EXAMINATION OF THE AUTHENTICITY OF SELECTED DARK CHOCOLATES

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Abstract: The aim of the study was to apply cocoa husk determination to test the authenticity of selected dark chocolates available in the Polish and other European markets. The chocolates met the EU requirements and did not contain above 5% of cocoa shells. The energy value of the chocolates was significantly influenced by the fat content. When determining the color parameters of any chocolate, a large range in the a* color parameter was found, which was responsible for the intensity of color, from green to red. The a* value in one of the chocolates indicated the possibility of an adulteration with an undeclared coloring substance. Consequently, it was also found that the level of information provided by chocolate packaging was insufficient in most cases.

Keywords: chocolate, authenticity, labeling, stone cells, color.

1. INTRODUCTION

Among the confectionery products containing cocoa, chocolate is undoubtedly the most important one. It is a product consumed not only for its unique sensory properties, but because it also causes satisfaction and exceptional pleasure when consumed [Parker, Parker and Brotchie 2006; Merlino et al. 2021]. In nutrition, it is especially recommended to eat chocolate with a high content of cocoa mass, i.e., dark chocolates [Kerimi and Williamson 2015].

Directive 2000/36/EC defines chocolate as the product obtained from cocoa products and sugars which contains not less than 35% total dry cocoa solids, including not less than 18% cocoa butter and not less than 14% of dry non-fat cocoa solids. In some EU Member States, the addition of vegetable fats other than cocoa butter to chocolate products in a maximum amount of 5% is allowed. This directive was subsequently amended by Regulation (EU) No 1021/2013 of the European Parliament and of the Council of 9 October 2013. Annex II to the Directive 2000/36/EC lists the vegetable fats that have been authorized for use in the production of chocolate. The following fats are authorized for chocolate production:
Borneo tallow nut oil (Tengkawang) obtained from Shorea spp., palm oil from Elaeis guineensis and Elaeis olifera, sal fat from Shorea robusta, shea fat from Butyrospermum parkii, kokum gurgi fat from Garcinia indica and mango caramel from Mangifera indica. Coconut oil is also a vegetable fat that is often marketed as a cocoa butter substitute.

Experiments have shown that replacing 1.5–4.5% cocoa butter with coconut oil improves the sensory properties of chocolate in terms of a shiny appearance, flavor and overall palatability. Moreover, chocolate with a high content of coconut oil (4.5%) had the slowest rate of bloom formation during its storage [Halim et al. 2019]. Recent research [Khairy et al. 2018] shows that rambutan seed fat (Nephelium opossum L.) in an amount of up to 20% can be used with cocoa butter in chocolate production to improve and obtain the best quality aroma in the final products.

Depending on the type of chocolate, the addition of sugar is also used. The type of sugar and the form of its labeling are specified in Council Directive 2001/111/EC of 20 December 2001 relating to certain sugars intended for human consumption. This directive was also partially amended by Regulation (EU) No 1021/2013 of the European Parliament and of the Council of 9 October 2013, but the list of sugars that can be used in the production of chocolate has not changed. The directive lists the following sugars: semi-white sugar, white sugar or sugar, extra-white sugar, sugar solution, invert sugar solution, invert sugar syrup, glucose syrup, dried glucose syrup, dextrose or dextrose monohydrate, dextrose or dextrose anhydrous, and fructose.

The subject of this research is dark chocolate, which contain at least 60% cocoa mass. The aim of the study was an attempt to identify the adulteration in the composition of chocolate via the addition of cocoa shells. Two hypotheses were put forward in the study: (1) dark chocolates available on the Polish market do not contain cocoa shells above the amount permitted by the EU directive, (2) the color of chocolate is affected by the fat content.

