plan

The extrusion experiments were carried out at five temperature profiles (Table 1a) and percentages of water feed contents (21.8%, 21.0%, 19.0%, 17.0%, 16.2% expressed as percentage of dry basis). All extrusion experiments were performed at least in triplicate.

Coded and actual values of variables are shown in Table 1a, the factorial design of two variables (temperature profile and feed water content) and five levels of values were used according to Central Composite Design (CCD) (BOX et al., 1978). This method was used to evaluate the single influences of the processing variables as well as their possible interactions. Eleven tests (Table 1b) with different combinations of process variable values were obtained.

2.4 Complexing Index

Complexing Index (CI) was determined using the method described by GURAYA et al. (1997). The iodine solution used for analysis was prepared by dissolving overnight 2 g of potassium iodine and 1.3 g of I₂ in 50 mL distilled water. Then the final volume was made to 100 mL using distilled water. A 5 g sample was mixed with 25 mL of distilled water in a test tube. The test tube was vortexed for 2 min and centrifuged for 15 min at 314.1 rad/s. The supernatant (500 ml) and distilled water (15 mL) were added to the iodine solution (2 mL). The tube was inverted several times and absorbance was measured at 690 nm through a UV/VIS spectrophotometer (Beckman DU 640, California). CI was calculated from the following equation (2):

\[
CI(\%) = \frac{(Abs_{control} - Abs_{sample})}{Abs_{control}} \times 100
\]  

The analysis was carried out in triplicate.

2.5 Iodine Spectra of Starch Samples

Starch samples were solubilised in 1 N NaOH as recommended by SCHOCH (1964). The absorbance spectra of starch-iodine complexes were measured using a spectrophotometer UV/VIS Perkinelmer Lambda 25 (Milan, Italy) from 400-700 nm, and wavelength of maximum absorption (\(\lambda_{max}\)) values were determined.

2.6. Breaking strength (N/mm²)

A stable dynamometer Micro System TADi Texture Analyser (ENCO s.r.l., Venezia, Italy) with a plunger was used for texture analysis. Extrudates were placed over two supports, 1.5 cm apart, and broken in the middle by a plunger that had a shape of a cone frustum (the thickness of contact surface with extrudate was 1 mm² and the speed was kept constant to 0.5 mm/s). Results were expressed as breaking strength (N/mm²), i.e. the strength needed to break the extrudate. This index is related to microstructure of samples and it simulates the incisors impact at biting (VAN HECKE et al., 1998). For each sample, at least ten repetitions were carried out.

2.7 Bulk Density (BD)

Bulk density was measured using a displacement method (YU et al. 2012). Extrudates were cut into strands of about 25 mm long and about 10 g strands were weighed (M, grams) and put in a 100 mL cylinder; then yellow millet particles were added to fill up the cylinder. The extrudates were taken out, and the volume of the yellow millet particles was measured (V, milliliters); ten measurements were performed to calculate the average. Bulk density (BD) was calculated as equation (3):

\[
BD = \frac{M}{100} \times \frac{g}{ml}
\]  

2.8 Statistical analysis

Data were submitted to statistical analysis using Statsoft, vers. 5.1 (Statsoft, Tulsa, USA) software. The analysis was carried out in two steps. The first involved a stepwise regression analysis to identify the relevant variables, and the second used a multiple regression analysis (Standard Least Square Fitting) to fit a second order mathematical model, according to the following polynomial equation:

\[
y = B_0 + \Sigma B_i x_i + \Sigma B_{ii} x_i^2 + \Sigma B_{ij} x_i x_j
\]

where y is the dependent variable (complex index, iodine spectrum of the soluble fractions of the extrudates; breaking strength and bulk density of extrudates), B₀ is a constant value, \(x_i\) and \(x_j\) are the independent variables (barrel temperature and water feed content) in coded values and Bi, Bii and Bij are the regression coefficients of the model. This model allowed the effects of the linear (\(x_i\)), quadratic (\(x_i^2\)) and combined (\(x_i x_j\)) terms of the independent variables to be assessed on the dependent variable.
Variables with a significance lower than 95% (p>0.05) were left out of the equation. Isoresponse surface were developed in order to describe both individual and interactive effects of the independent variables of the extrusion-cooking process on complex index, iodine spectrum of the soluble fractions of the extrudates; breaking strength and bulk density of extrudates.

Extrusion processing parameters were optimized by using the Design-expert version 8.07.1 (Stat-Ease Inc., Minneapolis, USA) through a conventional graphical method of RSM in order to obtain extrudates with acceptable properties. All the processing variables were kept within a range while the responses were either minimized (breaking strength and bulk density). Contour plots of all the responses were then superimposed, and the optimum region appeared. The contour plots were obtained by superimposing of contour plots from which one could determine the optimum process variables range (barrel temperature and water feed content) to obtain extrudates made up of rice and pistachio nut flour with specified properties.

3. RESULTS AND DISCUSSION

Fig. 2a shows the complexing index values as a function of barrel temperature and water feed content. The barrel temperature was the only processing variable that had a significant effect on complexing index. In particular, values of the complexing index increased with increasing of barrel temperature (Fig. 2a). This means that, in this case, the highest barrel temperatures did not involve the melting of starch-lipid complexes. It is possible to suppose that the presence of other components in the dough increase the melting temperature of starch-lipid complexes, that result then more protected by heating during processing. Moreover, the presence in lipid fraction of triglycerides, di-glycerides and fatty acids involves an increase of characteristic melting temperature of starch-oleic complexes (DE PILLI et al., 2008b; 2011).

