THE IMPACT OF THE MANUFACTURING PROCESS ON THE HARDNESS AND SENSORY PROPERTIES OF MILK CHOCOLATE

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The aim of this paper was to examine the impact of the manufacturing process on the textural characteristics and sensory properties of milk chocolate. The research was conducted on the samples of chocolate produced in a ball mill during 30, 60 and 90 minutes of refining, each of them being pre-crystallized at 26, 28 and 30°C. A chocolate mass of identical ingredient composition was also produced using a standard manufacturing process at the same pre-crystallization temperatures. Chocolate hardness was examined using a piece of equipment called Texture Analyser, measuring the stress intensity which leads to chocolate crushing. Sensory analysis was performed using the point scoring method. The new manufacturing process, i.e. the manufacturing of chocolate in a ball mill improves sensory properties and hardness of milk chocolate.

KEY WORDS: milk chocolate, ball mill, hardness, sensory evaluation

INTRODUCTION

Chocolate mass can be produced in two ways: using a standard or traditional manufacturing process (1, 2) and using the unconventional one in a ball mill (3,4). The traditional process includes: mixing of raw materials, refining in a five-roller mill, conching, tempering, moulding and final crystallization, and it has been well known and used in unaltered form for the last 150 years (5). Present-day trend of manufacturing rationalization has resulted in introducing a new method which combines the first two phases of the traditional process: conching and refining. A ball mill consists of a vertical or a horizontal double-wall cylinder with hot water flowing between them (6). In the central part of the cylinder there is a paddle mixer that operates at the speed of 50-70 rpm. The balls, i.e. the refining medium fill up to 60-80% of the cylinder. The balls are usually made of stainless steel or any other material used in the food industry. The mill must be provided with the mass recirculation system. The chocolate mass with constant recirculation goes through a thick layer of rotating balls, in the process of which the particles are constantly

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refined and subjected to shear force and friction. The speed of mass recirculation is 3-6 kg/min. The time of refining and processing of one loading is 90-120 minutes. The advantages of manufacturing chocolate in a ball mill in comparison with the conventional manufacturing process are reduced costs of maintenance, workforce, production and initial costs.

Ball mills are constantly improved, so that the chocolate could have suitable sensory and rheological properties. Mazzetti company has created a ball mill with a container for storing chocolate mass that is equipped with a thin layer evaporator, which in addition to refining also enables the correction of chocolate mass viscosity and taste (7). Duyvis Wiener company has created “Taste Changer” for eliminating unwanted moisture and volatile acids. According to this procedure, heated dry air is added to the chocolate mass under pressure and controlled flow rate. Dry air takes over moisture and volatile acids, while the increased temperature causes volume expansion of cocoa butter which in this way coats solid particles more easily and contributes to the improvement of the chocolate mass rheological properties (8, 9).

After being manufactured in a ball mill, the chocolate mass is tempered, moulded and hardened through final crystallization. Tempering or pre-crystallization is favourable for the creation of crystallization centres within a stable crystal form, which results in a product having good physical and sensory properties (10).

Chocolate hardness is one of the most important factors in defining physical properties of this product and it is determined by the measuring intensity of the force required for chocolate crushing. Chocolate hardness depends on the refinement of solid particles as well as on their distribution by size (11). Chocolate sensory properties are also an unavoidable factor in determining the consumers’ acceptance of this product (12).

EXPERIMENTAL

Material

The raw materials used for chocolate production included: cocoa butter (Theobroma, Amsterdam), cocoa liquor (Cargill), medium-grain sugar (Crvenka AD, Serbia), whole milk powder (total fat 25%, proteins 28% and carbohydrates 37%) - Imlek, Serbia, skimmed milk powder (total fat 1%, proteins 35% and carbohydrates 51%) - Imlek, Serbia, hazelnut paste (total fat 25%, proteins 28% and carbohydrates 37%) - Arslanturk, Turkey, Ethylvanilin (FCC, Norway), soya lecithin with a minimum insoluble content of 65% in acetone (Soyaprotein AD, Serbia), polyglycerol polyricinoleate or PGPR (Danisco, Malaysia) and SM-chocolate mass produced by standard manufacturing process (Jaffa, Srbija).