2. FACTORS INFLUENCING THE CONSUMPTION OF CHOCOLATE

Chocolate and related products are consumed primarily because of their sensory properties and not their nutritional properties. For this reason, sensory properties are most often taken into account when researching the product and process development, and their optimization. However, a number of studies indicate the health aspect of chocolate consumption in the positive and negative sense [Veronese et al. 2019]. Chocolate and cocoa powder are a dietary source of flavonoids and other ingredients that can have a positive effect on the body. The beneficial antioxidant and anti-inflammatory effects of cocoa powder and chocolate flavonoids and minerals on the health, however, may be diminished by other components such as the presence of oxalates and methylxanthines, as well as the addition of fat and sugar,
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which may have adverse health effects [Lippi et al. 2008; Schroder, Vanhanen and Savage 2011; Witt, Śmiechowska and Kłobukowski 2016; Seem, Yuan and Tou 2019; Veronese et al. 2019; Dala-Paula et al. 2021].

The type and amount of ingredients (cocoa, sugar, fat, powdered milk, emulsifiers) and the chocolate production process, especially conching, are also important factors affecting the sensory properties of chocolate. The flavor of chocolate is influenced by volatile components and the behavior of the continuous fat phase, affecting the release of volatile substances into the space above the mouth and the perception of taste [Toker et al. 2020]. In the search for innovative methods influencing the shaping of the sensory quality of chocolate, sonication was applied. This method makes the chocolate silky and more fluid by reducing the size of the crystals during the crystallization process [Devos, Reyman and Sanchez-Cortes 2021].

The increasing demand for chocolate and chocolate products means that they may lose authenticity due to various types of adulteration. The most common irregularities already occur at the stage of ingredients used during production. Information on the origin of the cocoa beans and other information that should appear on the packaging of chocolate and chocolate products is often missing [Torres-Moreno et al. 2012].

3. ENSURING THE QUALITY AND AUTHENTICITY OF CHOCOLATE

In the second half of the 1990s, there was a dispute between chocolate producers over the fact that some of them added fats not derived from cocoa beans to chocolate [Cidell and Alberts 2006]. The result of this dispute was the introduction by the European Parliament of Directive 2000/36/EC, commonly known as the Chocolate Directive, which allows the addition of vegetable fat to chocolate in an amount not exceeding 5%. However, this directive has been criticized recently due to campaigns against the growing share of palm oil in food products [Lebailly, Ahouissou and Namé 2015].

The addition of fat not derived from cocoa beans is a common adulteration in the process of chocolate manufacturing. Foreign fats added to chocolate are mostly vegetable fats not listed in Annex II to Directive 2000/36/EC as cocoa butter equivalents (CBE), cocoa butter substitutes (CBS) and cocoa butter replacers (CBR) [Simoneau, Hannaert and Anklam 1999; Kadivar et al. 2016; Suwała 2020]. For economic reasons, some small and medium-sized enterprises in developing countries add pork lard to their chocolate and chocolate products. Fourier Transform Infrared Spectroscopy (FTIR) [Che Man et al. 2005] is used to detect the potential presence of lard in chocolate and chocolate products on the market. The practice of using animal fats as cocoa fat substitutes in the production of chocolate is unacceptable.
The adulteration of chocolate may concern not only fats, sugars or other additives used, but also the cocoa powder itself and the cocoa mass. High-quality chocolate starts with a good quality raw material, i.e., fine quality cocoa beans. Their quality is influenced by many factors: climatic, agrotechnical, social, economic, political, and these have all been considered in many studies [Śmiechowska and Kłobukowski 2019].

Earlier studies highlighted the possibility of adding cocoa shells to cocoa powder [Śmiechowska and Żeglarska 2020]. Are the cocoa powder and cocoa liquor used in the production of chocolate of the highest quality? The answer to this question is even more important as the content of good quality cocoa powder and cocoa liquor above 60–65% increases the nutritional value of chocolate [Zugrevu and Otelea 2019].

