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Textural, rheological, melting properties, particle size distribution, and NMR relaxometry of cocoa hazelnut spread with inulin-stevia addition as sugar replacer

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Abstract

This study investigated the influence of substituting 60, 80, and 100% of the sugar in traditional cocoa hazelnut paste (control) formulation with inulin-stevia (90:10, w/w) mixture on textural and rheological characteristics, melting behavior, water activity (a_w), particle size distribution (PSD), and color. Textural, rheological, melting properties, and color of samples were analyzed after 1, 2, and 3 months of storage at 11°C. Nuclear magnetic resonance (NMR) relaxometry experiments were also performed to understand the interaction of new ingredients with oil. Replacement of sugar with inulin-stevia gave darker color, reduced Casson yield stress, and changed the textural parameters and melting profile of the samples depending on the level but did not create a remarkable effect on PSD and Casson plastic viscosity. Increasing inulin-stevia content yielded lower a_w and higher T_{2a} values indicating decreased mobility of water. Complete removal of sugar caused low spreadability. The results showed that an 80% replacement level yielded a product with similar textural parameters and fat-melting mouth feeling compared to control sample. Cocoa hazelnut spreads prepared with inulin and stevia showed good textural stability during storage.

KEYWORDS
DSC, hazelnut spread, inulin, stevia, TD-NMR, texture

1 | INTRODUCTION

Türkiye is the world's largest producer of hazelnut. It contributes over 60% of the world's hazelnut production, followed by Italy (TEPGE, 2021). The consumption of hazelnut may provide potential health benefits (Brown et al., 2022) since it is a rich dietary source of protein, fiber, oleic acid, α-tocopherol, β-sitosterol, and vitamin E (Karaosmanoglu, 2022). Hazelnut products are used as a side ingredient by food and beverage manufacturers for making various products like chocolate, biscuits, pastries, ice cream, etc. The chocolate industry is the leading customer of hazelnut for the production of different chocolate base confectionery products. Cocoa hazelnut spread is an emulsion having hazelnut puree, cocoa powder, and sugar as the main ingredients dispersed in a continuous fat phase (Aydemir, 2019).

Many consumers use cocoa hazelnut spread especially as a part of their breakfast. Although, it is a favorite snack for people of all ages, the consumers' willingness to consume cocoa hazelnut spread is negatively affected by its relatively high fat and sugar content. A diet high in fat and sugar has been linked to obesity (Rasool et al., 2018). Numerous systematic reviews and meta-analyses to date have found a positive association between consumption of excess sugar and risk for type 2 diabetes, cardiovascular diseases, cognitive impairments, and dental caries (Xie et al., 2024; Gillespie et al., 2023; Hancock et al., 2020; Stanhope, 2016). Growing evidence for the deleterious

role of excessive sugar consumption in human health prompted introduction of sugar guidelines by the WHO. In the guideline about sugar intake for adults and children by World Health Organization, it is strongly recommended to reduce the sugar intake below 10% of total energy intake (WHO, 2015). Walton et al. (2023) reported that overall intakes of free or added sugars remain above recommended levels, particularly in children. Following the requirements and growing demands of consumers for "low sugar" and "sugar-free" products, researchers and manufacturers have been looking for alternative ways of partial or complete replacement of sugar in these types of products without altering their sensory properties. Both flavor and texture attributes influence the consumer acceptability of hazelnut spread (Di Monaco et al., 2008) and these sensory attributes are affected by the sugar content.

Sucrose is the conventional sweetening agent used in chocolate spread. To replace sucrose in the formulation of chocolates, high potency sweeteners (aspartame, sucralose, stevia, etc.), sugar alcohols (xylitol, sorbitol, mannitol, and lactitol, maltitol, etc.), natural raw materials (yacon, agave syrup, palm sugar, etc.), low digestibility carbohydrates (inulin, maltodextrin, etc.), and sweet proteins (lactose-free milk protein, thaumatin) are being used alone or in combination (Aidoo et al., 2013; Selvasekaran & Chidambaram, 2021). Maltitol is widely used in commercial sugar-free chocolate bars. Sugar-free chocolates sweetened with stevia are also commercially available. Stevia is a non-nutritive sweetener extracted from the leaves of the stevia plant. Several studies have reported the various health benefits of stevia (Ahmad et al., 2020). Stevia is considered to be a healthier alternative to sugar and other artificial sweeteners and is utilized as a sucrose replacer in the food and beverage industry in various countries (Ozcan et al., 2021; Schiatti-Sisó et al., 2022).

