






Article

Physical Properties of Chocolates Enriched with Untreated Cocoa Bean Shells and Cocoa Bean Shells Treated with High-Voltage Electrical Discharge

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Abstract: Recently, the enrichment of chocolate has become a very interesting topic, along with the management of food industry by-products, such as cocoa shells. Cocoa shells could be a great raw material for the cocoa industry, both for economical reasons (maximized utilization of cocoa beans) and for their functional properties (increased fiber content). In this research, we used untreated and high-voltage electrical discharge (HVED)-treated cocoa shells in the production of chocolate. Different proportions of cocoa mass were replaced with cocoa shells to produce dark and milk chocolates in a ball mill. Additionally, dark chocolate with 15% and milk chocolate with 5% of shells were chosen for further research and to study the alteration of the composition. The rheology, particle size distribution, hardness, and color were determined for all the prepared samples. Treated cocoa shells provided chocolates with inferior physical properties compared to chocolates with untreated shells. Therefore, untreated cocoa shells were selected for further analysis. The addition of both treated and untreated cocoa shells resulted in softening and darkening of samples, which could have a positive effect for consumers. On the other hand, the particle size distribution and rheology were negatively affected. Further research is needed to find a solution for these problems.

Keywords: milk chocolate; dark chocolate; cocoa shells; high-voltage electrical discharge (HVED); particle size distribution; rheology; hardness; color

1. Introduction

Chocolate is one of the most favored confectionery products in the world. Although it is recognized as a good source of polyphenols, it is rich in sugar and fat and has a high caloric value, which are often pointed out as drawbacks of its consumption. Therefore, a large number of recent scientific studies have dealt with either the enrichment of chocolate with polyphenols and dietary fibers or cutting down the sugar and fat contents [1]. However, any intervention into the composition of chocolate interferes with its unique taste, texture, and flavor, which are the product of the combination of cocoa butter, sugar, and cocoa mass [2]. A possible solution to address this problem could be cocoa shells (CBSs), a by-product of the chocolate industry that is produced in large amounts every year. Rojo-Poveda et al. [3] state: "Taking into account the weight percentage of CBSs and the aforementioned cocoa production data, this would mean that more than 700 thousand tons of CBS waste is produced worldwide, from which more than 250 thousand tons is

only produced in Europe.” Many scientists are trying to find an application for cocoa shells in order to reach the sustainable production of nutritious food [4].

Large contents of dietary fibers (insoluble and soluble), proteins, polyphenols, and methylxanthines are the reason why cocoa shells are now regarded as a valuable raw material [5]. The dietary fibers in cocoa shells are mainly composed of cellulose, polysaccharides, and lignin [6], and the predominant polyphenols are flavan-3-ols that migrate to the shell from beans during processing [7]. Bernaert and Raysscher [8] produced cocoa shell powder and proved that it can be used as a cocoa powder replacer. They separated cocoa shells from cocoa beans, after which the cocoa shells were ground, washed in a buffer solution, and dried. After this process, the cocoa shells were alkalized and ground again to a particle size of less than 75 μm . They can also be used to improve the color of foods and inhibit fat bloom in cocoa-based products. The production of chocolate muffins, bread, cocoa beverages, cookies, pork sausages, extruded snack products, etc. are some examples of the successful application of cocoa shells in the food industry [4].

However, there are some negative aspects linked to cocoa shells, which must be resolved before their use in the production of food. Some of these are mycotoxins, heavy metals, polycyclic aromatic hydrocarbons (PAHs), and the high load of microorganisms [4]. In our previous paper [4], high-voltage electrical discharge (HVED) was presented as a method for resolving some of the problems (e.g., hydroxymethylfurfural and acrylamide). HVED is a non-thermal technology that generates electrical discharges directly in the water in which a material is dispersed; it is already used in the reduction of microbial load, showing great potential for use in the treatment of food industry by-products [4]. During HVED treatment, different radicals and UV radiation are generated, causing microbial inactivation and destruction of the membranes of microorganisms [9]. In addition, radicals can react with heavy metals, converting them into insoluble forms that are easily removed [10].

According to the authors’ knowledge, cocoa shells have not been used in chocolate production yet due to their coarse structure and the above-mentioned contaminants. The aim of this study was to evaluate the effect of cocoa shell addition on the physical properties of chocolate. Cocoa shells were treated with HVED and used as a replacement for a part of the cocoa mass. Untreated cocoa shells were also used in the same manner in order to reveal if the HVED treatment influences their behavior in chocolate. The rheological properties, hardness, color, and particle size distribution were determined to reveal whether the cocoa shells have an effect on the quality attributes of chocolate. Once it was determined which cocoa shells had a better effect on the physical properties of chocolates, they were used to further examine the effects of different chocolate compositions on the physical properties of chocolates enriched with cocoa shells.

2. Materials and Methods

2.1. Cocoa Shell Preparation

The cocoa shells used in the production of chocolates were obtained after the roasting of cocoa beans (West Africa mix supplied by Huyser, Möller B.V., Edam, Holland) at 135 °C for 55 min.

The treated cocoa shells (for the purpose of this research) were roasted cocoa shells treated with high-voltage electrical discharge (HVED), as described in our previous paper [4]. Briefly, the electric field density during the treatment was 15 kV/cm, the concentration was 3% (12 g in 400 g of water), the frequency was 40 Hz, and the treatment time was 15 min. HVED treatment was repeated until a sufficient number of cocoa shells were collected into one composite sample. After the treatment, the cocoa shells were frozen at $-80\text{ }^{\circ}\text{C}$ and freeze-dried (Alpha LCS Plus, Christ, Osterode am Harz, Germany). Dried cocoa shells were milled (M20, IKA, Staufen, Germany) and sieved through a series of sieves; the fraction below 71 μm was used in chocolate production. The prepared cocoa shells were stored at 4 °C until they were used in chocolate production.

Untreated cocoa shells (for the purpose of this research) were shells obtained after roasting, after which they were milled and sieved in the same manner as the treated shells.

