

## Article

# Influence of Fat Choice on Physical Properties of Sustainable, Nutritionally Improved Cocoa Spreads

Đurđica Ačkar <sup>1,\*</sup> , Veronika Barišić <sup>2,\*</sup> , Ivana Lončarević <sup>3</sup>, Jovana Petrović <sup>3</sup> , Kristina Doko <sup>4</sup> and Ivana Flanjak <sup>1</sup> 

<sup>1</sup> Faculty of Food Technology Osijek, Josip Juraj Strossmayer University of Osijek, Franje Kuhača 18, 31000 Osijek, Croatia; ivana.flanjak@ptfos.hr

<sup>2</sup> Faculty of Tourism and Rural Development in Požega, Josip Juraj Strossmayer University of Osijek, Vukovarska 17, 34000 Požega, Croatia

<sup>3</sup> Faculty of Technology Novi Sad, University of Novi Sad, Bul. Cara Lazara 1, 21000 Novi Sad, Serbia; ivana.loncarevic@uns.ac.rs (I.L.); jovana.petrovic@uns.ac.rs (J.P.)

<sup>4</sup> Federal Agro Mediterranean Institute, Biskupa Čule 10, 88000 Mostar, Bosnia and Herzegovina; kristina.skender@gmail.com

\* Correspondence: dackar@ptfos.hr (Đ.A.); vbarisic@ftrr.hr (V.B.)

## Featured Application

This research is ground for the use of cocoa shell in food processing, e.g., sweet spreads and creams. Due to the coarse particles of the shell, the fat type should be adjusted, and the research shows how different combinations influence texture and rheology.

## Abstract

The aim of this research was to examine the possibility of achieving optimum texture and rheological properties of cocoa spreads produced with cocoa shell, xylitol, and stevia as sugar replacers using different vegetable fats (palm and coconut oil). Samples with different proportions of cocoa shell and sweeteners used to completely replace sugar were produced under laboratory conditions, and rheology, texture, colloidal stability, and particle size were determined. The results showed that texture properties (spreadability and firmness) and yield stress directly correlated with the type of fat used in the recipe, with the best values obtained for coconut fat. Cocoa shell addition increases Casson plastic viscosity (from approximately 2 to 7 Pas) but may reduce Casson yield stress if added in a proportion up to 25% (from approximately 26 to 18 Pa for samples produced with a combination of palm and coconut oil). Overall, samples with coconut fat had better texture properties regardless of the cocoa shell and sweetener quantities, and the sample with the lowest content of cocoa shell had the best spreadability. By careful adjustment of ingredients and the right choice of substitute fat, sustainable, nutritionally improved cocoa spreads with satisfactory texture and rheology may be produced.



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## 1. Introduction

Sweet spreads, such as cocoa and hazelnut spreads, are products made of sugar, vegetable fat, hazelnut paste, and/or cocoa powder, often with milk powder. These are energy-dense products with relatively low nutritional value, but they are a very popular choice for breakfast and dessert among all age groups worldwide. According to Statista Market Insights [1], in 2024, 2.44 billion kilograms of chocolate spreads were sold

worldwide, with a predicted rise to almost 3 billion kilograms in 2030. Among European countries, Croatia is leading with 1.84 kg per capita, followed by Italy (1.65 kg per capita) and Bulgaria (1.22 kg per capita) [2]. However, problems related to the increased incidence of obesity in developed countries, linked to non-communicable diseases, such as type II diabetes, hypertension, stroke, and even some cancers, have led to increasing public awareness regarding a healthy diet. The World Health Organization published healthy diet recommendations: eating plenty of fruit and vegetables, reducing the intake of fat, sugar, and salt [3]. The need for a reduction in sugar consumption has triggered numerous studies regarding sugar substitutes and their use in food. In addition to sweeteners approved for use in food, scientists are constantly seeking new sugar substitutes and innovative solutions for sugar content reduction in different products. For example, the possibility of microbial synthesis of sweeteners has been investigated by Qu et al. [4], Luu et al. [5], and Seo et al. [6], who investigated the influence of erythritol on gut health, and Lei et al. [7] investigated the influence of stevia on diabetes management. Sucrose replacement was researched in cookies [8,9], biscuits [10,11], and chocolate [12], and Petkovic et al. [13] examined the influence of maltitol on textural and sensory properties of cocoa spreads.

Pirouzian et al. [14] examined the use of polyols—maltitol, lactitol, and xylitol—in the production of probiotic chocolate. For the 100% replacement of sugar with xylitol, a significant increase in particle size and Casson viscosity was observed, with a decrease in Casson yield and hardness. Goktas and Agan [15] used xylitol in chocolate pudding, reporting the increase in pH value and rheological parameters, and Shanthamma et al. [16] reported lowering GI while retaining acceptable sensory properties of chocolates by substituting sucrose with sorbitol, fructooligosaccharide, inulin, xylitol, and palm sugar.

The issues related to sugar content reduction in chocolate and sweet spreads are not only the changes in taste of the products but also the textural [13], rheological, and even color changes can occur due to the addition of novel ingredients. Acan et al. [17] used grape pomace, a by-product from wine production, as a nutritional, sustainable, and cost-effective substitute for sucrose and milk powder and observed an increase in firmness and reduced spreadability of the novel products compared to the standard. Berk et al. [18] reported a darker color, reduced Casson yield stress, and changes in textural properties due to the replacement of sugar in cocoa hazelnut spreads with stevia and inulin, and B  ker et al. [19] reported that the use of apple pomace, a by-product from fruit processing, as a sustainable sugar substitute imparted color, hardness, and affected the overall acceptability of chocolate spreads.

On the other hand, the choice of fat often influences the texture, melting properties, mouthfeel, appearance, and flavor of food products. The ratio of short-, medium-, and long-chain fatty acids in fats gives different solubility, stability, and viscosity characteristics to the fat [20]. Palm and coconut oil contain high contents of saturated long-chain fatty acids (lauric and myristic); however, they differ in contents of medium-chain fatty acids—while coconut oil contains significant proportions of caproic (C6), caprylic (C8), and capric acid (C10) [21], palm oil does not contain these fatty acids [22].

To the best of our knowledge, there has not been any attempt to combine xylitol with cocoa shell as a source of fiber in the production of sweet spreads, other than our research, part of which has already been published [23]. We presuppose that problems with rheological and textural properties caused by the addition of cocoa shell might be resolved by the adjustment of fat type and quantity in the recipe, and the aim of this research was to investigate the influence of palm and coconut oil on the properties of cocoa spreads produced with cocoa shell and stevia as sugar substitutes.

## 2. Materials and Methods

For the purpose of this research, cocoa powder produced by Kandit d.o.o., Osijek, Croatia, palm oil donated by Kandit d.o.o., Osijek, Croatia, coconut oil from Zvijezda d. d., Zagreb, Croatia, hazelnut paste from DGF, Paris, France, xylitol from Nutrigold, Tvornica zdrave hrane, Zagreb, Croatia, stevia from Nutrimea d.o.o., Zagreb, Croatia, sugar from Viro, Virovitica, Croatia, vanillin from Acros Organics, Geel, Belgium, and lecithin from A.C.E.F, FLorencuola d'Arda, Italy, were used. Cocoa shell was obtained under industrial conditions after roasting cocoa beans at 135 °C for 55 min.