Inulin belongs to the fructan group of polysaccharides. It is water soluble and naturally present in many edible plants. Chicory roots (*Cichorium intybus* L.) and Jerusalem artichoke tubers (*Helianthus tuberosus*) are the major sources used to obtain commercial inulin. It is a water-soluble dietary fiber having prebiotic properties and can act as a good bulking agent (Esmaeilnejad Moghadam et al., 2019; Hughes et al., 2021). It was shown in a study that the combined use of inulin and stevia yielded improvement in the growth and activity of probiotic bacteria (Ozcan et al., 2021). Inulin is also used in the production of various types of food products as fat and sugar replacers and texture modifiers (Jackson et al., 2022; Mudannayake et al., 2022; Ozcan & Eroglu, 2022). Inulin has already been used in chocolate formulations by several researchers. Shah et al., (2010) developed sucrose-free chocolate containing stevia as a sweetener and inulin and maltodextrin as bulking agents. Konar et al., (2018) studied the influence of inulin with different degrees of polymerization on some quality attributes of sucrose or maltitol-containing chocolates. Some researchers investigated the influence of different sugar substitutes (isomalt, maltitol, stevia, and thaumatin) on the physical and rheological properties of inulin-containing chocolates (Konar, 2013; Aidoo et al., 2015). There are also studies investigating some quality characteristics of sugar-free chocolates produced by varying proportions of inulin and polydextrose (Aidoo et al., 2014), inulin, and D-tagatose (Shourideh et al., 2012).

Although there are various studies performed on chocolate, it is seen that the studies on the development of reduced sugar or sugar-free cocoa hazelnut spread formulations are quite limited. In a study, Ermis and Özkan (2021) used spray-dried whole sugar beet root powder instead of sucrose in cocoa hazelnut spread and obtained good overall acceptability. Tolve et al. (2021) developed a chocolate spread fortified with micronutrients using inulin and maltitol as sugar replacers. The utilization of dried apple pomace (Büker et al., 2021) and red grape pomace (Acan et al., 2021) as a partial replacement of sugar in chocolate spread formulations has also been searched.

Dietary habits across the globe are evolving. Consumer demand for healthy foods is gradually increasing. Cocoa hazelnut spread is a favorite breakfast meal, especially for children, but it contributes to high sugar intake. The development of cocoa hazelnut spread with reduced calories, and improved health benefits will drag the health-conscious consumers' focus toward this product. To our knowledge, no studies have been conducted on the use of different combinations of stevia and inulin to replace sucrose in cocoa hazelnut spread formulation.

In the present study, it was aimed to investigate the effects of the use of stevia and inulin mixtures as sucrose replacers on textural and rheological characteristics, melting behavior, water activity (a_w), particle size distribution (PSD), and color of cocoa hazelnut spread. Nuclear magnetic resonance (NMR) relaxometry was used to study the interaction of these new ingredients with the water and oil present in the matrix. Another objective was to evaluate the textural stability of cocoa hazelnut spreads prepared with inulin and stevia during storage at 11°C for 3 months.

2 | MATERIALS AND METHODS

2.1 | Materials

Skim milk powder and cocoa powder were purchased from Enka (Konya, Türkiye) and Altınmarka (İstanbul, Türkiye) companies, respectively. Powder sugar, lecithin, palm oil (Esiflow, Felda Iffco, İzmir, Türkiye), whey powder, hazelnut oil, and hazelnut puree were kindly provided by Karimex Gıda (Ordu, Türkiye). Chicory-originated inulin (Orafti® HIS, Beneo-Orafti, Tienen, Belgium) with a degree of polymerization (DP) ranging between 2 and 60, and stevia (Egepak, İzmir, Türkiye) was used in the formulations.

2.2 | Production of cocoa hazelnut spread formulations

Cocoa hazelnut spread samples were prepared according to the formulations given in Table 1. The control sample (IS0) contained no inulin and no stevia. Oil, powdered sugar, and hazelnut puree were mixed at 40°C for 20 min then inulin, stevia, lecithin, cocoa powder, whey powder, and skim milk powder were added, and mixing continued for 40 min. After that, all ingredients were transferred to a ball mill and milled at 60°C for 1.5 h. Following the milling, the mixture was set at

TABLE 1 Formulations applied for the preparation of cocoa hazelnut spread samples.