2.2. Chocolate Production

Milk and dark chocolate samples were produced in a laboratory ball mill, and their compositions are shown in Tables 1 and 2. The speed of mixing was set at 60 rpm and the temperature was 55 °C (controlled by a circulating water bath). According to preliminary research, 3 kg of balls were used for milk chocolates and 2.5 kg for dark (stainless steel balls with a diameter of 9.525 mm). All the samples were set at 500 g. The control samples without added cocoa shells (M0 and D0) were mixed for 3 h. All other samples with added cocoa shells were prepared as follows. Cocoa shells and fats were mixed for 30 min before other components were added (sugar, cocoa liquor, and, if applicable, whole-fat milk powder and whey), and the chocolate mass was mixed for an additional 3 h. Cocoa liquor and butter were purchased from DGF (France), milk powder from Dukat d.d. (Croatia), and whey powder from Vindija d.d. (Croatia).

Table 1. Composition of the first group of chocolate samples.

Sample *	Cocoa Mass (%)	Cocoa Butter (%)	Milk Powder (%)	Untreated Cocoa Shells (%)	Treated Cocoa Shells (%)	Lecithin (%)
D0	36.00	21.57	0	0	0	0.40
DU5	31.00	21.57	0	5.00	0	0.40
DU10	26.00	21.57	0	10.00	0	0.40
DU15	21.00	21.57	0	15.00	0	0.40
DT5	31.00	21.57	0	0	5.00	0.40
DT10	26.00	21.57	0	0	10.00	0.40
DT15	21.00	21.57	0	0	15.00	0.40
M0	14.74	24.83	15.00	0	0	0.40
MU2.5	12.24	24.83	15.00	2.50	0	0.40
MU5	9.74	24.83	15.00	5.00	0	0.40
MT2.5	12.24	24.83	15.00	0	2.50	0.40
MT5	9.74	24.83	15.00	0	5.00	0.40

* All the dark chocolates had 42% sugar and milk chocolates had 45% sugar; all the chocolate samples had 0.03% vanillin. D0—dark chocolate without cocoa shells; DU—dark chocolate with untreated shells; DT—dark chocolate with treated shells; M0—milk chocolate without cocoa shells; MU—milk chocolate with untreated shells; MT—milk chocolate with treated shells; the number in the sample codes represents the percentage of shell addition.

Table 2. Composition of the second group of chocolate samples.

Sample *	Cocoa Mass (%)	Cocoa Butter (%)	Milk Powder (%)	Untreated Cocoa Shells (%)	Palm Oil (%)	Coconut Oil (%)	Whey (%)	Lecithin (%)	PGPR (%)
DU15—palm oil	21.00	16.57	0	15.00	5.00	0	0	0.40	0
DU15—coconut oil	21.00	16.57	0	15.00	0	5.00	0	0.40	0
DU15—PGPR + LEC	21.00	21.57	0	15.00	0	0	0	0.20	0.20
MU5—whey	9.74	24.83	10.00	5.00	0	0	5.00	0.40	0
MU5—palm oil	9.74	19.83	15.00	5.00	5.00	0	0	0.40	0
MU5—coconut oil	9.74	19.83	15.00	5.00	0	5.00	0	0.40	0
MU5—PGPR + LEC	9.74	24.83	15.00	5.00	0	0	0	0.20	0.20

* All dark chocolate samples had 42% sugar and milk chocolate samples had 45% sugar; all chocolates had 0.03% vanillin. DU—dark chocolate with untreated shells; MU—milk chocolate with untreated shells; the number in the sample codes represents the percentage of shell addition.

In all samples, emulsifiers (lecithin, A.C.E.F., Italy), and polyglycerol polyricinoleate (PGPR, supplied by Azelis, Croatia) were added one hour before the end of mixing, and vanillin was added half an hour before the end. Palm oil was purchased from Rapunzel (Germany) and coconut oil from Zvijezda d.o.o. (Croatia). Tempering was carried out by

hand, and the temperindex (4–7) was measured using the Sollich Tempermeter E3 (Bad Salzuflen, Germany). The tempered chocolate was molded and cooled at 8 °C for 30 min.

The first group of chocolate samples was produced with 5, 10, and 15% of untreated and HVED-treated cocoa shells for dark chocolate samples (DU5, DU10, DU15, DT5, DT10, DT15) and 2.5 and 5% of untreated and HVED-treated cocoa shells for milk chocolate samples (MU2.5, MU5, MT2.5, MT5).

To determine if the physical properties of chocolates with cocoa shells may be improved by different selections of raw materials, a second group of samples was produced. The dark chocolate with 15% of untreated cocoa shells and the milk chocolate with 5% of untreated cocoa shells were chosen for this part of the research based on their physico-chemical properties (along with the results presented in this paper, polyphenolic profile, antioxidant activity, HMF (5-hydroxymethylfurfural) and acrylamide content, peroxide value, and DSC (differential scanning calorimetry) results were taken into consideration). In dark chocolates, the part of cocoa butter was replaced with palm and coconut oil, and the part of lecithin with PGPR. In milk chocolates, the part of milk powder was replaced with whey powder, the part of cocoa butter with palm and coconut oil, and the part of lecithin with PGPR (Table 2).

2.3. Rheological Properties

The IOCCC (International Office of Cocoa, Chocolate and Sugar Confectionery) [11] method was used for the determination of the rheological properties of the chocolates. The Rheo Stress 600 (Haake, Vreden, Germany) was used, and the temperature of the samples was set to 40 °C. A concentric cylinder system was used for applying the method of the hysteresis loop. During the first 180 s, the shear rate was increased from 0 to 60 s⁻¹; after achieving the maximum speed, it was kept constant for 60 s, followed by a decrease in the shear rate from 60 to 0 s⁻¹ over 180 s. The results are presented as flow curves from shear stress (Pa) and shear rate (1/s).

2.4. Particle Size Distribution

Particle size distribution of chocolates was determined using the Mastersizer 2000 laser diffraction particle size analyzer (Malvern Instruments, Malvern, United Kingdom). The chocolates were dispersed in sunflower oil at room temperature and added to the Hydro 2000 µP unit, and adequate obscuration was obtained (10–20%). Measurements were performed in three repetitions and the results were quantified as the volume-based particle size distribution (Mastersizer v.2000 software). Values for the SSA, D [4,3], d(0,1), d(0,5), and d(0,9) were obtained. SSA is the specific surface area (m²/g), D [4,3] is the volume-weighted mean (µm), d(0,1) shows that 10% of the sample mass has particles that are smaller than the specified value, d(0,5) shows that 50% of the sample mass has particles that are smaller than the specified value, and d(0,9) shows that 90% of the sample mass has particles that are smaller than the specified value.