Samples were produced using a custom-made laboratory ball mill, Faculty of Food Technology Osijek & D&D, Osijek, Croatia, according to the recipes shown in Table 1. The ball mill was thermostated at 50 °C, the shearing speed was set to 60 rpm, and the stainless-steel ball/spread ratio was 6:1. Total shearing time was 3 h for samples without cocoa shell and 3.5 h for samples with cocoa shell. Namely, when cocoa shell was used, it was sheared with fats for 30 min, and then other raw materials were added. After further 2 h, lecithin was added, and vanillin was added 30 min before the end of the process.

**Table 1.** Recipes of cocoa spreads produced with the substitution of sugar with cocoa shell, xylitol, and stevia, and the addition of palm and coconut oil (percentages are expressed as a % of the total mass of the sweet spread).

|      | Cocoa Powder (%) | Hazelnut Paste (%) | Palm Oil (%) | Coconut Oil (%) | Sweetener (%)                   | Cocoa Shell (%) |
|------|------------------|--------------------|--------------|-----------------|---------------------------------|-----------------|
| N0   | 6.3              | 17                 | 10           | 10              | sugar: 52.0                     | -               |
| N6   | 6.3              | 17                 | 10           | 10              | xylitol: 52.0                   | -               |
| N6-K | 6.3              | 17                 | -            | 20              | xylitol: 52.0                   | -               |
| N6-P | 6.3              | 17                 | 20           | -               | xylitol: 52.0                   | -               |
| N5   | 6.3              | 17                 | 10           | 10              | xylitol: 46.8<br>stevia: 0.0208 | 5.1792          |
| N5-K | 6.3              | 17                 | -            | 20              | xylitol: 46.8<br>stevia: 0.0208 | 5.1792          |
| N5-P | 6.3              | 17                 | 20           | -               | xylitol: 46.8<br>stevia: 0.0208 | 5.1792          |
| N4   | 6.3              | 17                 | 10           | 10              | xylitol: 41.6<br>stevia: 0.0424 | 10.3576         |
| N4-K | 6.3              | 17                 | -            | 20              | xylitol: 41.6<br>stevia: 0.0424 | 10.3576         |
| N4-P | 6.3              | 17                 | 20           | -               | xylitol: 41.6<br>stevia: 0.0424 | 10.3576         |
| N3   | 6.3              | 17                 | 10           | 10              | xylitol: 36.4<br>stevia: 0.0624 | 15.5376         |
| N3-K | 6.3              | 17                 | -            | 20              | xylitol: 36.4<br>stevia: 0.0624 | 15.5376         |
| N3-P | 6.3              | 17                 | 20           | -               | xylitol: 36.4<br>stevia: 0.0624 | 15.5376         |
| N2   | 6.3              | 17                 | 10           | 10              | xylitol: 31.2<br>stevia: 0.0832 | 20.7168         |
| N2-K | 6.3              | 17                 | -            | 20              | xylitol: 31.2<br>stevia: 0.0832 | 20.7168         |

Table 1. Cont.

|      | Cocoa Powder (%) | Hazelnut Paste (%) | Palm Oil (%) | Coconut Oil (%) | Sweetener (%)                   | Cocoa Shell (%) |
|------|------------------|--------------------|--------------|-----------------|---------------------------------|-----------------|
| N2-P | 6.3              | 17                 | 20           | -               | xylitol: 31.2<br>stevia: 0.0832 | 20.7168         |
| N1   | 6.3              | 17                 | 10           | 10              | xylitol: 26.0<br>stevia: 0.104  | 25.8960         |
| N1-K | 6.3              | 17                 | -            | 20              | xylitol: 26.0<br>stevia: 0.104  | 25.8960         |
| N1-P | 6.3              | 17                 | 20           | -               | xylitol: 26.0<br>stevia: 0.104  | 25.8960         |

All samples contained 4.1% milk powder, 0.4% lecithin, 0.1% vanillin, and 0.1% salt.

The fatty acid profile of the sweet spreads was determined after extraction of lipids by Folch, using GC-FID, according to a previously described method [23]. Mono- (MUFA), poly- (PUFA), and saturated fatty acid (SFA) contents were calculated from the mean values obtained by chromatographic analysis.

Dietary fiber content was determined according to AOAC method 991.43 [24], using the Megazyme Total Dietary Fiber Assay Kit.

Protein content was determined according to the ISO 5983-2:2005 method [25], using a factor of 6.25 for conversion.

Colloidal stability was determined as oil separated after heating samples at 80 °C for 15 min, cooling to ambient temperature, and centrifuging at 2900 × g for 20 min [23].

Firmness and spreadability were determined as the maximum force and area below the curve obtained by measurement using a TA.XT Plus texture analyzer (Stable Micro System, Godalming, UK) with a TTC spreadability rig [23].

Density was calculated from the weight of a known volume of the samples [23].

Rheological properties (flow curves, Casson yield stress, and Casson plastic viscosity) were determined using a Rheo Stress 600 rheometer (Haake, Thermo Scientific, Waltham, MA, USA). Samples were thermostated at 40 °C, and the parameters were obtained using the computer software [23].

Particle size was determined using a Mastersizer 2000 (Malvern Instruments, Malvern, UK) after dispersing samples in sunflower oil [23].

All analyses were performed in triplicate. Statistical analysis was performed using Statistica® 14.2.2.18 software and breakdown and one-way ANOVA. Mean values and standard deviations were calculated, and least significant difference (LSD) and correlation analysis were determined at  $p < 0.05$ .

### 3. Results and Discussion

In our previous paper [20], we have shown that the addition of cocoa shell influences the fatty acid profile (SFA, MUFA, PUFA, SFA/MUFA, and SFA/PUFA) of the sweet spreads. In the present paper, we further examined the influence of palm and coconut oil on the fatty acid profile, shown in Table 2 and Figure 1. Coconut oil addition resulted in C6 and C8 appearance in sweet spreads. These fatty acids were not detected in samples produced with palm oil as a single fat (Table 2). Coconut oil is considered a good source of medium-chain fatty acids, which are important in the management of metabolic diseases [26] (Roopashree et al., 2021). However, Cao et al. [27] recommend a daily supplementation of 10–20 g to gain a therapeutic effect. Although sweet spreads cannot be considered a dietary supplement, they may contribute to the overall intake of medium-chain fatty acids.

**Table 2.** Fatty acid profile of sweet spreads produced with different contents of cocoa shell and sweeteners (marked with numbers) and coconut (K) and palm oil (P). The values are expressed as a % of total fatty acids (mean value of triplicate analysis).