	Quantity (g)			
Ingredient	IS0 (control)	IS60	IS80	IS100
Palm oil	20	20	20	20
Hazelnut puree	13	13	13	13
Whey powder	7.1	7.1	7.1	7.1
Cocoa powder	5	5	5	5
Hazelnut oil	5	5	5	5
Skim milk powder	4	4	4	4
Lecithin	0.9	0.9	0.9	0.9
Sucrose	45	18	9	–
Inulin	–	24.3	32.4	40.5
Stevia	–	2.7	3.6	4.5
Total	100	100	100	100

40°C and then tempered at 27–29°C. When the tempering was completed, spread formulations were filled into the jars at 29°C. All production steps were performed in the pilot production plant of Karimex Gıda company.

2.3 | Water activity

Water activity of the samples was measured using a water activity meter (LabStart-aw, Novasina, Lachen, Switzerland). About 1 g of the sample was placed in the instrument cup and aw was measured automatically at 25°C.

2.4 | Nuclear magnetic resonance (NMR) relaxometry

Time-domain nuclear magnetic resonance (TD-NMR) relaxometry experiments were performed using a 0.48 Tesla (^{1}H frequency of 20.34 MHz) NMR system (Spin Track, Resonance Systems GmbH, Kirchheim/Teck, Germany) having 10 mm radiofrequency (RF) coil. Samples were put into the test tubes at 1.5 cm height. To measure the T_2 relaxation time of the samples, a CPMG pulse sequence with the parameters of 1000 ms echo time, 800 echoes, 1000 ms relaxation period, and 16 scans was used. For T_2 data, a 2-component model (Equation 1) was selected and Relax 8 software was used for the curve fitting operation (Resonance Systems GmbH, Kirchheim/Teck, Germany).

$$M = M_{2a}e^{-\frac{t}{T_{2a}}} + M_{2b}e^{-\frac{t}{T_{2b}}} \quad (1)$$

where M is the magnitude of the signal, T_{2a} and T_{2b} are relaxation time values of two different proton populations, and M_{2a} and M_{2b} are relative contributions of those proton pools.

2.5 | Particle size distribution (PSD)

The particle size distribution of the samples was determined using the laser diffraction technique with the Mastersizer 2000 instrument (Malvern, Worcestershire, UK) equipped with Hydro 2000 SM (A). Prior to measurement, the sample (approximately 0.2 g) underwent ultrasonic dispersion in deionized water (with a refractive index of 1.33) at ambient temperature (20 ± 2°C) with a ratio of 1:100 (w/v). The dispersion process continued until achieving an obscuration rate of 10%.

PSD parameters obtained included specific surface area (SSA, $m^2\ g^{-1}$), surface weighted mean diameter ($D_{[3,2]}$, μm) (Equation 2), volume-weighted mean diameter ($D_{[4,3]}$, μm) (Equation 3) and polydispersity (span) (Equation 4) (Malvern, Mastersizer 2000 Ver. 6.01).

$$D_{[3,2]} = \sum n_i d_i^3 / \sum n_i d_i^2 \quad (2)$$

$$D_{[4,3]} = \sum n_i d_i^4 / \sum n_i d_i^3 \quad (3)$$

$$\text{Span} = \frac{[Dv_{0.9} - Dv_{0.1}]}{Dv_{0.5}} \quad (4)$$

where d_i is the geometric mean of diameters (μm) and n_i is the number of particles with diameter d_i. $Dv_{0.1}$, $Dv_{0.5}$, and $Dv_{0.9}$ denote that 10%, 50%, and 90%, respectively, of all particles, have smaller sizes than the given value.

2.6 | Color

Color measurements were conducted to determine the L* (whiteness/darkness), a* (redness/greenness), and b* (yellowness/blueness) values of the samples using a colorimeter (CSM1, PCE Deutschland GmbH, Meschede, Germany). Color analyzes were conducted with 10 replications.

2.7 | Determination of melting profile by using differential scanning calorimeter (DSC)

DSC4000 (Perkin Elmer, MA, USA) was used to see the melting profile of the cocoa hazelnut spread formulations. The flow rate of the N_2 purge gas stream was set to 20 mL min^{-1}. The samples were put into aluminum pans having no hermetical sealing at about 20 mg of sample load. Samples were first cooled to −40°C, then heated to 80°C at the rate of 10°C min^{-1}. To examine the peak area and corresponding melting temperatures, Pyris Manager (Perkin Elmer, MA, USA) was used.