Methods

Production of chocolate mass in a ball mill. The chocolate was manufactured in a laboratory ball mill with a homogenizer (capacity 5 kg), of a domestic manufacturer. All raw materials were added to the homogenizer except for the 10% of cacao butter. The mixing time was 20 minutes. After mixing, the mass was transferred into the ball mill. The
milling time was 90 minutes and the remaining quantity of the cacao butter was added in the 80th minute. The diameter of the balls in the mill is 9.1 mm and the rotation speed of mixer is 50 rpm. The ball mill is equipped with the mass-circulation system, with a speed of 10 kg/hour. The internal diameter of the ball mill is 0.250 m, and the height is 0.31 m. The volume of the space provided for balls and 5 kg of chocolate mass is 0.0152 m³.

**Chemical analyses.** The basic chemical composition is determined using standard AOACC methods (13), as shown in Table 1.

**Table 1.** Chemical methods for basic chemical composition of milk chocolate mass determination

<table>
<thead>
<tr>
<th>Quality factor</th>
<th>Method/Principle</th>
</tr>
</thead>
<tbody>
<tr>
<td>Moisture [%]</td>
<td>Thermogravimetric</td>
</tr>
<tr>
<td>Total fat [% d.m.]</td>
<td>Determination of the petrol ether extract</td>
</tr>
<tr>
<td>Proteins [% d.m.]</td>
<td>Kjeldahl method</td>
</tr>
<tr>
<td>Carbohydrates [% d.m.]</td>
<td>Polarimetric</td>
</tr>
<tr>
<td>Cocoa solids [% d.m.]</td>
<td>Spectrophotometric</td>
</tr>
<tr>
<td>No fat cocoa components [% d.m.]</td>
<td>Spectrophotometric</td>
</tr>
<tr>
<td>Saccharose [% d.m.]</td>
<td>Polarimetric</td>
</tr>
<tr>
<td>Lactose [% d.m.]</td>
<td>Iodine metric titration</td>
</tr>
<tr>
<td>Energy value [kcal]</td>
<td>Calculation</td>
</tr>
<tr>
<td>Energy value [kJ]</td>
<td>Calculation</td>
</tr>
</tbody>
</table>

**Production of the chocolate mass using standard manufacturing process.** The chocolate mass manufactured in a standard way is mixed in a melanger for about 20 minutes (with one half amount of the cocoa butter and emulsifier) and then refined in a five-roller mill and conched. The remaining amount of cocoa butter is added while conching. Conching lasted for 12 hours, after which the remaining amount of emulsifier is added and the process is continued for another 6 hours.

**Pre-crystallization of the chocolate mass.** Pre-crystallization of the chocolate mass is performed in the laboratory precrystallizer – a modified Brabender farinograph (14). The process of pre-crystallization is controlled indirectly by the changes of the mass resistance on the occasion of mixing, which is registered on a force/time diagram – the thermoreogram. The following pre-crystallization temperatures were applied: 26°C, 28°C and 30°C for both chocolate masses.

**Symbols used in the work.** Symbols of the chocolate masses used in work are listed in Table 2.

**Determination of chocolate hardness.** The determination of the chocolate textural properties was performed using Texture Analyser following the original method 3-Point Bending Rig HDP/3PB. Working conditions were: measuring cell 5 kg; temperature 20°C; speed of cylindrical sonde before the analysis: 1.0 mm/s; speed of cylindrical sonde during the analysis: 3.0 mm/s; speed of cylindrical sonde after the analysis: 10.0 mm/s; distance: 40 mm; texture measuring was performed on 3 repeated occasions, after seven days of stabilization of the manufactured chocolate. The subject of the measurement was the intensity of force used to crush the chocolate.
Table 2. Symbols of the chocolate masses