4. RESEARCH SECTION

4.1. Materials

The study material consisted of dark chocolate containing over 60% of the raw material obtained from cocoa beans from 8 different producers, purchased on the Polish and other European markets. Chocolates from industrial producers as well as conventional and organic craft factories were tested: Roshen Biedronka bitter 78%, E. Wedel bitter chocolate 64%, Lindt Excellence dark chocolate 70%, CoCoa classic organic bitter chocolate made of unroasted cocoa beans 70%, Madagascar Chocolate Tsara Menako 100% Infiniti, America Latina 100% cacao Alce nero biologico organic fairtrade, Belgian chocolate for Biedronka 72% bitter dark, and Wawel dark chocolate 70% sugar free. The chocolates were encoded and analyzed for the presence of cocoa shells, and their color was determined. The characteristics of the research material are presented in Table 1.

4.2. Methods

The determination of the content of stone cells in chocolate was carried out in accordance with the methodology of AOAC (Association of Analytical Chemists) 970.23 “Stone Cell and Group Count of Cacao Products” in our own modification. The principle of the determination is based on the mineralization of the chocolate sample with a mixture of nitric and acetic acid (Belluci’s reagent), which results in the decomposition of the organic matter, apart from the stone cells, which cannot be digested at such acid concentrations. After proper preparation, a drop of mineralizate should be placed in the field of view of a strobe microscope and the stone cells should be counted. The result is given as a percentage of the husk content.

The chocolate color as a parameter was determined in the international CIE L*a*b* D65 system using a Konica Minolta CR-400 colorimeter for a standard 10° observer and the light source D65 (CIE DS. 014-4.3/E:2007). The measurement was
made in an optical cell with an optical path length of 10 mm. The L* component, describing brightness, and a* and b* components, describing the proportion of green and red, as well as yellow and blue, respectively, were determined. The determination was made in two replications for each infusion.

**Table 1. Characteristics of the research material**

<table>
<thead>
<tr>
<th>Sample code</th>
<th>Declared composition of chocolate</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 RB</td>
<td>Cocoa mass, sugar, cocoa butter, cocoa powder, emulsifier: lecithin (from soy), vanilla flavor. Cocoa mass min. 78%.</td>
</tr>
<tr>
<td>2 EW</td>
<td>Cocoa mass, sugar, reduced fat cocoa, cocoa fat, emulsifiers (soy lecithin and E476), flavor. Cocoa mass min. 64%.</td>
</tr>
<tr>
<td>3 LE</td>
<td>Cocoa liquor, sugar, cocoa butter, vanilla.</td>
</tr>
<tr>
<td>4 CC</td>
<td>Raw cocoa beans, coconut sugar, cocoa butter. Cocoa mass content min. 70%. Product may contain nuts.</td>
</tr>
<tr>
<td>5 MC</td>
<td>Cocoa beans from Madagascar, cane sugar, cocoa butter, emulsifier: sunflower lecithin. Cocoa content min. 80%.</td>
</tr>
<tr>
<td>6 AL</td>
<td>Cocoa mass*, cane sugar* (17%), cocoa butter*, shredded roasted cocoa beans* (10%), vanilla*. Cocoa mass min. 80%. (* certified organic ingredient).</td>
</tr>
<tr>
<td>7 BB</td>
<td>Cocoa mass, sugar, cocoa powder, emulsifier: lecithin (from soy). Dark chocolate: cocoa mass min. 70%.</td>
</tr>
<tr>
<td>8 WC</td>
<td>Cocoa mass (origin: Africa), sweetener: maltitol, cocoa butter, reduced fat cocoa, emulsifier: lecithin (from soy). Cocoa mass min. 70%.</td>
</tr>
</tbody>
</table>

Source: own study based on information contained on unit packages.