3. Hardness

Hardness was measured using the Texture Analyser TA.TX (Stable Micro systems, Surrey, United Kingdom) with a “three-point bending rig” in five repetitions at ambient temperature. Four chocolate squares were used for the measurements. Hardness represents the force needed to break the chocolate, and it is expressed in grams.

3.1. Color

The color of the chocolates was determined after cooling and after 24 h, 48 h, one week, two weeks, one month, and two months of storage at room temperature. A Konica Minolta CR-400 (Tokyo, Japan) chromameter was used. Measurements were performed in five repetitions in a CIEL*a*b* system. Mean values and standard deviations were

calculated from these results. The total color change (ΔE) and the whiteness index (WI) were calculated from the L^* , a^* , and b^* parameters according to Equations (1) and (2):

$$\Delta E = \sqrt{(L - L_0)^2 + (b - b_0)^2 + (a - a_0)^2} \quad (1)$$

$$WI = 100 - [(100 - L)^2 + a^2 + b^2]^{0.5}, \quad (2)$$

where L_0 , a_0 , and b_0 represent the values for the chocolates after cooling and L , a , and b are the values after storage.

3.2. Statistical Analysis

For statistical analysis, Statistica[®] Version 13.4.0.14 (1984-2018 TIBCO Software Inc.) was used. One-way analysis of variance (ANOVA) ($p < 0.05$) was used to determine whether the chocolate type, the cocoa shell type, and the content of cocoa shells (grouping variables) had a statistically significant effect on the dependent variables. After that, the least significant difference (LSD) post-hoc test was conducted ($p < 0.05$).

4. Results and Discussion

4.1. Rheological Properties

The rheological parameters of chocolate are important for sensory acceptability and the processing of chocolate. A change in chocolate formulation and the incorporation of unconventional materials should not significantly change the flow properties of chocolate [12]. Figure 1 shows the flow curves of the dark chocolates (the first group of samples). Dark chocolate without added cocoa shells (D0) had the lowest viscosity. In addition, chocolates with untreated cocoa shells had lower viscosities than those with HVED-treated shells. In our previous study, it was shown that treated cocoa shells had a higher oil-binding capacity than untreated ones [13]. Bolenz et al. [14] stated that wheat fibers and cellulose can absorb oil, which leads to a reduced continuous fat phase. Namely, fat is trapped within the fiber structure, and it leads to more contact between non-fat particles and decreased flow properties.

Cocoa shells treated with HVED had lower density than untreated cocoa shells (results not shown), probably because of the higher porosity due to the combination of freeze-drying and the HVED treatment. Freeze-drying results in water sublimation and pore formation [15], and HVED treatment disrupts cell walls [16]. This results in larger contact surfaces of solid particles and higher viscosities of chocolates when HVED-treated shells are used. The same was reported by Rezende et al. [17] for inulin- and β -glucan-substituted chocolates.

Based on the overall properties (not all are shown in this paper), the best overall quality attributes were achieved when 15% of untreated cocoa shells were added, so we chose the chocolate with 15% of untreated cocoa shells to examine the influence of other ingredients on the flow properties (Figure 2). The additions of 5% of coconut and palm oil did not show any significant changes. When 0.2% of PGPR was used, the chocolate had lower values of yield stress (the value of stress at which chocolate starts to flow).

Milk chocolates with added cocoa shells showed the same trend as the dark chocolates (Figure 3). Each addition of shells increased the viscosity of the chocolates, with a more pronounced effect from the treated cocoa shells. The chocolate with 5% of untreated cocoa shells was chosen for the second group of milk chocolate samples (Figure 4). The additions of 5% of whey, 0.2% of PGPR, and 5% of palm and coconut oil had different effects on the flow curves from those of the dark chocolates. Coconut oil had the best effect on lowering the viscosity of milk chocolate, followed by palm oil, which had flow properties that were similar to those of the control sample (MU5). Abdul Halim et al. [18] also reported that coconut oil in chocolate could cause lower viscosity. Compared to palm oil and cocoa butter, coconut oil has the lowest melting point, which may cause a better flow of the chocolate. The results obtained in this research indicate that defects in the flow properties of chocolate

due to the addition of non-standard ingredients may be improved by the addition of other fats or emulsifiers. The addition of whey increased the viscosity of chocolate, while the addition of PGPR resulted in lower values of yield stress, as observed in dark chocolate.

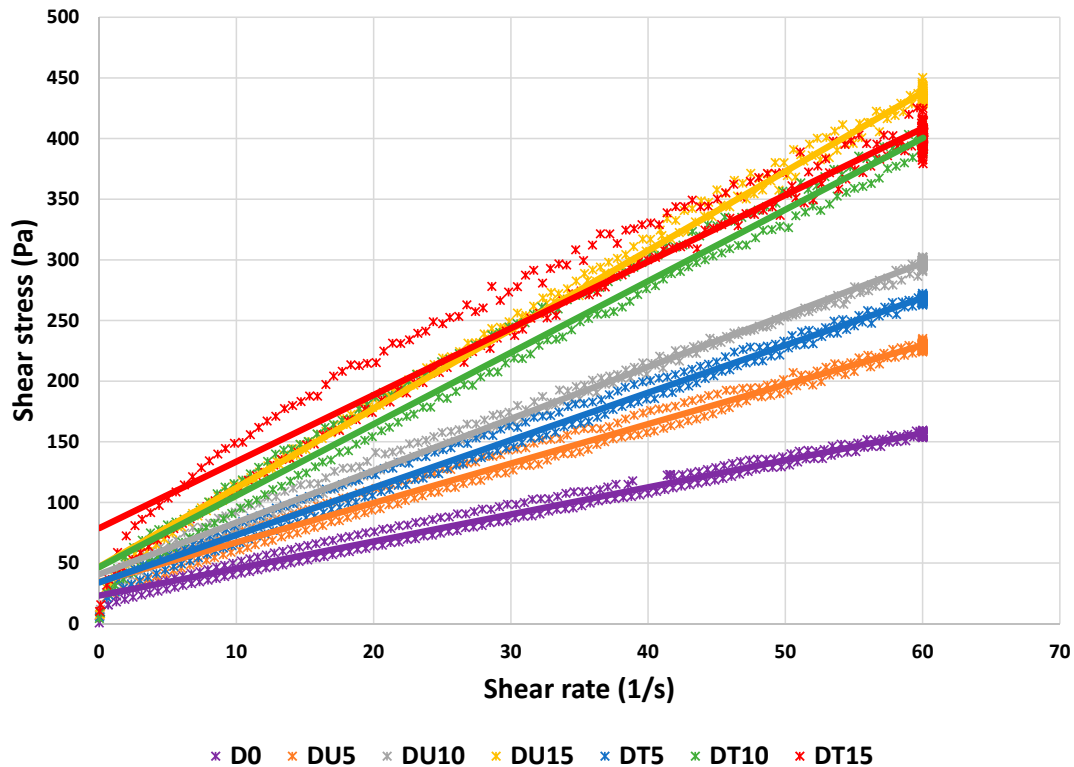


Figure 1. Flow curves of dark chocolates from the first group of samples. (DU—dark chocolate with untreated shells; MU—milk chocolate with untreated shells; numbers in the sample codes represent the percentage of shell addition).