<table>
<thead>
<tr>
<th>Symbol of the chocolate mass</th>
<th>Refining time (min)</th>
<th>Pre-crystallization temperature (ºC)</th>
</tr>
</thead>
<tbody>
<tr>
<td>R1-90-26</td>
<td>90</td>
<td>26</td>
</tr>
<tr>
<td>R1-90-28</td>
<td>90</td>
<td>28</td>
</tr>
<tr>
<td>R1-90-30</td>
<td>90</td>
<td>30</td>
</tr>
<tr>
<td>R1-60-26</td>
<td>60</td>
<td>26</td>
</tr>
<tr>
<td>R1-60-28</td>
<td>60</td>
<td>28</td>
</tr>
<tr>
<td>R1-60-30</td>
<td>60</td>
<td>30</td>
</tr>
<tr>
<td>R1-30-26</td>
<td>30</td>
<td>26</td>
</tr>
<tr>
<td>R1-30-28</td>
<td>30</td>
<td>28</td>
</tr>
<tr>
<td>R1-30-30</td>
<td>30</td>
<td>30</td>
</tr>
<tr>
<td>SM -26 in five-roller mill</td>
<td></td>
<td>26</td>
</tr>
<tr>
<td>SM -28 in five-roller mill</td>
<td></td>
<td>28</td>
</tr>
<tr>
<td>SM -30 in five-roller mill</td>
<td></td>
<td>30</td>
</tr>
</tbody>
</table>

**Sensory analysis.** A committee of 6 members scored from 1 to 5 the following quality parameters: appearance, structure, chewing, taste and smell. The obtained scores of these parameters were multiplied by a defined coefficient of importance (14), and the category of quality was defined on the basis of the total number of points. The chocolate samples were analysed seven days after their stabilization.

**Statistical analyses.** The results of measuring hardness and the total number of pondered points were processed by statistical testing for significant difference between two means using t-test at the significance threshold of 95%, \( \alpha = 0.05 \) (program packages Statistica 8.0 and Origin 6.1). The influence of independent variables (x and y) on the above dependent variables (z) was mathematically defined by means of regression analysis of experimental values. The response function z was defined by the regression equation (mathematical model) of the following form:

\[
z = b_0 + b_1x + b_2y + b_{11}x^2 + b_{12}xy + b_{22}y^2,
\]

in which: \( b_0, b_1, b_2, b_{11}, b_{12} \) and \( b_{22} \) are regression coefficients; \( x \) is pre-crystallization temperature (\( t_p \)); \( y \) is refining time (\( \tau \)); \( z \) is response function of characteristic parameter value: the chocolate hardness, the total number of points for sensory quality). The regression coefficients \( b_1 \) and \( b_2 \) indicate linear effect of the independent variables \( x \) and \( y \) on the dependent variable \( z \), \( b_{11} \) and \( b_{22} \) indicate square effect, while \( b_{12} \) indicates linear interaction of independent variables.

On the basis of obtained experimental - real (\( z_e \)) and theoretical - expected values (\( z_t \)) the following statistical parameters were calculated: the standard error of the regression \( \sigma \), \( p \) and t-values, the coefficient of determination and analysis of variance for the selected regression expression.

The standard error of the regression is defined by the following relation:

\[
\sigma = \sqrt{\frac{\sum (z_e - z_t)^2}{n-2}}
\]

The calculation of the coefficient of determination \( (r^2) \) solved to determine the discrepancy between the experimental and the theoretical values.
Statistical processing of the results included only the examination of the chocolate mass manufactured in the ball mill.

RESULTS AND DISCUSSION

Chemical analysis

Both chocolate masses had identical chemical composition, as they were made from the same raw materials, but in different manufacturing processes, which is shown in Table 3.