**5. RESULTS AND DISCUSSION**

The analysis of the composition of the chocolates selected for the study indicated on the packaging of the products shows that all products contain over 60% of cocoa mass. A consumer unfamiliar with interpreting such information may be convinced that chocolate contains more than 60% cocoa powder and/or cocoa liquor (Table 1). However, this is misleading as this information applies to all ingredients obtained from cocoa beans, including cocoa butter, which is clearly indicated by the nutritional value of the chocolate (Table 2). Producers of these chocolates declared different nutritional values and the contents of fat, total carbohydrates, simple sugars, protein and salt (Table 2).
The energetic value of the tested chocolates ranged widely, from 502 to 650 kcal, and was strictly dependent on the fat content in the chocolates, which was within the range 32–60 g (Table 2). The Pearson correlation coefficient linking these two values was as high as 0.927 (p = 0.001), which emphasized the dependence of the nutritional value of chocolate primarily on the fat content. At the same time, the latter statistically significantly reduced the carbohydrate content in the product (p = 0.005).

Table 2. Nutritional value of the studied chocolates (per 100 g)

<table>
<thead>
<tr>
<th>Chocolate</th>
<th>Nutritional value [kcal]</th>
<th>Fat [g]</th>
<th>Carbohydrates [g]</th>
<th>Sugars [g]</th>
<th>Protein [g]</th>
<th>Salt [g]</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 RB</td>
<td>560.00</td>
<td>43.00</td>
<td>25.00</td>
<td>21.00</td>
<td>10.00</td>
<td>0.01</td>
</tr>
<tr>
<td>2 EW</td>
<td>502.00</td>
<td>32.00</td>
<td>39.00</td>
<td>35.00</td>
<td>8.40</td>
<td>0.02</td>
</tr>
<tr>
<td>3 LE</td>
<td>566.00</td>
<td>41.00</td>
<td>34.00</td>
<td>29.00</td>
<td>9.50</td>
<td>0.10</td>
</tr>
<tr>
<td>4 CC</td>
<td>590.00</td>
<td>45.00</td>
<td>33.00</td>
<td>29.00</td>
<td>8.00</td>
<td>0.20</td>
</tr>
<tr>
<td>5 MC</td>
<td>635.74</td>
<td>51.00</td>
<td>30.54</td>
<td>0.00</td>
<td>13.60</td>
<td>0.02</td>
</tr>
<tr>
<td>6 AL</td>
<td>650.00</td>
<td>60.00</td>
<td>8.00</td>
<td>0.00</td>
<td>12.00</td>
<td>0.02</td>
</tr>
<tr>
<td>7 BB</td>
<td>569.00</td>
<td>41.00</td>
<td>36.00</td>
<td>25.00</td>
<td>9.40</td>
<td>&lt;0.10</td>
</tr>
<tr>
<td>8 WC</td>
<td>503.00</td>
<td>39.00</td>
<td>34.00</td>
<td>0.80</td>
<td>9.70</td>
<td>0.02</td>
</tr>
</tbody>
</table>

Source: own study based on information contained on unit packages.

The total carbohydrate content in the tested chocolates also remained in the large range of 8–39 g and was influenced mostly by added sugars of 0–35 g (Table 2). The prospective consumer, guided by the information on the label about 80% of ingredients derived from cocoa beans, may be surprised by the fact that the chocolate is also a product of high energy value due to its high fat content or added cane sugar or sucrose.

The assessment of the information on the chocolate packaging varies. Many chocolate manufacturers provide information about the origin of the raw material, treating it as a marketing element. The packaging also frequently carries fair trade or organic certificates. However, this does not apply to all producers, possibly due to the fact that chocolate can be made from a mixture of seeds of different origins. Criollo, Forastero, Trinitario and Nacional are the main original botanical varieties of cocoa. It is well known that the Criollo variety produces mild, earthy, floral, nutty and tea flavors. This variety is grown in the Caribbean, Ecuador, and Papua New
Guinea, and is most commonly used to make dark chocolate. Forastero is a variety whose seeds are darker and give an intense cocoa flavor, and in comparison to Criollo it produces less delicate chocolate notes. Trinitario is a hybrid of Criollo and Forastero, offering a strong original chocolate flavor and wine-like character. Nacional creates a full cocoa flavor with floral and spicy flavors [Toker et al. 2020].