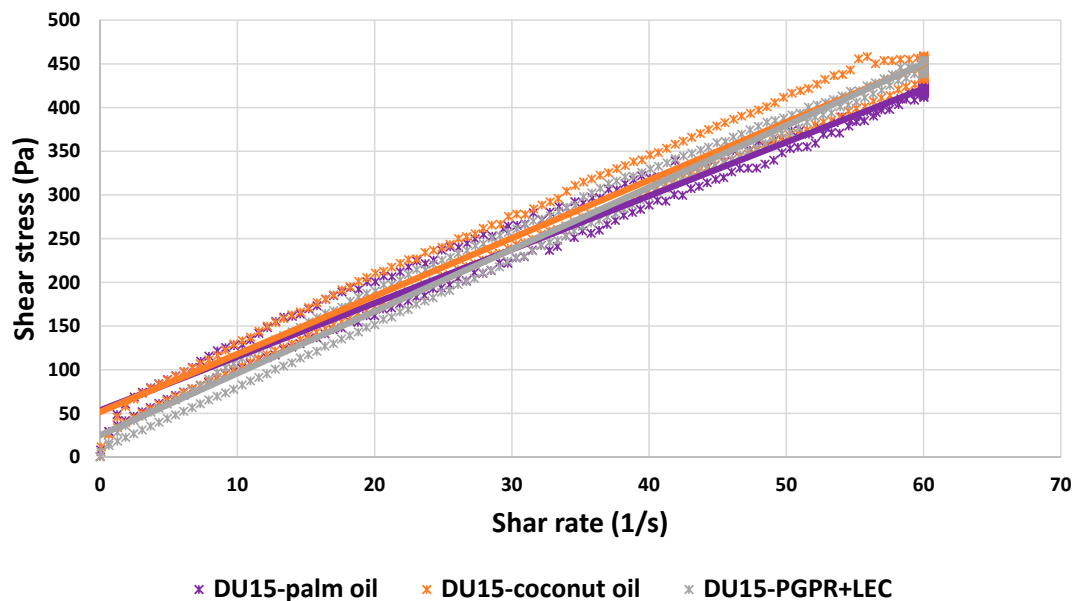


Figure 2. Flow curves of dark chocolates from the second group of samples (DU—dark chocolate with untreated shells; numbers in the sample codes represents the percentage of shell addition).

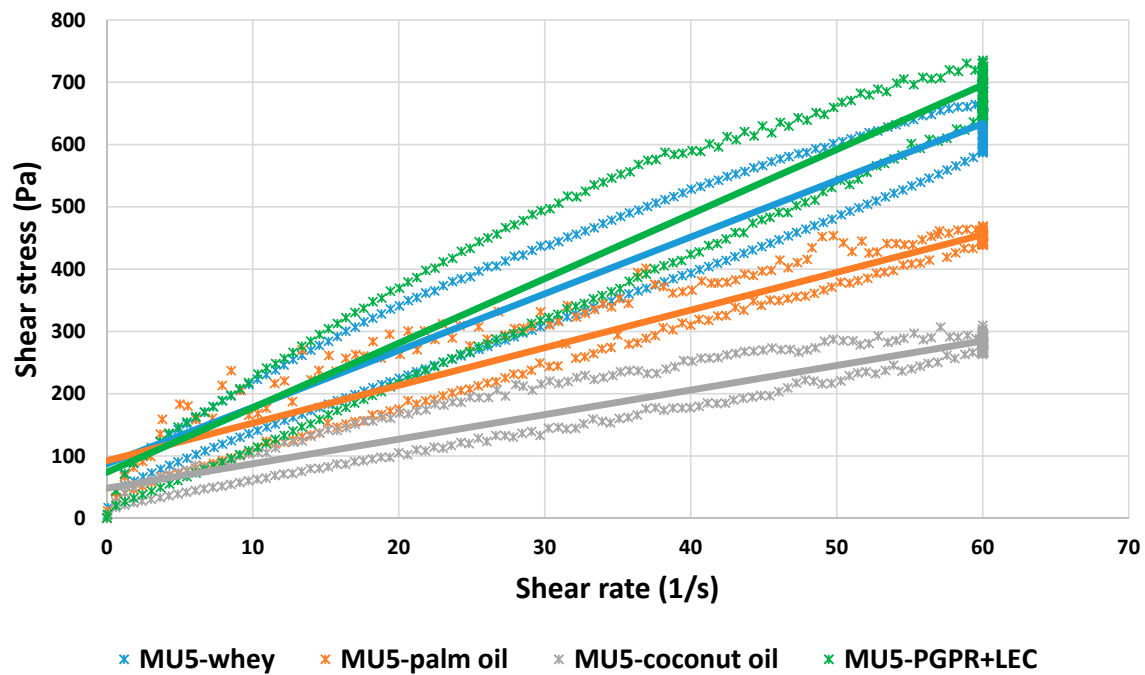


Figure 3. Flow curves of milk chocolates from the first group of samples (MU—milk chocolate with untreated shells; numbers in the sample codes represent the percentage of shell addition).