|      | C6:0 | C8:0 | C10:0 | C12:0 | C14:0 | C16:0 | C16:1 | C18:0 | C18:1n9c+t | C18:2n6c | C18:3n3 | C20:0 | C20:1 | C22:0 |
|------|------|------|-------|-------|-------|-------|-------|-------|------------|----------|---------|-------|-------|-------|
|      | %    | %    | %     | %     | %     | %     | %     | %     | %          | %        | %       | %     | %     | %     |
| N0   | 0.22 | 2.11 | 1.64  | 13.41 | 5.54  | 20.26 | 0.15  | 4.07  | 44.83      | 7.28     | 0.12    | 0.18  | 0.10  | 0.09  |
| N6   | 0.17 | 1.65 | 1.26  | 10.62 | 4.68  | 19.80 | 0.14  | 4.50  | 48.89      | 7.75     | 0.14    | 0.21  | 0.10  | 0.10  |
| N6-K | 0.38 | 4.30 | 3.24  | 28.51 | 10.61 | 9.13  | 0.11  | 3.59  | 34.88      | 5.08     | n.d.    | 0.11  | n.d.  | 0.06  |
| N6-P | n.d. | n.d. | 0.11  | 0.39  | 0.89  | 32.04 | 0.18  | 4.47  | 52.32      | 9.00     | 0.17    | 0.24  | 0.10  | 0.08  |
| N5   | 0.19 | 1.80 | 1.50  | 11.22 | 4.76  | 19.63 | 0.15  | 4.45  | 48.13      | 7.64     | 0.14    | 0.18  | 0.11  | 0.11  |
| N5-K | 0.38 | 4.28 | 3.23  | 28.29 | 10.57 | 9.15  | 0.12  | 3.74  | 35.07      | 4.96     | n.d.    | 0.12  | n.d.  | 0.07  |
| N5-P | n.d. | n.d. | 0.10  | 0.34  | 0.87  | 31.85 | 0.18  | 4.55  | 52.56      | 8.98     | 0.16    | 0.25  | 0.08  | 0.08  |
| N4   | 0.18 | 1.71 | 1.31  | 10.73 | 4.66  | 19.73 | 0.15  | 4.56  | 48.69      | 7.71     | 0.14    | 0.21  | 0.10  | 0.11  |
| N4-K | 0.38 | 4.25 | 3.20  | 28.16 | 10.52 | 9.38  | 0.12  | 3.75  | 34.98      | 5.04     | n.d.    | 0.12  | n.d.  | 0.09  |
| N4-P | n.d. | n.d. | 0.10  | 0.36  | 0.86  | 31.13 | 0.18  | 4.55  | 53.20      | 9.01     | 0.17    | 0.25  | 0.08  | 0.11  |
| N3   | 0.18 | 1.66 | 1.28  | 10.52 | 4.64  | 20.01 | 0.16  | 4.61  | 48.65      | 7.75     | 0.15    | 0.21  | 0.10  | 0.09  |
| N3-K | 0.38 | 4.27 | 3.20  | 28.09 | 10.44 | 9.27  | 0.12  | 3.78  | 35.17      | 5.08     | n.d.    | 0.12  | n.d.  | 0.08  |
| N3-P | n.d. | n.d. | 0.10  | 0.33  | 0.83  | 30.30 | 0.20  | 4.52  | 53.98      | 9.13     | 0.16    | 0.25  | 0.11  | 0.10  |
| N2   | 0.18 | 1.74 | 1.33  | 10.78 | 4.64  | 20.05 | 0.17  | 4.71  | 47.84      | 8.03     | 0.11    | 0.22  | 0.11  | 0.09  |
| N2-K | 0.39 | 4.31 | 3.24  | 28.23 | 10.52 | 9.34  | 0.13  | 3.86  | 34.77      | 5.02     | n.d.    | 0.12  | n.d.  | 0.09  |
| N2-P | n.d. | n.d. | 0.10  | 0.34  | 0.87  | 31.94 | 0.19  | 4.75  | 52.30      | 8.90     | 0.16    | 0.26  | 0.10  | 0.09  |
| N1-K | 0.32 | 3.59 | 2.67  | 23.79 | 9.50  | 9.54  | 0.13  | 4.49  | 39.87      | 5.85     | n.d.    | 0.16  | n.d.  | 0.10  |
| N1   | 0.16 | 1.46 | 1.12  | 9.47  | 4.37  | 19.88 | 0.17  | 5.02  | 49.42      | 8.36     | 0.13    | 0.24  | 0.11  | 0.10  |
| N1-P | n.d. | n.d. | 0.10  | 0.35  | 0.82  | 28.96 | 0.21  | 4.57  | 54.99      | 9.36     | 0.19    | 0.25  | 0.10  | 0.11  |

n.d., not detected.

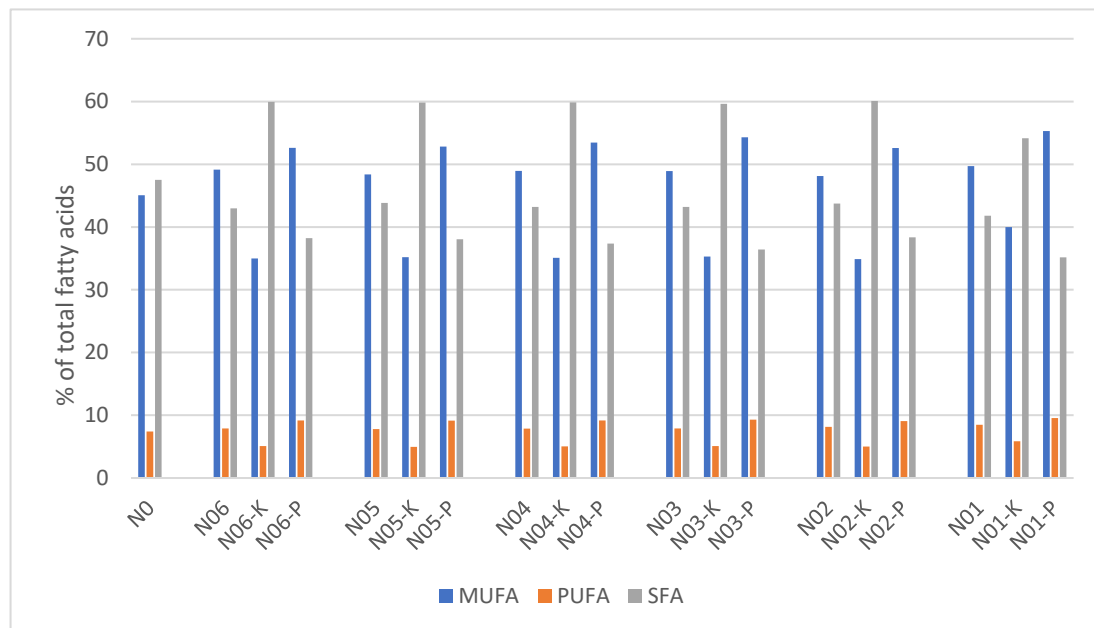


**Figure 1.** Ratio of mono-(MUFA), poly-(PUFA), and saturated (SFA) fatty acids in sweet spreads with different content of cocoa shell (marked with numbers 0–6) and coconut (K) and palm (P) oil.

The relative ratio of mono-, poly-, and saturated fats in sweet spreads is shown in Figure 1. High contents of both monounsaturated and saturated fatty acids are visible from the graph in all analyzed samples.

To further evaluate the nutritional improvement of the sweet spreads, dietary fiber and protein contents were determined. Xylitol did not have an effect on the dietary profile (Table 3), but an increase in cocoa shell content resulted in a significant increase in insoluble and soluble dietary fiber content, with approximately 5.5 times higher values of TDF in the samples with the highest proportion of cocoa shell. Additionally, protein content also increased proportionally with the addition of cocoa shell (Table 3), with approximately 1.7 times higher values determined at the highest proportion of cocoa shell compared to the control sample. Taking into consideration that, in the samples with cocoa shell, sucrose was eliminated from the recipe, we claim that the nutritional profile of the sweet spreads was indeed improved. Soares et al. [28] reported that they have nutritionally improved cookies with the addition of cocoa shell, enriching them with fiber and bioactive compounds, and Sanchez et al. [29] evaluated cocoa bean shell as a functional food ingredient.