To confirm the formation of starch-lipid complexes, values of $\lambda_{\max}$ for native starch extruded with and without pistachio nut flour were also determined. Rice flour, extruded without nut flour and with an amylose contents of 89 %, showed $\lambda_{\max}$ within 592-595 nm.

Fig. 2b shows that the increase of barrel temperature shifted $\lambda_{\max}$ from 595 nm towards the amylopectin side (520 nm), due to the decrease of available amylose that is bounded with lipids. These results are in agreement with those of complex index and confirm the formation of starch-lipid complexes (Fig. 2a).

In Fig. 3a is reported the break strength (BS) values as a function of barrel temperature and water feed content. Also in this case, the only variable that had a significant effect on microstructure of the extrudates was the barrel temperature. In particular, the extrudates obtained at the highest values of barrel temperature (128°C) opposed the highest resistance to break, while low values of break strength were obtained at the lowest barrel temperature (70°C) (Fig. 3a). The formation of starch-lipid complexes obtained with the increase of barrel temperature could explain the high compactness of extrudates (BHATNAGAR and HANNA, 1994; DE PILLI et al., 2008a,b).

Data of bulk density (BD) are in agreement with those of the break strength. In fact, the extrudates showed high values of bulk density at the highest barrel temperature (Fig. 3b). The increase of bulk density and break strength values of extrudates may be caused by an alteration in the ratio between free amylose and amylopectin. According to GUY and HORNE (1988), the elastic character of the molten extrudates creates a swell at the die that controls the overall phenomenon of expansion of the extrudates. LAUNAY and LISCHE (1983) suggested that amylose–lipid complex formation was the key factor influencing the flow properties of starch pastes. When starch is extruded, expansion is dependent on the formation of a starch matrix that entraps the water vapor, resulting in the formation of bubbles (GUY and HORNE, 1988).
is reasonable to speculate that the addition of lipids might have affected the character of this matrix (i.e., the viscoelastic properties of molten extrudate) so that it could no longer hold water vapor, resulting in lower expansion and higher break strength. The increase of bulk density and break strength caused by decrease of swelling of starch can also be compared to that one of native starch upon gelatinization. Swelling is generally considered a property of amylopectin while amylose is considered a diluent. The amylose and native lipids contained in cereal starches may inhibit swelling under particular conditions when amylose–lipid complexes are likely to be formed (TESTER and MORRISON, 1990). According to KROG (1973), complex formation with the linear component of starches makes the structure more rigid and stabilizes the swollen granule against breakdown, resulting in restricted swelling. These statements are in agreement with DE PILLI et al. (2008b).

In this study, a conventional graphical method of multiresponse optimization technique was applied to obtain the combination of optimum process variable for the production of extrudates enriched with pistachio nut flour. To determine the extrudates with acceptable properties, main criteria of optimization constraints were related to bulk density (< 1.2 g/mL) and breaking strength (< 40 N/mm²). Superimposing the individual contour plots for the product response variables resulted in the identification of a region (shown by the blank space area) that satisfied all constraints as shown in Fig. 4. Superimposed contour plots indicated the ranges of variables that could be considered as the optimum range to obtain the best characteristics of extrudates in terms of bulk density and braking strength. The optimum ranges of variables obtained from the superimposed contours were 16–17 % water feed content and 70-95°C barrel temperature. Extrusion-cooking

Fig. 4 - superimposed contours for the product responses affect by water feed content and barrel temperature. *BD: bulk density and BS: breaking strength.

Overlay Plot
was carried out for confirmation under the optimum process conditions and the responses were recorded (mean of five measurements). In particular, the following operating conditions were chosen: 72 °C barrel temperature and 16% water feed content. The values predicted by the software were ≤ 0.8 g/mL for bulk density and ≤ 20 N/mm² for breaking strength. The data obtained by extrusion experiments carried out at the same operating conditions were respectively 0.78 g/mL and 7.38 N/mm². The veracity of values of operating conditions were respectively 0.78 g/mL and 7.38 N/mm². The veracity of values of operating conditions were respectively 0.78 g/mL and 7.38 N/mm². The veracity of values of operating conditions were respectively 0.78 g/mL and 7.38 N/mm². The veracity of values of operating conditions were respectively 0.78 g/mL and 7.38 N/mm².

Therefore, the developed model was suitable in representing the optimum operating conditions for this particular application.

4. CONCLUSIONS

The obtained results showed that the barrel temperature was the variable that has mainly affected the formation of starch-lipid complexes and structure of the extrudates. In particular, the worst characteristics of extrudates (hardness and bulk density of extruded products) were obtained at the highest temperature that corresponds to the maximum formation of starch-lipid complex. Moreover, the model was found to be statistically valid and demonstrated adequate information regarding the behaviour of the responses upon variations of the process variables. Optimum process conditions and the corresponding predicted responses could be obtained with the help of the models. The predicted responses at the optimum conditions were not significantly different from the experimental values. According to the optimum conditions given for the variables, the process could be referred to standardization of industrial production of snack food made up of rice and pistachio nut flours with high qualitative characteristics.

5. REFERENCES