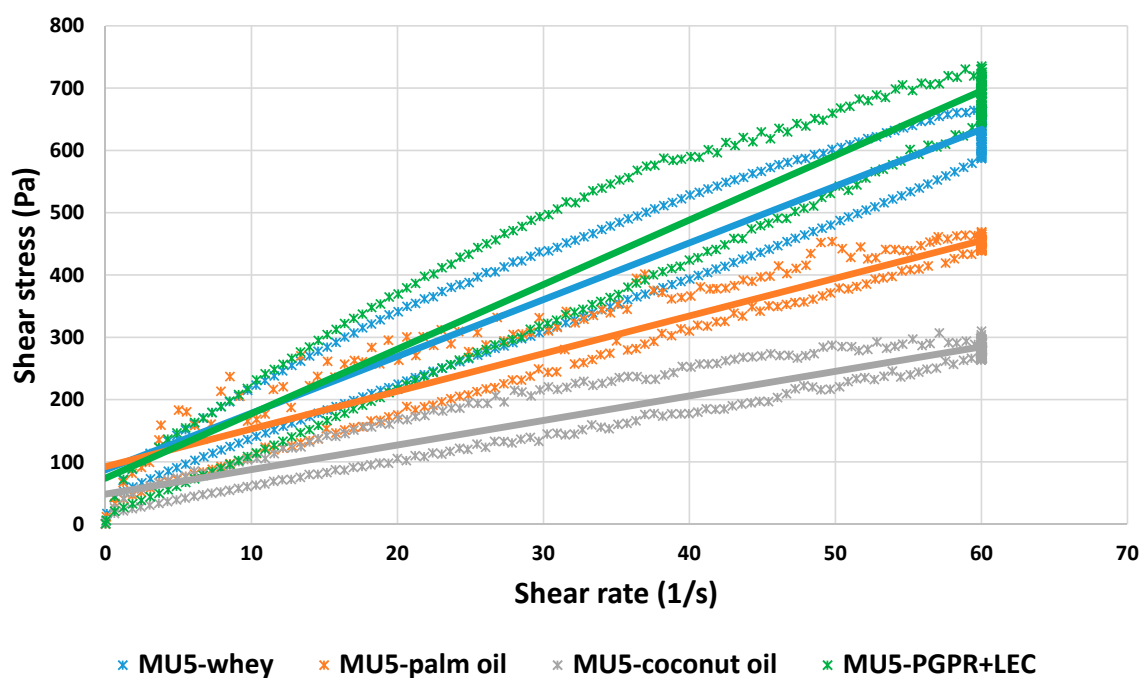


Figure 4. Flow curves of milk chocolates from the second group of samples (MU—milk chocolate with untreated shells; numbers in the sample codes represent the percentage of shell addition).

4.2. Particle Size Distribution

Particle size is one of the most important properties of chocolate. The distribution of particle sizes can affect the hardness, color, and rheological properties. The desired particle size range for chocolate is 17–30 μm [19] because it improves the sensory properties [20]. The specific surface area (SSA), particle size ($d(0.1)$, $d(0.5)$, and $d(0.9)$), and

volume-weighted mean ($D[4,3]$) are in direct correlation. The smaller the particles are, the more SSA increases, and the more fat is needed to coat the particles and to allow chocolate to flow [21]. Figures 5 and 6 show that samples DT10 and DU15—coconut oil, which had a larger particle size, had lower SSAs compared to the other chocolates. All dark and milk chocolate samples with added cocoa shells had larger particles. Bolenz et al. [14] reported that the addition of cellulose and wheat fiber caused an increase in particle size. Since cocoa shells are also mostly composed of cellulose and other fibers, it is more difficult to reduce their particle size during the chocolate production process. Therefore, only particles of cocoa shells smaller than $71\ \mu\text{m}$ were added in the ball mill half an hour before other ingredients in order to overcome this issue. Namely, particles that were too large were discarded before the addition to chocolate, and a prolonged mixing time in the ball mill was more effective in the milling. However, future research is needed to evaluate whether even longer mixing of cocoa shells in a ball mill can produce smaller particles.

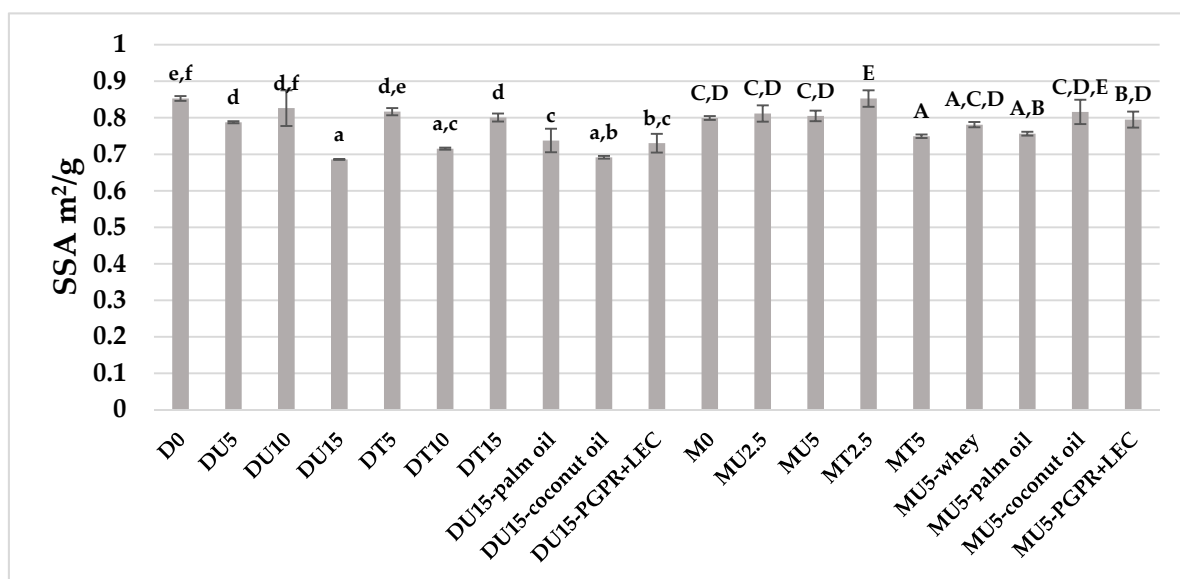


Figure 5. Specific surface area (SSA) of chocolate samples (DU—dark chocolate with untreated shells; MU—milk chocolate with untreated shells; numbers in the sample codes represent the percentage of shell addition; different superscripts within the same row indicate significant differences between samples).

The results also show that the dark and milk chocolate samples with HVED-treated cocoa shells had larger particles than the chocolates with untreated cocoa shells. This is in accordance with our previous research [13], where we showed that cocoa shells treated with HVED were harder to grind. According to Konar et al. [22], particle size distribution inversely affects the hardness of chocolate because of the particle–particle interactions. This is in correlation with the results for hardness discussed later in the text. The statistical analysis showed that $D[4,3]$, $d(0.9)$, and $d(0.5)$ were significantly affected by the chocolate type and the type and content of cocoa shells, while $d(0.1)$ was not affected by any of them, which is also shown in Figure 6. Dark chocolates and chocolates with treated cocoa shells had larger values of $D[4,3]$, $d(0.9)$, and $d(0.5)$. In addition, with a larger amount of cocoa shells (untreated and treated), these values increased. This indicates that the cocoa shells were probably harder to grind and caused an increase in particle size.