The largest density (Table 4) among investigated cocoa spreads was observed for the null sample (N0), in which a standard recipe for cocoa spread production was used, followed by samples produced with the use of coconut oil as a single substitute fat (N1-K–N6-K), samples with a combination of palm and coconut oil (N2–N6), and sample N1-P (containing the highest proportion of cocoa shell and palm oil as a single substitute fat) with the lowest density. Although a statistically significant difference was observed, the range of density values was 1.18–1.26 g/cm<sup>3</sup>, with the majority of samples being in the same statistical category. In addition, according to the results shown in Table 4, density correlated only with fat type (palm and coconut oil), indicating that melting of the fat and distribution of non-fat solid particles within the fat phase influence the density more significantly than the particle size distribution. The largest density of the control sample (N0) might be a result of the presence of sucrose, since Bonarius et al. [30] reported that sucrose had a significantly higher density compared to fibrous food industry by-products (grape pomace, pecan fiber, lemon peel, and spent grain).

**Table 3.** Dietary fiber (IDF, insoluble, SDF, soluble, TDF, and total dietary fiber) and protein contents of sweet spreads.

|      | IDF                         | SDF                        | TDF                         | Protein                      |
|------|-----------------------------|----------------------------|-----------------------------|------------------------------|
| N0   | 2.688 ± 0.037 <sup>a</sup>  | 0.730 ± 0.074 <sup>a</sup> | 3.418 ± 0.037 <sup>a</sup>  | 5.944 ± 0.190 <sup>a,b</sup> |
| N6   | 2.433 ± 0.083 <sup>a</sup>  | 0.833 ± 0.125 <sup>a</sup> | 3.267 ± 0.042 <sup>a</sup>  | 6.165 ± 0.118 <sup>a</sup>   |
| N6-K | 2.690 ± 0.175 <sup>a</sup>  | 0.713 ± 0.008 <sup>a</sup> | 3.403 ± 0.166 <sup>a</sup>  | 4.665 ± 0.067 <sup>a</sup>   |
| N6-P | 2.294 ± 0.245 <sup>a</sup>  | 0.774 ± 0.162 <sup>a</sup> | 3.068 ± 0.083 <sup>a</sup>  | 5.344 ± 0.081 <sup>a</sup>   |
| N5   | 4.990 ± 0.279 <sup>b</sup>  | 1.831 ± 0.141 <sup>b</sup> | 6.820 ± 0.137 <sup>b</sup>  | 6.990 ± 0.280 <sup>b</sup>   |
| N5-K | 5.251 ± 0.121 <sup>b</sup>  | 1.559 ± 0.141 <sup>b</sup> | 6.810 ± 0.021 <sup>b</sup>  | 6.504 ± 0.045 <sup>b</sup>   |
| N5-P | 5.265 ± 0.433 <sup>b</sup>  | 1.672 ± 0.137 <sup>b</sup> | 6.937 ± 0.570 <sup>b</sup>  | 6.863 ± 0.289 <sup>b</sup>   |
| N4   | 7.657 ± 0.187 <sup>c</sup>  | 2.449 ± 0.067 <sup>c</sup> | 10.106 ± 0.121 <sup>c</sup> | 7.983 ± 0.150 <sup>b</sup>   |
| N4-K | 7.763 ± 0.154 <sup>c</sup>  | 2.058 ± 0.137 <sup>c</sup> | 9.820 ± 0.017 <sup>c</sup>  | 7.488 ± 0.100 <sup>b</sup>   |
| N4-P | 7.735 ± 0.017 <sup>c</sup>  | 2.498 ± 0.062 <sup>c</sup> | 10.233 ± 0.046 <sup>c</sup> | 7.268 ± 0.290 <sup>b</sup>   |
| N3   | 10.526 ± 0.428 <sup>d</sup> | 3.073 ± 0.062 <sup>d</sup> | 13.600 ± 0.366 <sup>d</sup> | 8.745 ± 0.153 <sup>c</sup>   |
| N3-K | 11.155 ± 1.298 <sup>d</sup> | 2.932 ± 0.424 <sup>d</sup> | 14.087 ± 0.873 <sup>d</sup> | 7.840 ± 0.253 <sup>c</sup>   |
| N3-P | 10.904 ± 0.653 <sup>d</sup> | 3.076 ± 0.229 <sup>d</sup> | 13.981 ± 0.882 <sup>d</sup> | 8.889 ± 0.336 <sup>c</sup>   |
| N2   | 13.107 ± 0.977 <sup>e</sup> | 3.606 ± 0.412 <sup>e</sup> | 16.713 ± 0.566 <sup>e</sup> | 8.921 ± 0.180 <sup>c</sup>   |
| N2-K | 12.815 ± 0.711 <sup>e</sup> | 3.383 ± 0.037 <sup>e</sup> | 16.198 ± 0.749 <sup>e</sup> | 8.542 ± 0.794 <sup>c</sup>   |
| N2-P | 13.025 ± 0.736 <sup>e</sup> | 3.514 ± 0.075 <sup>e</sup> | 16.539 ± 0.811 <sup>e</sup> | 9.363 ± 0.120 <sup>d</sup>   |
| N1   | 14.149 ± 0.611 <sup>f</sup> | 4.225 ± 0.383 <sup>f</sup> | 18.375 ± 0.229 <sup>f</sup> | 10.243 ± 0.644 <sup>d</sup>  |
| N1-K | 16.013 ± 0.911 <sup>f</sup> | 4.106 ± 0.616 <sup>f</sup> | 20.119 ± 0.295 <sup>f</sup> | 9.413 ± 0.610 <sup>d</sup>   |
| N1-P | 14.962 ± 0.354 <sup>f</sup> | 4.375 ± 0.379 <sup>f</sup> | 19.337 ± 0.025 <sup>f</sup> | 10.297 ± 0.053 <sup>d</sup>  |

Samples with different superscripts in the same column are statistically different ( $p < 0.05$ ).

A similar result was observed for the colloidal stability, which was in the range 98.66–99.76 (Table 4), showing that all the investigated samples were highly stable and resistant to oil leakage on the surface of the product during storage. According to Babin [31], the stability of the colloidal system (in which particles are dispersed in a continuous solvent, with particles being larger than solvent molecules) is largely determined by the properties of the continuous phase. Our research shows a correlation between colloidal stability and fat type; however, it is not strong. This is probably because colloidal stability also depends on the interactions between particles, e.g., van der Waals' attraction forces, steric forces, hydrogen bonding, and hydrophobic interactions. The intensity of the mentioned interactions determines the tendency of the solid particles to aggregate and destabilize the colloidal system [31]. A cocoa spread is a complex system, with different types and shapes of solid particles, which may interact in many different ways, and it is hard to establish an evident correlation between certain characteristics.

The texture of sweet spreads is an important sensory and physical property. It directly influences the ease of pumping the spread during manipulation in the factory, spreadability on the bread/pancakes/waffles, etc., and mouthfeel [32]. In addition to unique sweetness and flavor properties, sugar acts as a texturizing agent in sweet spreads [18]. Introduction of raw materials rich in fiber increases the firmness of cocoa spreads due to increased water-binding capacity and larger agglomeration of particles, an increase in total solids content, and the interaction of fiber-containing particles with milk proteins [17,19]. Sugar replacement with cocoa shell and sweeteners had a variable influence on spreadability and firmness, depending on the type of fat used in the recipe. Samples produced with the palm oil as the single substitute fat had the highest firmness (0.237–0.618 N) and the highest

resistance to spreading (1570.997–4936.179 gs), followed by samples with a combination of palm and coconut oil and the samples with coconut oil as a single substitution fat. Tarakci and Yildirim [33] also reported the highest firmness of spreads with palm fat compared to olive oil and cocoa butter ones. Melting properties of fats (palm oil melts at 33–39 °C, and coconut oil at 24–25 °C) evidently influence the texture. However, unlike expected, there was no observed trend between the proportion of the cocoa shell and these parameters. The sample N5-K, with approximately 5% of cocoa shell and coconut oil, had the lowest firmness (0.028 N) and the best spreadability (170.040 gs), whereas the sample with the same proportion of cocoa shell but with palm oil had the highest values (0.618 N and 4936.179 gs, respectively). The firmness of chocolate spreads produced with hazelnuts roasted in different conditions in the research of Aydemir, Sattari, and Kucuk [34] ranged between 972 and 1242 g, with spreadability ranging from 907 to 1171 gs, which are closest to our samples produced with a combination of palm and coconut oil. The research of Berk et al. [18] and Bucker et al. [19] reported that the samples with the highest values of firmness had the highest values for spreadability, supporting the findings of our research.