Table 3. Chemical composition of the milk chocolate mass

<table>
<thead>
<tr>
<th>Quality factor</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Moisture [% d.m.]</td>
<td>1.10</td>
</tr>
<tr>
<td>Total fat [% d.m.]</td>
<td>32.41</td>
</tr>
<tr>
<td>Proteins [% d.m.]</td>
<td>8.76</td>
</tr>
<tr>
<td>Carbohydrates [% d.m.]</td>
<td>52.98</td>
</tr>
<tr>
<td>Cocoa components [% d.m.]</td>
<td>30.14</td>
</tr>
<tr>
<td>No fat cocoa components [% d.m.]</td>
<td>4.74</td>
</tr>
<tr>
<td>Saccharose [% d.m.]</td>
<td>42.67</td>
</tr>
<tr>
<td>Lactose [% d.m.]</td>
<td>10.31</td>
</tr>
<tr>
<td>Emulsifiers [% d.m.]</td>
<td>0.50</td>
</tr>
<tr>
<td>Energy value [kcal]</td>
<td>538.64</td>
</tr>
<tr>
<td>Energy value [kJ]</td>
<td>2251.53</td>
</tr>
</tbody>
</table>

Hardness of the chocolate mass

The graph showing experimental results of measuring the hardness of chocolate R1 and SM using Texture Analyser following the original method 3- Point Bending Rig HDP/3PB is presented in Fig. 1.

![Graph showing experimental results of measuring the hardness of chocolate R1 and SM using Texture Analyser following the original method.](image)

Figure 1. Comparative illustration of the hardness of the chocolate mass manufactured in the ball mill (R1) and the chocolate mass made by standard manufacturing process (SM)
In the chocolate mass produced by standard manufacturing process it was noted that an increase in the pre-crystallization temperature leads to a decrease in the force required for chocolate crushing. An increase in the pre-crystallization temperature in the chocolate mass produced in the ball mill, irrespective of the refining time, leads to an increase in the force required for chocolate crushing (increased hardness values), which is most clearly seen in the most of the homogeneous systems – the one in which the chocolate mass was refined during 90 minutes. The increase in hardness as a result of the increase in pre-crystallization temperature was also confirmed by the statistical data processing.

The results of statistical data processing are shown in the 3D and contour diagram in Fig. 2. The increase in the pre-crystallization temperature had a significantly greater influence on the increase in the hardness of the chocolate mass R1, than on the refining time. By testing statistical significance of certain parameters in the regression equation it has been noted that the regression coefficients b1, b0 and b11 indicate certain changes of the dependent variable, i.e. hardness, by changing the independent variable x, i.e. the pre-crystallization temperature.

\[
Z_1 = 3518.727 - 261.002 \tau_p + 1.213 \tau + 4.942 \tau_p^2 - 0.118 \tau_p \tau + 0.017 \tau^2
\]

**Figure 2.** The impact of the refining time and pre-crystallization temperature on the hardness of the chocolate mass manufactured in the ball mill R1:

a) 3D diagram, b) contour diagram

The calculated value of standard error of the regression (σ = 13.864) confirms that the selected mathematical model gives a bit larger dispersion of experimental values, while the value of coefficient of hardness determination (r² = 0.894) indicates that physical properties of chocolate R1 are determined by variations of independent variables by 89.4%.

The analysis of variance in regression equation confirms that at the 95% significance level (α = 0.05) and by applying the selected regression equation, it is possible to predict the behaviour of the chocolate hardness under varying refining time and pre-crystallization temperature (calculated F = 12.874 > tabular F_{0.05;6;3} = 8.94).
Sensory analysis of chocolate

The graph showing comparative experimental results of the milk chocolate mass produced in the ball mill (R1) and of that produced by standard manufacturing process (SM) is shown in Fig. 3.