2.8 | Textural analysis

Textural properties of the formulations were measured at 25°C using a texture analyzer (CT3, Brookfield Ametek, USA) with 10 kg of load

cell configuration. A spread test fixture consisting of a set of matched male and female 90° cones was used. Sample was placed into the female cone and the top was leveled. The male cone penetrated into the sample until it reached a depth of 5 mm above the female cone. Test speed and post-test speed were adjusted to 3 mm s^{-1} with the trigger load of 0 N. Texture analyzes were conducted with 6 replications. From the recorded force–time curves, mean values of spreadability (N s) (positive area from the curve), firmness (N) (the maximum positive force required to attain the given deformation), adhesive force (N) (the maximum negative force during withdrawal of probe from sample), and adhesiveness (N s) (negative area from the curve) were evaluated using a MATLAB program (R2021b; MathWorks, Inc., Natick, MA).

2.9 | Rheological analysis

The rheological properties of the formulations were analyzed by using a rheometer (RST-CC Touch Rheometer, Brookfield Ametek, USA), following the testing standards set by the International Confectionery Association (ICA). The test configurations used were a coaxial cylinder (bob in cup) with CCT-25 spindle and DIN 53019 adaptor. Prior to measurement, a sample was transferred into the cylindrical adapter up to the mark (about 17 mL) and heated to 40°C with gentle mixing. Measurements were performed at 40°C from 0.001 s^{-1} to 100 s^{-1} shear rates with the logarithmic increase, waiting at 100 s^{-1} shear rate for 10 s then returning to 0.001 s^{-1} to see the thixotropy. From the results, rheograms were plotted and the data were fitted to the Casson model (Equation 5).

$$\tau^{0.5} = \tau_0^{0.5} + (\eta\dot{\gamma})^{0.5} \quad (5)$$

where τ_0 (Pa) is the Casson yield stress, η (Pa s) is the Casson plastic viscosity, and γ is the shear rate (s^{-1}) (Wells, 2009).

2.10 | Storage stability

Hazelnut spread samples were stored at 11°C for 3 months. Color, textural and rheological properties, and DSC thermograms of the formulations were determined each month during storage.

2.11 | Statistical analysis

Unless otherwise specified, all analyzes were conducted with 3 replications. The results were analyzed by analysis of variance (ANOVA) followed by Tukey's comparison test at 95% confidence interval (Version 21, Minitab Inc., Coventry, UK). Pearson correlation test was applied to T_{2a} and η values with 8 observations using Minitab (Version 21, Minitab Inc., Coventry, UK).

3 | RESULTS AND DISCUSSION

3.1 | Water activity

The water activity of the cocoa hazelnut spread is an important factor in terms of stability. The water activity of IS0 (control), IS60, IS80, and IS100 samples was 0.31, 0.30, 0.28, and 0.27, respectively. The a_w value of all samples was low enough (a_w <0.4) to maintain the product stability. Replacement of sucrose with inulin and stevia decreased the a_w values. However, only a_w of the control and IS100 samples were significantly different from each other ($p < 0.05$). This was probably due to the differences in the water-binding capacities of sucrose and inulin. The presence of hydrophilic groups in the amorphous structure of inulin might have increased hygroscopicity (Van de Walle et al., 2018; Selvasekaran & Chidambaram, 2021). Due to the strong water interaction of inulin, the availability of the water in the formulation decreased, and a_w was depressed (Wen et al., 2020). The findings of this study also agree with the one observed by Konar et al., (2018) where a reduction in a_w value of chocolate was reported when sucrose was replaced with inulin. Similarly, Shourideh et al. (2012) detected higher moisture content but lower a_w values for the chocolate samples prepared with inulin instead of sucrose.

3.2 | Nuclear magnetic resonance (NMR) relaxometry

T_2 relaxation time values of the samples are given in Table 2. Relaxation decay curves were expressed by a 3-component model. The contribution of each component to the signal is represented by M_{2a}, M_{2b}, and M_{2c}. Samples containing inulin/stevia in their formulations had longer relaxation time values compared to the control sample.

TABLE 2 T_2 relaxation time values of the cocoa hazelnut spread samples.