**Table 4.** Density, colloidal stability, and texture of cocoa spreads produced with the substitution of sugar with cocoa shell, xylitol, and stevia, and the addition of palm and coconut oil.

|      | Density * (g/cm <sup>3</sup> ) | Colloidal Stability *       | Firmness * (N)               | Spreadability * (gs)            |
|------|--------------------------------|-----------------------------|------------------------------|---------------------------------|
| N0   | 1.266 ± 0.009 <sup>c</sup>     | 99.43 ± 0.04 <sup>b,c</sup> | 0.179 ± 0.004 <sup>d</sup>   | 1180.430 ± 26.851 <sup>c</sup>  |
| N6   | 1.216 ± 0.002 <sup>b</sup>     | 99.30 ± 0.04 <sup>b,c</sup> | 0.047 ± 0.001 <sup>a,b</sup> | 334.672 ± 20.775 <sup>a,b</sup> |
| N6-K | 1.228 ± 0.006 <sup>b</sup>     | 99.16 ± 0.04 <sup>b</sup>   | 0.052 ± 0.001 <sup>a,b</sup> | 277.083 ± 6.610 <sup>a,b</sup>  |
| N6-P | 1.197 ± 0.008 <sup>a,b</sup>   | 99.60 ± 0.18 <sup>c</sup>   | 0.485 ± 0.026 <sup>g</sup>   | 3525.037 ± 199.071 <sup>f</sup> |
| N5   | 1.213 ± 0.004 <sup>b</sup>     | 99.33 ± 0.00 <sup>b,c</sup> | 0.072 ± 0.002 <sup>b</sup>   | 486.778 ± 19.462 <sup>a,b</sup> |
| N5-K | 1.234 ± 0.010 <sup>b</sup>     | 98.73 ± 0.37 <sup>a,b</sup> | 0.028 ± 0.000 <sup>a</sup>   | 170.040 ± 5.269 <sup>a</sup>    |
| N5-P | 1.197 ± 0.008 <sup>a,b</sup>   | 99.76 ± 0.23 <sup>c</sup>   | 0.618 ± 0.069 <sup>h</sup>   | 4936.179 ± 804.804 <sup>g</sup> |
| N4   | 1.219 ± 0.001 <sup>b</sup>     | 99.10 ± 0.04 <sup>b</sup>   | 0.053 ± 0.001 <sup>a,b</sup> | 343.278 ± 9.859 <sup>a,b</sup>  |
| N4-K | 1.240 ± 0.012 <sup>b,c</sup>   | 99.16 ± 0.23 <sup>b</sup>   | 0.032 ± 0.000 <sup>a,b</sup> | 193.708 ± 18.828 <sup>a</sup>   |
| N4-P | 1.199 ± 0.047 <sup>a,b</sup>   | 99.29 ± 0.06 <sup>b,c</sup> | 0.208 ± 0.008 <sup>d</sup>   | 1570.997 ± 62.200 <sup>d</sup>  |
| N3   | 1.206 ± 0.007 <sup>a,b</sup>   | 99.40 ± 0.18 <sup>b,c</sup> | 0.090 ± 0.002 <sup>c</sup>   | 654.015 ± 52.566 <sup>b</sup>   |
| N3-K | 1.236 ± 0.005 <sup>b</sup>     | 99.20 ± 0.09 <sup>b</sup>   | 0.036 ± 0.000 <sup>a,b</sup> | 214.096 ± 7.883 <sup>a</sup>    |
| N3-P | 1.222 ± 0.014 <sup>b</sup>     | 99.26 ± 0.18 <sup>b,c</sup> | 0.237 ± 0.019 <sup>e</sup>   | 1732.212 ± 260.711 <sup>d</sup> |
| N2   | 1.216 ± 0.009 <sup>b</sup>     | 99.30 ± 0.23 <sup>b,c</sup> | 0.093 ± 0.002 <sup>b,c</sup> | 672.704 ± 59.258 <sup>b</sup>   |
| N2-K | 1.234 ± 0.003 <sup>b</sup>     | 98.66 ± 0.09 <sup>a</sup>   | 0.062 ± 0.001 <sup>b</sup>   | 385.270 ± 28.663 <sup>a,b</sup> |
| N2-P | 1.217 ± 0.016 <sup>b</sup>     | 99.63 ± 0.14 <sup>c</sup>   | 0.375 ± 0.023 <sup>e</sup>   | 2996.405 ± 275.432 <sup>e</sup> |
| N1   | 1.180 ± 0.016 <sup>a</sup>     | 99.16 ± 0.14 <sup>b</sup>   | 0.112 ± 0.002 <sup>c</sup>   | 837.310 ± 55.501 <sup>b,c</sup> |
| N1-K | 1.224 ± 0.022 <sup>b</sup>     | 99.03 ± 0.14 <sup>a,b</sup> | 0.059 ± 0.001 <sup>a,b</sup> | 369.267 ± 27.574 <sup>a,b</sup> |
| N1-P | 1.180 ± 0.037 <sup>a</sup>     | 99.06 ± 0.37 <sup>b</sup>   | 0.419 ± 0.080 <sup>f</sup>   | 3753.872 ± 920.380 <sup>f</sup> |

\* Mean values ± SD of three replicates are shown. Samples with different superscripts in the same column are statistically different ( $p < 0.05$ ).

Yield stress is the minimum force needed to set the mass in motion [32]. In the present research, it was highly affected by the recipe (Table 5). The null sample (N0), produced according to the standard recipe, had the lowest value of yield stress (6.385 Pa), followed by samples with coconut oil, a mixture of coconut and palm oil, and finally samples with palm oil. The substitution of sugar with xylitol (N6) resulted in a four-fold increase

in yield stress, similar to the addition of cocoa shell in the highest proportion (N1). The smallest increase in the yield stress was observed for the sample N2-K (10.69 Pa), which was produced with approximately 20% of cocoa shell and coconut oil. Unlike N0, which had the smallest particles, with 90% of the particles smaller than 26.205  $\mu\text{m}$  (Table 5), N2-K had the fourth largest particles, with 90% of the particles below 53.780  $\mu\text{m}$ . Sample N1-P, with the highest proportion of cocoa shell (approximately 25%) and palm oil, had the highest yield stress (51.43 Pa) and the second largest particles, with 90% of the particles smaller than 56.893  $\mu\text{m}$ . Nevertheless, the increase in yield stress is not proportional to the increase in the cocoa shell content (Table 1) or to the particle size distribution (Table 5, Figure 2), which is visible from the correlation coefficients shown in Table 6. Principato et al. [32] also found differences in the yield stress of commercial cocoa spreads, but these could not be linked to differences in fat content or the particle size distribution. Yield stress is not only influenced by particle size but also by particle shape and number. Namely, in order for the sweet spread to start flowing, all particles must align in the flow direction. Irregularly shaped particles will align more difficultly, but a larger number of smaller particles will flow more difficultly as well, due to the larger surface area that needs to be covered by fat [35].