![Figure 3](image)

**Figure 3.** Comparative illustration of sensory marks of the chocolate mass manufactured in the ball mill (R1) and the chocolate mass manufactured by traditional manufacturing process (SM)

The chocolate mass produced by standard manufacturing process, pre-crystallized at 30°C provides a chocolate of suitable, i.e. very good sensory quality (highly glossy surface, tiny grainy structure, shelly fracture and suitable taste and smell). Its external properties are significantly better in comparison with the chocolates pre-crystallized at 26°C and 28°C. These very good sensory properties result from the optimal viscose properties and suitable pre-crystallization of cocoa butter. The chocolate SM-28 had the worst fracture and the lowest total of pondered points.

The chocolate mass produced in the ball mill refined during 90 minutes and pre-crystallized at higher temperatures provides a chocolate of suitable, i.e. excellent sensory quality (highly glossy surface, tiny grainy structure, shelly fracture, and suitable taste and smell). Such good sensory properties result from the optimal viscose properties and suitable crystallization of cocoa butter. The chocolate R1-60 had a very good sensory quality as well as better marks for external properties, structure and chewing in comparison with the chocolate R1-90. Refining time of 30 minutes provides a chocolate of good sensory quality only, i.e. the obtained chocolate form had weaker deformation, partially damaged surface, coarse grain fracture, uneven structure and evident properties of slower melting in the mouth.

The graph showing statistically processed data on dependence of the chocolate mass R1 sensory properties on the pre-crystallization temperature and refining time is seen in the 3D and contour diagram in Fig. 4. In the regression equation which defines the dependence of the total of sensory analysis pondered points on the refining time and pre-crystallization temperature, the coefficient of square dependence of the refining time and the coefficient of linear interaction between the refining time and the pre-crystallization temperature have the greatest influence.
$Z_2 = 8.476667 - 0.365833 t_p - 0.024056 \tau + 0.006250 t_p^2 + 0.00100 t_p \tau + 0.000117 \tau^2$

**Figure 4.** Impact of the refining time and precrystallization temperature on the sensory scores of the chocolate mass manufactured in the ball mill R1:

a) 3D diagram b) contour diagram

The value of standard error of the regression shows a slight dispersion between the experimental values and the theoretical curve ($\sigma = 0.095$). The selected regression equation is representative, as the variations of independent variables are significantly responsible for the sensory quality of the chocolate mass ($r^2 = 0.9824$). The variance analysis confirms that the regression equation is statistically significant as a whole, as it has been calculated that $F > F_{0.05;63} = 8.94$.

**CONCLUSIONS**

Milk chocolate produced in a ball mill is harder than the chocolate produced by standard manufacturing process, irrespective of the applied pre-crystallization temperature. The force required for crushing the chocolate produced in a ball mill is in average 2.25 times greater in comparison with the chocolate produced by standard manufacturing process. By introducing the new manufacturing process, i.e. the ball mill, the sensory properties of milk chocolate have been improved.

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**REFERENCES**


УТИЦАЈ ПОСТУПКА ПРОИЗВОДЊЕ НА ЧВРСТОЋУ И СЕНЗОРНЕ КАРАКТЕРИСТИКЕ МЛЕЧНЕ ЧОКОЛАДЕ

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Циљ овог рада био је да се испита утицај поступка производње на текстуру и сензорне карактеристике млечне чоколаде. Испитивани су узорци чоколаде која је произведена у кугличном млину утисњавањем 30, 60, 90 минута и свака чоколада је преткрсталисана на 26, 28 и 30°C. Такође је произведена чоколадна маса идентич-
ног сировинског састава стандардним поступком, која је преткрсталисана на исте температуре. Чврстоћа чоколада је испитана на уређају Texture Analyser, мерећи јачину силе која доводи до лома чоколаде. Нови поступак производње чоколаде у кугличном млину побољшава сензорне карактеристике и чврстоћу млечне чоколаде.

Кључне речи: млечна чоколада, куглични млин, чврстоћа, сензорна анализа

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