If the influence of cocoa butter equivalents (CBEs) was observed for dark chocolates, the striking difference between samples was seen in $d(0.9)$ particles (Figure 6). While the size of the particles in chocolate produced with palm oil was below $40\ \mu\text{m}$ and the size of the largest particles without CBEs was close to $40\ \mu\text{m}$, the size of particles when coconut oil was used got close to $50\ \mu\text{m}$. This could be due to the low melting point of coconut oil and the better lubrication during ball milling, resulting in less effective contact of the balls

with the particles of the chocolate ingredients. Specific surface area, however, was more alike between the coconut oil sample and the sample without CBEs (Figure 5). For milk chocolates, the particle size increased with both palm and coconut oil addition, but again, coconut oil did not significantly influence the SSA. PGPR addition also resulted in a larger particle size. This can also be attributed to the lubricating effect of the emulsifier and the lower shear stress observed above.

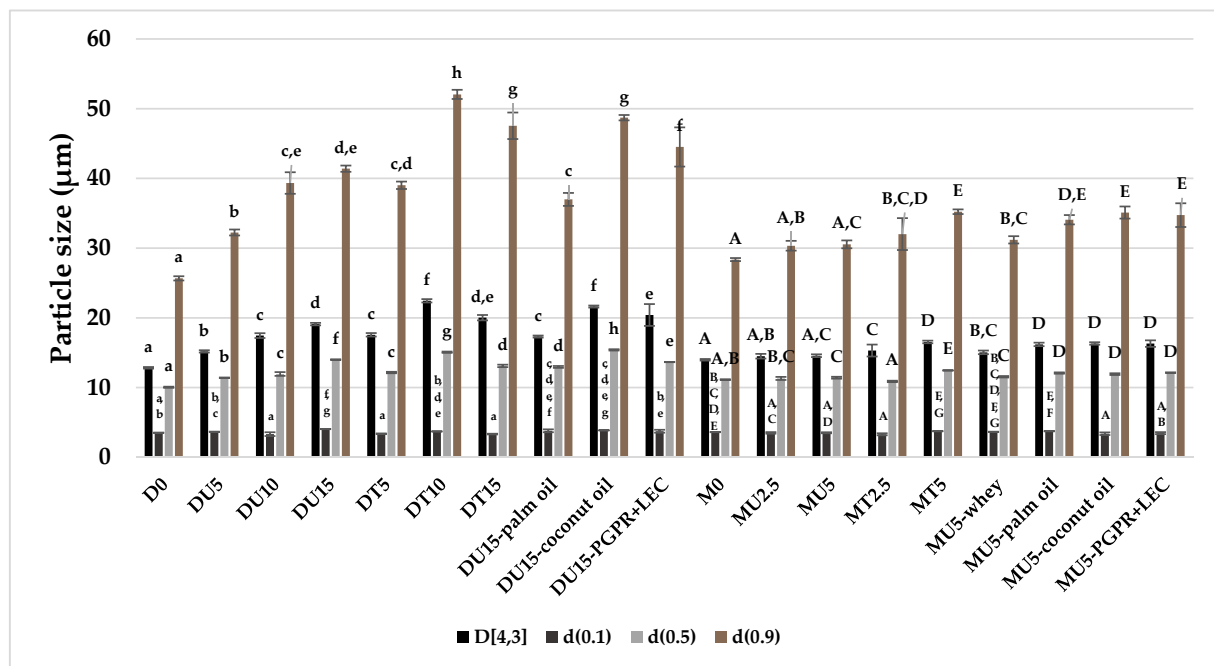


Figure 6. Particle sizes of chocolate samples (DU—dark chocolate with untreated shells; MU—milk chocolate with untreated shells; numbers in the sample codes represent the percentage of shell addition; different superscripts within the same row indicate significant differences between samples).

4.3. Hardness

The hardness of chocolate depends on the content and type of fat, sugar, and cocoa particles, and can predict the melting time in the human mouth during consumption [20]. A quality chocolate should be soft and smooth. Figure 7 shows the hardness of the dark and milk chocolate samples with added cocoa shells. In the first group of samples, the dark chocolate without added cocoa shells had the greatest hardness. All samples with added cocoa shells had lower values. The cocoa shells replaced a part of the cocoa mass in all the samples, resulting in lower total fat content in the enriched samples. Do et al. [23] reported that increases in fat content decrease the hardness of chocolates. However, the chocolates in their research were not substituted with raw materials that are not normally used in chocolate production. Lee et al. [24] produced chocolates with a β -glucan-rich hydrocolloid that replaced cocoa butter. In their research, the hardness also decreased as β -glucan content increased.

Among the samples with HVED-treated cocoa shells, dark chocolate with 10% of shells had the greatest hardness, but among the samples with untreated cocoa shells, the dark chocolate with 10% of shells had the lowest value. Do et al. [23] also reported that there was an inverse relationship between particle size and hardness because of the relative strengths of particle–particle interactions. If particles are larger, interactions are going to be weaker and the hardness of the chocolate will be lower.

The milk chocolates showed the opposite trend—the hardness slightly increased with the addition of cocoa shells. Although increasing the content of cocoa shells resulted in a decrease in hardness, it was still greater than that of the control. The addition of

milk fat softened the chocolate; therefore, the effect of cocoa shell addition on particle size distribution (larger particles—better flow) was overcome by the effect of reduced fat-to-nonfat-mass ratio (more non-fat particles to be coated by the same amount of fat result in an increase in viscosity).