**Table 5.** Rheological properties and particle size parameters of cocoa spreads produced with the substitution of sugar with cocoa shell, xylitol, and stevia, and the addition of palm and coconut oil.

|      | Casson Yield Stress (Pa) | Casson Plastic Viscosity (Pas) | Particle Size Parameters ( $\mu\text{m}$ ) |         |         |          |
|------|--------------------------|--------------------------------|--------------------------------------------|---------|---------|----------|
|      |                          |                                | d (0.1)                                    | d (0.5) | d (0.9) | D [4, 3] |
| N0   | 6.385 <sup>a</sup>       | 3.708 <sup>i</sup>             | 3.264                                      | 9.761   | 26.205  | 12.712   |
| N6   | 26.010 <sup>k</sup>      | 2.792 <sup>b</sup>             | 2.986                                      | 12.123  | 35.306  | 16.228   |
| N6-K | 13.220 <sup>d</sup>      | 1.833 <sup>a</sup>             | 3.407                                      | 10.928  | 32.997  | 16.034   |
| N6-P | 46.360 <sup>q</sup>      | 2.934 <sup>d</sup>             | 3.168                                      | 10.367  | 30.514  | 14.185   |
| N5   | 22.210 <sup>i</sup>      | 3.002 <sup>e</sup>             | 1.026                                      | 11.104  | 64.800  | 22.186   |
| N5-K | 13.680 <sup>e</sup>      | 2.865 <sup>c</sup>             | 3.596                                      | 11.535  | 36.155  | 26.833   |
| N5-P | 36.750 <sup>n</sup>      | 3.378 <sup>f</sup>             | 2.998                                      | 10.685  | 33.165  | 15.043   |
| N4   | 20.580 <sup>g</sup>      | 3.847 <sup>j</sup>             | 3.098                                      | 11.568  | 39.533  | 21.480   |
| N4-K | 13.020 <sup>c</sup>      | 3.452 <sup>g</sup>             | 3.792                                      | 12.258  | 34.226  | 16.171   |
| N4-P | 35.680 <sup>m</sup>      | 4.166 <sup>k</sup>             | 3.459                                      | 12.467  | 39.397  | 17.722   |
| N3   | 18.280 <sup>f</sup>      | 18.280 <sup>s</sup>            | 3.529                                      | 12.705  | 40.767  | 18.227   |
| N3-K | 13.220 <sup>d</sup>      | 3.515 <sup>h</sup>             | 3.650                                      | 13.233  | 43.448  | 19.737   |
| N3-P | 42.760 <sup>p</sup>      | 7.582 <sup>q</sup>             | 3.844                                      | 16.280  | 55.723  | 23.919   |
| N2   | 24.010 <sup>j</sup>      | 4.447 <sup>l</sup>             | 3.981                                      | 15.209  | 50.688  | 22.238   |
| N2-K | 10.690 <sup>b</sup>      | 7.224 <sup>o</sup>             | 3.597                                      | 15.505  | 53.780  | 23.012   |
| N2-P | 37.750 <sup>o</sup>      | 7.032 <sup>n</sup>             | 4.029                                      | 16.449  | 53.346  | 23.431   |
| N1   | 26.680 <sup>l</sup>      | 7.380 <sup>p</sup>             | 4.529                                      | 16.182  | 51.532  | 22.902   |
| N1-K | 20.830 <sup>h</sup>      | 6.381 <sup>m</sup>             | 4.414                                      | 16.640  | 51.976  | 23.164   |
| N1-P | 51.430 <sup>r</sup>      | 7.909 <sup>r</sup>             | 4.091                                      | 15.824  | 56.893  | 24.912   |

Samples with different superscripts in the same column are statistically different ( $p < 0.05$ ).

Plastic viscosity was lowest for the samples with xylitol without the cocoa shell and with the lowest proportion of cocoa shell (Table 5). Although cocoa shell content positively correlates to plastic viscosity (Table 6), the correlation coefficient is approximately 0.5, so the correlation is not marked. This is reflected in plastic viscosity values for the samples with

higher proportions of cocoa shell, with no linear connection between cocoa shell content and plastic viscosity values. The plastic viscosity is largely influenced by the proportion of large particles, which may cause an increase in viscosity after the initial flow of the spreads. Viscosity increase during shearing may be observed from the flow curves shown in Figure 2. The slope of the curves reflects the volume mean diameter  $D_{[4,3]}$  shown in Table 5: the larger the  $D_{[4,3]}$  values, the steeper the slope, indicating a larger rise in the viscosity.