5.1. Content of stone cells (cocoa husk) in the studied chocolates

The most important ingredients in chocolate are cocoa mass, cocoa butter and cocoa powder. Among these ingredients, the most valuable from the point of view of the nutritional and health value of chocolate are cocoa mass and cocoa powder. Earlier publications on the quality of cocoa powder highlighted the problems associated with cocoa cultivation [Smiechowska and Kłobukowski 2019]. The factors influencing the supply of cocoa beans include climatic, social, economic and political factors. Other factors influencing cocoa production and its quality are soil factors, agronomic treatment, protection against pests and fungal diseases.

This situation favors the adulteration of cocoa beans and cocoa products, such as cocoa mass, cocoa powder and chocolate. The lack of authenticity of these products reduces the confidence of cocoa processors and consumers of chocolate products and drives the search for methods to detect adulteration. The simplest and easiest way to adulterate cocoa powder is by adding ground shells of the cocoa fruit (often called a pod) or inaccurate hulling of cocoa beans after roasting, which affects the organoleptic quality of cocoa powder and chocolate products.

The results of the percentage of stone cells in terms of the cocoa shell content in the studied chocolates are presented in Table 3. The results are the average of two separately performed determinations.

Table 3. Average husk content in the studied chocolates [%]

<table>
<thead>
<tr>
<th>Chocolate</th>
<th>1 RB</th>
<th>2 EW</th>
<th>3 LE</th>
<th>4 CC</th>
<th>5 MC</th>
<th>6 AL</th>
<th>7 BB</th>
<th>8 WC</th>
</tr>
</thead>
<tbody>
<tr>
<td>Husk content [%]</td>
<td>1.24</td>
<td>1.25</td>
<td>0.27</td>
<td>0.10</td>
<td>0.25</td>
<td>0.23</td>
<td>1.46</td>
<td>0.48</td>
</tr>
</tbody>
</table>

Source: own study.

According to the results, the 4 CC chocolate was characterized by the lowest amount of stone cells, followed by 6 AL and 5 MC. 1 RB and 7 BB chocolates had the highest stone cell content. This may result from incorrect cleaning of cocoa beans before starting the production of chocolate. The presence of cocoa shells in products made from cocoa beans, including chocolate, is undesirable as it adversely affects the process and quality of the end product and therefore the shell content of cocoa products is an important quality parameter.

In this context, the European Union Cocoa and Chocolate Directive sets the maximum level of shell, including germ, in cocoa nibs of up to 5% in relation to the content of fat-free cocoa powder [EEC, 2000].
Levels higher than this value should be considered a factor that diminishes the value of the cocoa products. The husk content of the studied chocolate samples met the requirements of the EEC 2000 directive.

5.2. Determination of the color of chocolate in the CIE L*a*b* system

The color of chocolate is formed already at the stage of cocoa cultivation, because the color of cocoa mass and cocoa powder depends partly on the climatic and soil conditions of cultivation. Other processes influencing the color of cocoa beans and cocoa products are the method and fermentation, the alkalization process and the roasting conditions of the cocoa beans [Krysiak 2006; Le et al., 2012; Saltini, Akkerman and Frosch 2013]. The results of the color determination of the studied chocolates in the CIE L*a*b* system are presented in Table 4.