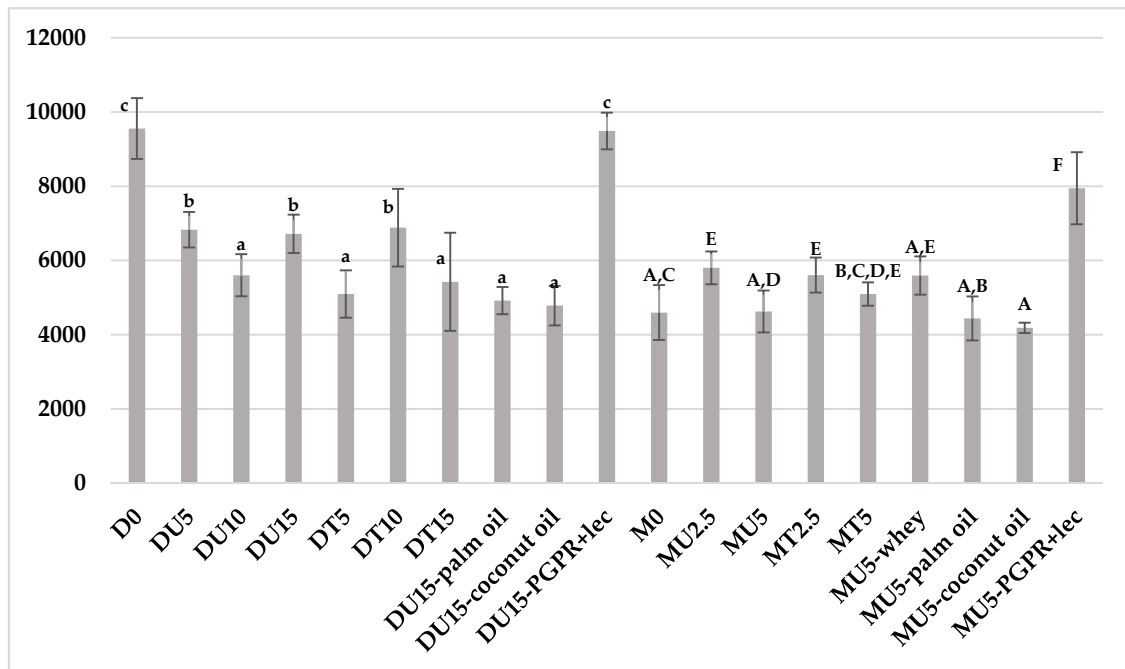


Figure 7. Hardness of chocolate samples (DU—dark chocolate with untreated shells; MU—milk chocolate with untreated shells; numbers in the sample codes represent the percentage of shell addition; different superscripts within the same row indicate significant differences between samples).

The addition of palm and coconut oil in dark and milk chocolate samples can result in softer chocolates. In our previous research, milk and dark chocolate samples with coconut and palm oil had lower hardness values [25]. These oils contain lauric acid and therefore melt at lower temperatures than cocoa butter [26]. For the same reason, they could alter the crystal network of the stable (V) crystals of cocoa butter and cause lower hardness values. The addition of whey and the replacement of the part of lecithin with PGPR resulted in harder chocolates than chocolates with the same content of cocoa shells. Sözeri et al. [27] also reported that chocolate with the highest content of PGPR had the highest hardness. They stated that this was probably due to a more homogenous distribution of particles in chocolate with PGPR, which had caused increased cohesiveness between particles.

4.4. Color

The color of chocolate is the first property that will have an impact on consumers and their desire for consumption. A darker color of chocolate indicates a higher content of cocoa particles and is a desired feature. In addition, white lines on the chocolate's surface can be associated with fat bloom [28]. The dark and milk chocolate samples were darker than the reference sample, as shown in Figures 8 and 9. Only the chocolates with 10% of untreated (TU10) and treated (TT10) cocoa shells had a larger total color change after two months in comparison with the reference sample (T0). This shows that chocolates with cocoa shells would be more desired because cocoa shells caused darkening and reduced the color change over the two-month period. Darkening of chocolates was also observed after the addition of inulin [29] and polydextrose [30]. Shourideh et al. [29] reported that fiber could absorb moisture and decrease light scattering, due to which chocolate seems darker.

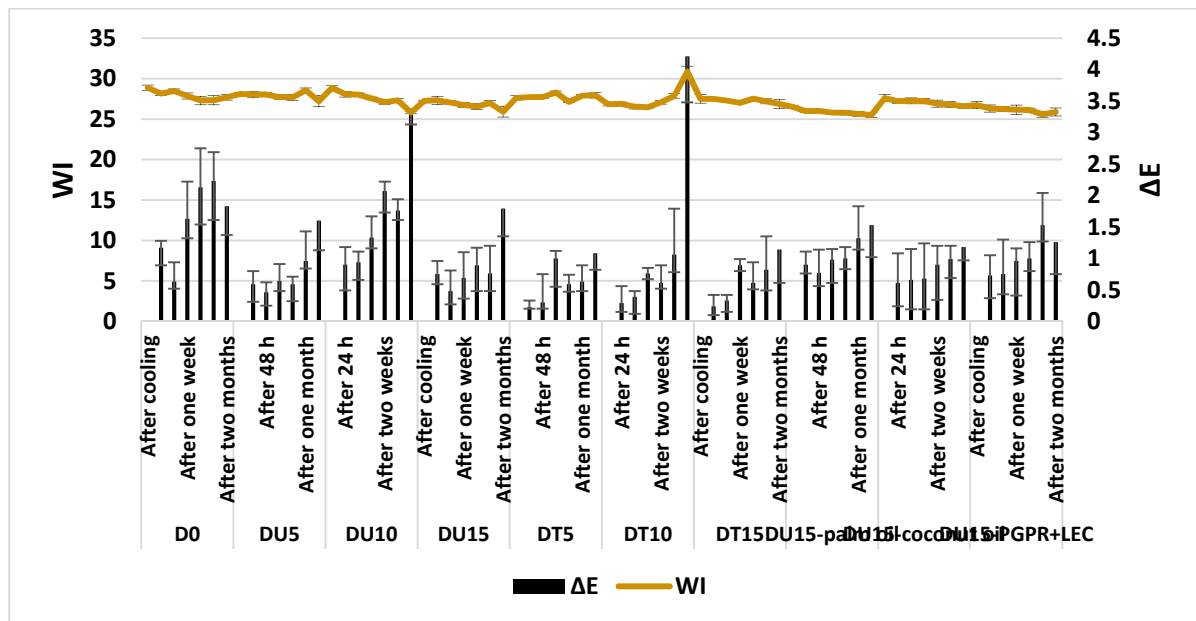


Figure 8. Whiteness index (WI) and the total color change (ΔE) of dark chocolates during two months of storage.