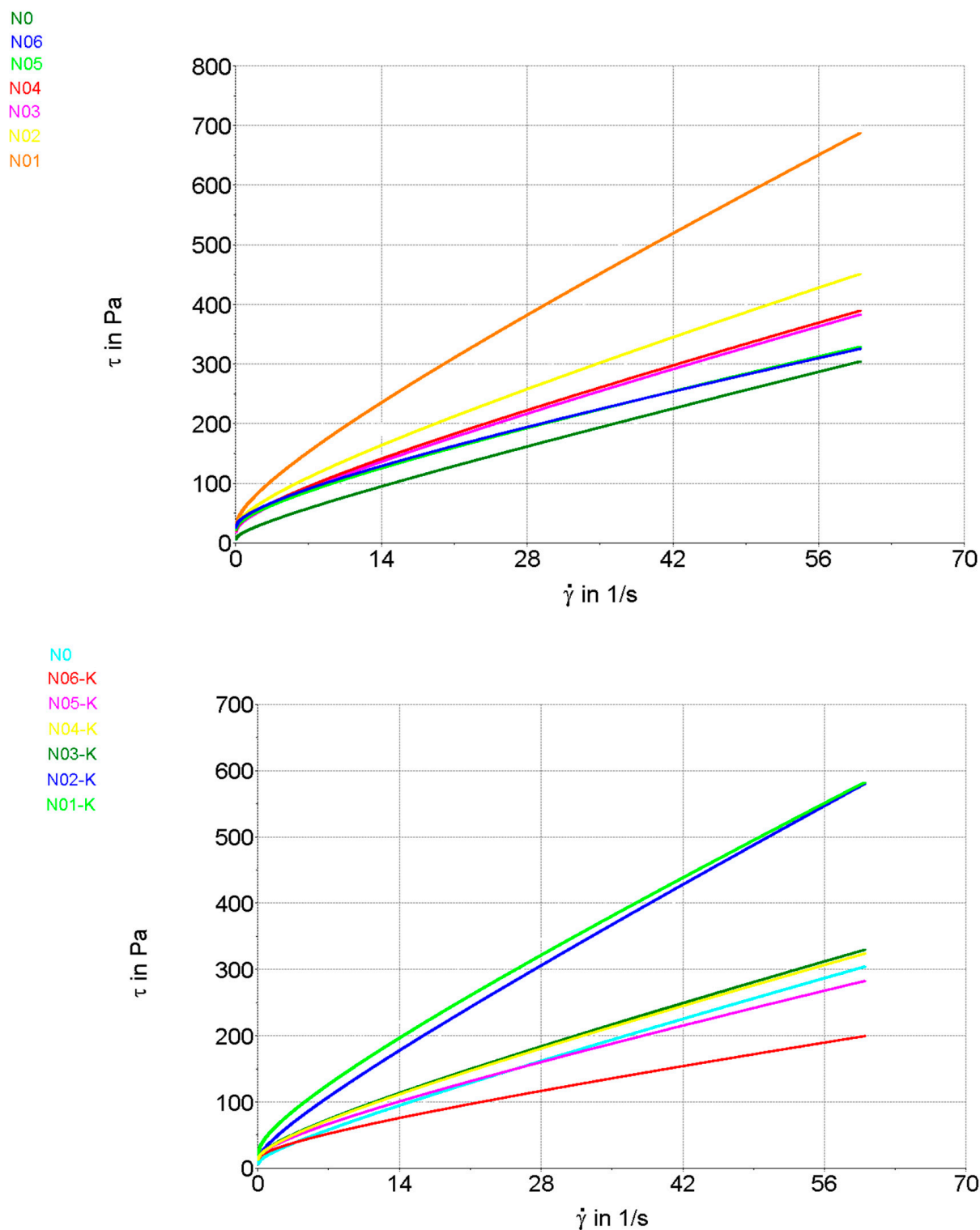


Figure 2. Cont.

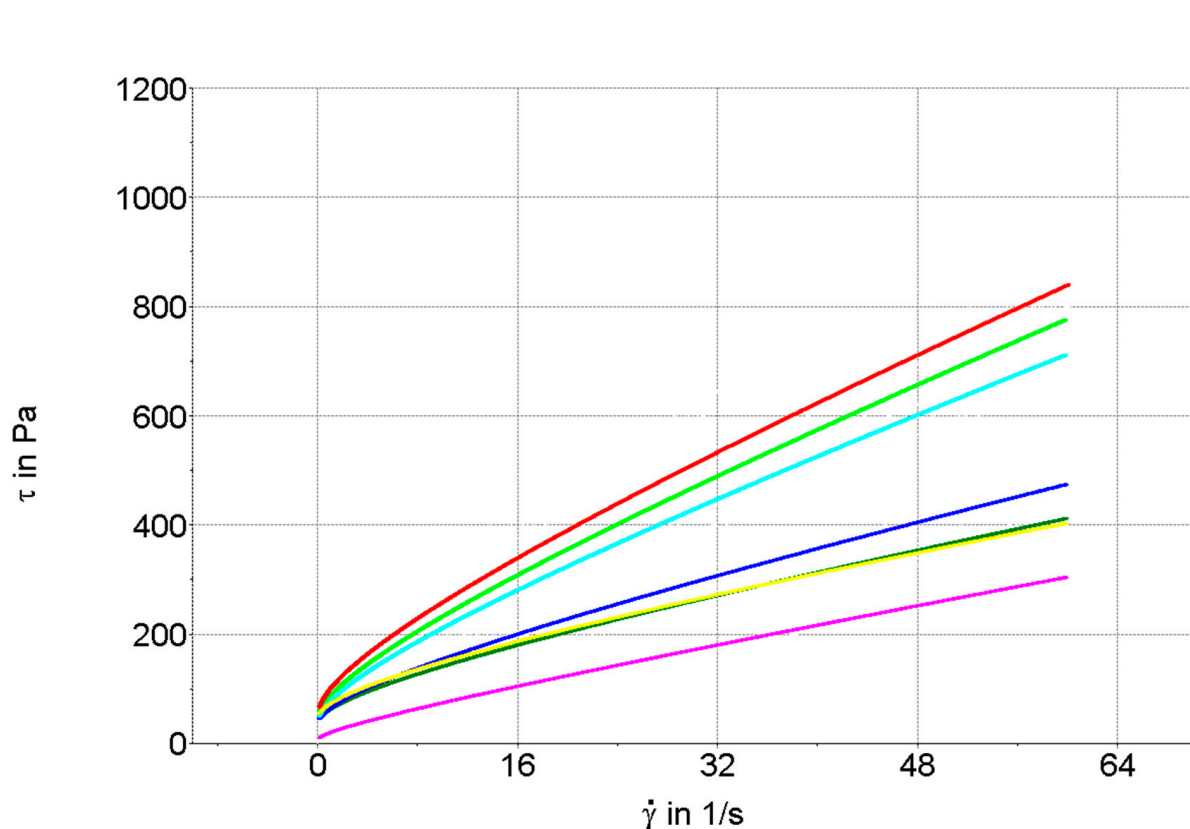


**Figure 2.** Flow curves of cocoa spreads produced with the substitution of sugar with cocoa shell, xylitol, and stevia, and the addition of palm and coconut oil.

Torregrossa et al. [36] reported that chocolates with high levels of sugar substitutes have higher yield stress, apparent and Casson viscosity, which highly corresponds to our samples produced with stevia and cocoa shell. Rezende et al. [37] reported an increase in plastic viscosity of chocolates with an increase in inulin content, and Nebesny and Zyzelewicz [38] reported reduced Casson yield stress in chocolates produced with isomalt compared to sucrose-containing chocolates, which is consistent with the results obtained for the samples with xylitol (series marked with N6). Polyols might have a similar effect to PGPR, which is a polyol emulsifier, and the same effect was reported for inulin [39]. Merachli et al. [40] reported that cocoa fibers on droplet surfaces in chocolate ganaches induce interfacial roughness, resulting in their restricted mobility and higher viscosity, while Bonarius et al. [30] ascribed higher plastic viscosity values of model suspensions of food industry by-products in fat to higher hydrodynamic forces between their particles compared to sugar. They also stated that fibrous materials absorb part of the oil by swelling, which in turn increases their size and volume and decreases free-flowing oil content. Although a similar effect on Casson plastic viscosity is observed if samples with the same fat composition in the present research are compared (N0–N6; N1–K–N6–K; and N1–P–N6–P), no such effect on Casson yield is visible. Moreover, the increase in cocoa shell content is linked to the drop in Casson yield stress or its retention up to 15.5% of cocoa shell. A decrease in Casson yield stress was reported for cocoa hazelnut spreads in which sugar was replaced with stevia and inulin, without a marked effect on Casson plastic viscosity [18]. Bonarius et al. [30] reported significantly lower yield stress for model suspensions of fibrous food industry by-products in fat compared to sucrose, with less pronounced effect of lecithin on reduction in yield stress. They linked the rheological properties of the model systems to aggregation of particles, showing that sugar particles are more prone to aggregation compared to fibrous food industry by-products, esp. in the presence of small quantities of water.