Table 4. Average value of the color parameters of the studied chocolates

<table>
<thead>
<tr>
<th>Chocolate</th>
<th>L*</th>
<th>a*</th>
<th>b*</th>
<th>Fat [g]</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 I RB</td>
<td>29.83</td>
<td>3.98</td>
<td>2.64</td>
<td>43.00</td>
</tr>
<tr>
<td>2 EW</td>
<td>29.47</td>
<td>4.16</td>
<td>2.74</td>
<td>32.00</td>
</tr>
<tr>
<td>3 LE</td>
<td>30.06</td>
<td>4.43</td>
<td>2.50</td>
<td>41.00</td>
</tr>
<tr>
<td>4 CC</td>
<td>30.63</td>
<td>7.00</td>
<td>5.34</td>
<td>45.00</td>
</tr>
<tr>
<td>5 MC</td>
<td>31.39</td>
<td>5.05</td>
<td>4.15</td>
<td>51.00</td>
</tr>
<tr>
<td>6 AL.</td>
<td>29.10</td>
<td>3.94</td>
<td>3.22</td>
<td>60.00</td>
</tr>
<tr>
<td>7 BB</td>
<td>28.77</td>
<td>3.65</td>
<td>3.51</td>
<td>41.00</td>
</tr>
<tr>
<td>8 WC</td>
<td>29.11</td>
<td>3.15</td>
<td>3.24</td>
<td>39.00</td>
</tr>
</tbody>
</table>

Source: own study.

In the studied chocolates, the value of the L* parameter, determining the brightness, ranged from 28.77 to 31.39. The lowest value of this parameter was found for 7 BB chocolate, and the highest for 5 MC chocolate. The value of the a* parameter of the trichromatic component, describing the color change from green to red, remained in the range of 3.15–7.00. The value of the b* parameter, reflecting the color changes from blue to yellow, remained in the range of 2.64–5.34.

Pearson’s correlation coefficients, linking fat content with the L*a*b* parameters, proved to be statistically insignificant (p = 0.632, 0.695, 0.483, respectively), even though the value of the L* parameter often depends on the fat content in chocolate. It was pointed out [Kłobukowski and Śmiechowska 2016] that the fat content in cocoa powder affects the color and properties of cocoa drinks.
Fat not only affects the palatability of chocolate products, but is often also a factor influencing their color.

The color of chocolate is influenced not only by the fat content but also by the presence of certain coloring additives. Such substances include red beet products and caramel. A recent study [Baycar et al. 2022] has shown that the addition of dried red beet juice affects the color of chocolate and increases its nutritional value. According to these researchers, such chocolate can be considered a functional product. However, one should be aware that the undeclared addition of red beetroot powder may be considered an adulteration of the composition of the chocolate. It was found [Baycar et al. 2022] that the addition of red beet powder increased the a* component, which is responsible for the green to red color scale in the CIE L*a*b* system.

The value of this component in the research conducted by these authors was in the range 2.47–12.80, depending on the amount of dried red beet product added. It can be assumed that in our research, the 4 CC chocolate, for which the component a* was 7.00, may possibly indicate the presence of coloring additives.

The present studies should be continued to exclude or confirm the addition of such coloring substances.

6. CONCLUSIONS

The research has shown that the information value on chocolate packaging is insufficient. The information about the geographic origin of cocoa beans and the variety of beans used in the production of chocolate is lacking. The packaging contains information with a chocolate mass of more than 60, 70 or 80%, which is misleading, as not all consumers know that the term “chocolate mass” applies simultaneously to cocoa mass, cocoa powder and cocoa butter. According to the manufacturers’ declarations, the content of cocoa butter, including other fats, can sometimes be very high.

The hypothesis that the chocolates on the Polish market are not adulterated with excessive addition of cocoa shells was confirmed. The experiments showed that all the chocolates met the requirements of Directive 2000/36/EC of the European Parliament and did not contain more than 5% of cocoa shells.

A result of the research was that it was found that the color of the studied chocolates does not depend on the fat content. There is a high probability that the a* parameter in the CIE L*a*b* color system may be an indication of the quality control applied to the chocolate, as its value may be influenced by the addition of colorants. Therefore, we believe that these studies should be continued to confirm this hypothesis.
REFERENCES