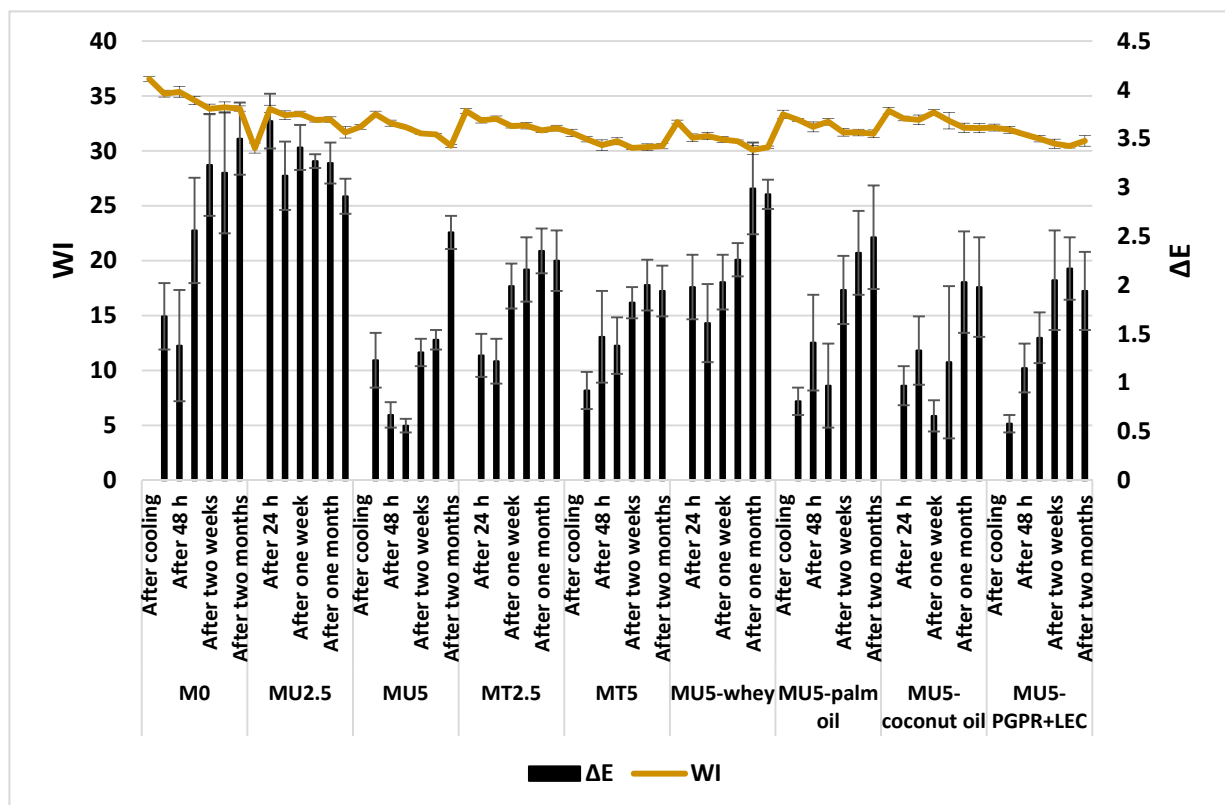


Figure 9. Whiteness index (WI) and the total color change (ΔE) of milk chocolates during two months of storage.

Statistical analysis (Table 3) showed that the cocoa shell type (treated or untreated) had a significant effect on the WI after cooling, on ΔE after two months, and on the WI after two months. The chocolate type had a statistically significant effect on the WI after cooling and after two months of storage, and the content of cocoa shells had significant effects on all three dependent variables. The total color change (ΔE) increased after two

months in all chocolates, but with the addition of treated cocoa shells (5 and 15%), it was lower than with the addition of untreated shells in the same share. The whiteness index (WI) was lower in all chocolates with added cocoa shells compared to ones without cocoa shells, and the values decreased as the proportion of cocoa shells increased (after cooling and after two months).

Table 3. One-way analysis of variance (ANOVA).

Depending Variable	Grouping Variable	p-Value *	Depending Variable	Grouping Variable	p-Value *
Hardness	Chocolate type	0.000039	SSA	Chocolate type	0.269958
	Cocoa shell type	0.128834		Cocoa shell type	0.226690
	Content of cocoa shells	0.180568		Content of cocoa shells	0.009543
WI after cooling	Chocolate type	<0.000001	d(0.1)	Chocolate type	0.709513
	Cocoa shell type	0.003174		Cocoa shell type	0.410736
	Content of cocoa shells	0.000001		Content of cocoa shells	0.328927
ΔE after 2 months	Chocolate type	0.068028	d(0.5)	Chocolate type	0.016237
	Cocoa shell type	0.326695		Cocoa shell type	0.002778
	Content of cocoa shells	<0.000001		Content of cocoa shells	<0.000001
WI after 2 months	Chocolate type	<0.000001	d(0.9)	Chocolate type	0.000828
	Cocoa shell type	0.01627		Cocoa shell type	0.000107
	Content of cocoa shells	0.000001		Content of cocoa shells	<0.000001
D [3,4]	Chocolate type	0.001408			
	Cocoa shell type	0.000124			
	Content of cocoa shells	<0.000001			

* Bold values were considered significant at $p < 0.05$.

The same trend as that in dark chocolates could be observed in milk chocolates. All the chocolates with added cocoa shells had lower values of the whiteness index and total color change after two months of storage. Generally, milk chocolates are more resistant to fat bloom than dark chocolates because milk fat has a lower melting point than cocoa butter and slows down the polymorphic transformation of cocoa butter crystals, which is the main reason for fat bloom [31,32]. It is possible that cocoa shells also affect the cocoa butter crystal network and slow down the migration of fat and the formation of fat bloom.

5. Conclusions

Chocolates with the addition of cocoa shells showed potential for actual application in the industry. The hardness and color of chocolates with both untreated and HVED-treated cocoa shells proved to be desirable, with even better properties than conventional chocolate in terms of consumer acceptance. However, in terms of particle size and rheology, it is necessary to further investigate the impacts of different processing conditions (time of refining and conching, time of addition and proportions of different ingredients, etc.) and applications of different ingredients (fat, emulsifiers) so that such chocolates do not differ significantly from conventional chocolates. These are the properties that are in direct correlation to the mouthfeel of chocolate, and they largely influence the perception of the aroma. Additionally, the potential for labeling the chocolate as a functional product (in terms of increased fiber content) and safety aspects (microbial properties, PAHs, and acrylamide content) will also be taken into consideration in future research, as well as a sensory evaluation by a trained sensory panel and an assessment of consumer acceptability.

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