**Table 6.** Correlation coefficients. Marked correlations are statistically significant at  $p < 0.05$ .

|                         | Palm Oil | Coconut Oil | Xylitol   | Stevia    | Cocoa Shell | Colloidal Stability | Firmness  | Spreadability | Density   | Casson Yield Stress | Cason Plastic Viscosity | d (0.1)  | d (0.5)  | d (0.9)  | D [4, 3] |
|-------------------------|----------|-------------|-----------|-----------|-------------|---------------------|-----------|---------------|-----------|---------------------|-------------------------|----------|----------|----------|----------|
| Palm oil                | 1.00000  |             |           |           |             |                     |           |               |           |                     |                         |          |          |          |          |
| Coconut oil             | −1.00000 | 1.00000     |           |           |             |                     |           |               |           |                     |                         |          |          |          |          |
| Xylitol                 | 0.00000  | 0.00000     | 1.000000  |           |             |                     |           |               |           |                     |                         |          |          |          |          |
| Stevia                  | 0.00000  | 0.00000     | −0.440853 | 1.000000  |             |                     |           |               |           |                     |                         |          |          |          |          |
| Cocoa shell             | 0.00000  | 0.00000     | −0.441864 | 0.999968  | 1.000000    |                     |           |               |           |                     |                         |          |          |          |          |
| Colloidal stability     | 0.57671  | −0.57671    | 0.126079  | −0.329114 | −0.327843   | 1.000000            |           |               |           |                     |                         |          |          |          |          |
| Firmness                | 0.76043  | −0.76043    | 0.076607  | −0.121523 | −0.120081   | 0.678243            | 1.000000  |               |           |                     |                         |          |          |          |          |
| Spreadability           | 0.75462  | −0.75462    | 0.094418  | −0.103210 | −0.101813   | 0.670296            | 0.996943  | 1.000000      |           |                     |                         |          |          |          |          |
| Density                 | −0.56582 | 0.56582     | −0.265544 | −0.333284 | −0.330896   | −0.210109           | −0.295599 | −0.307347     | 1.000000  |                     |                         |          |          |          |          |
| Casson yield stress     | 0.86396  | −0.86396    | 0.185425  | 0.194580  | 0.195030    | 0.353921            | 0.687708  | 0.690214      | −0.684550 | 1.000000            |                         |          |          |          |          |
| Cason plastic viscosity | 0.14267  | −0.14267    | −0.286365 | 0.521904  | 0.523024    | −0.099057           | −0.034964 | −0.020026     | −0.215860 | 0.095239            | 1.000000                |          |          |          |          |
| d (0.1)                 | −0.07925 | 0.07925     | −0.407782 | 0.645803  | 0.645583    | −0.225610           | −0.042437 | −0.042843     | −0.141014 | 0.103918            | 0.314514                | 1.000000 |          |          |          |
| d (0.5)                 | 0.05667  | −0.05667    | −0.348168 | 0.915348  | 0.916373    | −0.259059           | −0.110402 | −0.095930     | −0.344389 | 0.285293            | 0.426572                | 0.659668 | 1.000000 |          |          |
| d (0.9)                 | 0.10356  | −0.10356    | −0.151795 | 0.701980  | 0.703468    | −0.235615           | −0.148611 | −0.128287     | −0.321676 | 0.269373            | 0.300155                | 0.014771 | 0.733418 | 1.000000 |          |
| D [4, 3]                | −0.09534 | 0.09534     | −0.093983 | 0.674859  | 0.675738    | −0.410641           | −0.281731 | −0.255412     | −0.165983 | 0.137097            | 0.229696                | 0.278731 | 0.698209 | 0.765495 | 1.000000 |

Colored values mark high correlation.

The distribution of particle size (Figure 3) is rather broad, which is common, considering that the process was performed in a ball mill, where it is more difficult to achieve uniform particle size, unlike in a five-roller mill [35]. Broader curves for the samples with cocoa shell in Figure 3 show that cocoa shell was harder to grind and contributed to the higher viscosity and inferior texture properties. Additionally,  $d(0.9)$  values significantly increase with the proportion of cocoa shell (Table 5), marking larger contents of coarser particles. When lower quantities of cocoa shell were used, a decrease in  $d(0.9)$  values with the addition of cocoa and palm oil was observed, but at a higher proportion of the shell, the opposite effect was observed. However, the particle size distribution is more uniform than the bimodal distribution in commercial spreads reported by Principato et al. [32] and Lima et al. [41]. Apparently, rotation speed and processing time affect the particle size and morphological properties of the particles [42].

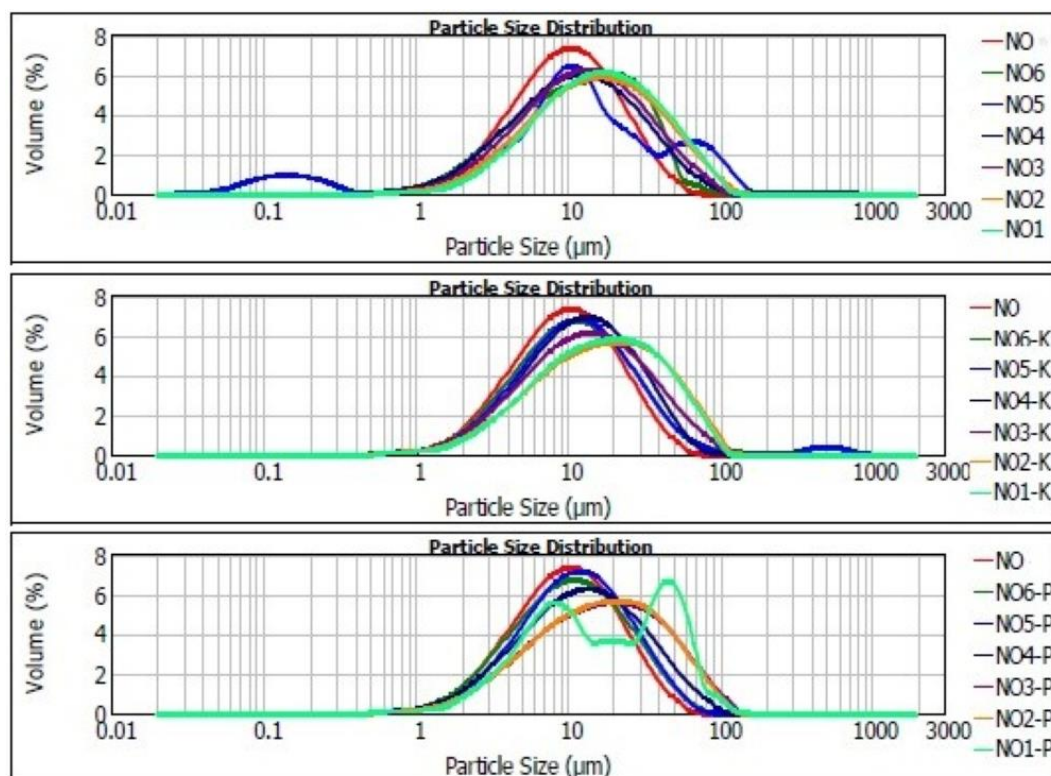


**Figure 3.** Particle size distribution of cocoa spreads produced with the substitution of sugar with cocoa shell, xylitol, and stevia, and the addition of palm and coconut oil.

#### 4. Conclusions

Physical properties of cocoa spreads are important both from the technological and sensory (consumer acceptance) point of view. Any intervention in the recipe, such as the substitution of sucrose with cocoa shell, xylitol, and stevia, results in changes in firmness, spreadability, and flow properties of cocoa spreads. Texture properties (spreadability and firmness) and yield stress of the investigated cocoa spreads are directly correlated with the type of fat used in the recipe, showing lower values for samples produced with coconut fat, regardless of the amount of cocoa shell and sweeteners used in the investigated range. The best texture was observed for the sample with the lowest content of cocoa shell; however, other samples had satisfactory results as well. The results of the study showed that protein and fiber content increased proportionally with the added cocoa shell, proving that nutritional improvement was achieved under the experimental conditions investigated. By careful adjustment of ingredients and the right choice of the substitute fat,

sustainable, nutritionally improved cocoa spreads with satisfactory texture and rheology may be produced. Further research is needed to examine fat migration, crystallization over time, and texture changes during storage, as well as to test consumer acceptance and optimize recipes according to newly gathered results.

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