Formulation	T_{2a}, ms	T_{2b}, ms	T_{2c}, ms	M_{2a}	M_{2b}	M_{2c}
IS0	18.16 ± 0.08^{B*}	62.04 ± 0.54^{D}	169.33 ± 1.03^{C}	0.128 ± 0.001^{C}	0.560 ± 0.001^{C}	0.312 ± 0.001^{A}
IS60	17.55 ± 0.33^{B}	64.29 ± 0.67^{C}	178.67 ± 2.32^{B}	0.136 ± 0.003^{B}	0.585 ± 0.003^{B}	0.279 ± 0.005^{B}
IS80	22.72 ± 0.05^{A}	71.38 ± 0.86^{B}	193.53 ± 2.55^{A}	0.168 ± 0.004^{A}	0.584 ± 0.003^{B}	0.248 ± 0.007^{C}
IS100	22.88 ± 0.34^{A}	73.90 ± 0.61^{A}	196.97 ± 0.61^{A}	0.162 ± 0.003^{A}	0.609 ± 0.007^{A}	0.228 ± 0.008^{D}

*Mean ± standard deviation within a column followed by different letters is significantly different ($p < 0.05$).

Sucrose is a disaccharide and is known to bind water well (Tas et al., 2022) and with the effect of the lecithin and the following mixing process, it is emulsified to the chocolate matrix efficiently. However, according to the T_2 results, for inulin/stevia formulations, the behavior was different. Inulin is a polysaccharide, and it may not be emulsified to the matrix as well as sucrose. As seen in the results, the increase in the relaxation times is parallel with the decrease in sucrose concentration.

T_{2a} is mostly associated with the fat that has been emulsified well within the matrix. The strong interaction of sucrose/lecithin/fat resulted in shorter relaxation times. Since emulsification is expected less with the presence of inulin/stevia, a slight increase in T_{2a} was observed. However, despite the less emulsification hypothesis, contribution of that component to overall signal has increased and % contribution changed significantly from 12% to almost 17%. Since inulin is a polymer, it is likely that during the emulsification process it will trap more oil at the surface, but the interaction will be less resulting in longer relaxation times with a higher contribution to the signal.

The second component has been associated with the more mobile oil protons that have been trapped in the cocoa spread network and with the increase in inulin/stevia concentration, this fat could have been trapped within the inulin network and may have more mobility. The contribution of that component increasing with inulin concentrations strongly confirms this hypothesis.

The third component which is mostly associated with the bulk oil in the formulation had longer relaxation times with the increase in inulin/stevia concentrations. The longer relaxation times indicated higher mobility for the oil phase within the network. The contribution of that component decreased since oil has been trapped more within the polymeric network of inulin and it is likely that we observed exchange between the 2nd and 3rd proton pools.

As discussed later, the change in rheological properties is also strongly related to the changes in the relaxation times.

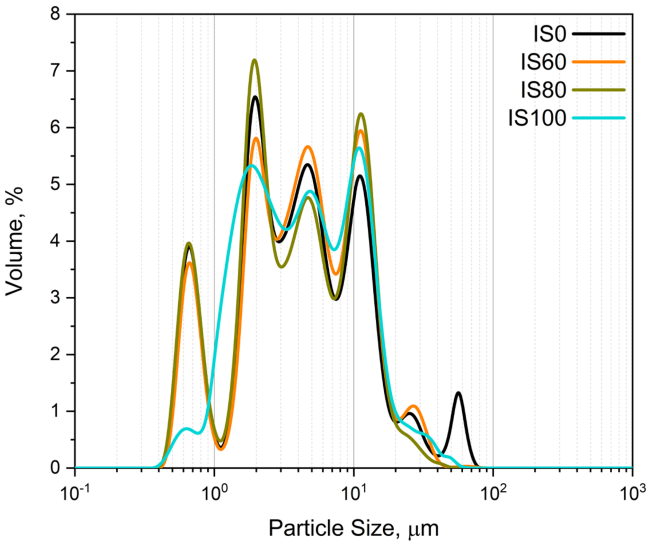

FIGURE 1 Particle size distribution of cocoa hazelnut spread samples.

3.3 | Particle size distribution (PSD)

Particle size distribution is an important quality parameter for food emulsions. It influences rheological behavior, textural properties, and sensory perception of the product. Laser diffraction method is widely used to determine PSD due to its wide range measurement scale. Particle size analysis indicated that the cocoa hazelnut spread samples had a broad PSD (polydisperse). As shown in Figure 1, the PSD of all samples was multimodal. Afoakwa et al. (2007) reported that PSD of many chocolate products are bimodal and trimodal. For different food, emulsions like peanut butter (Mohd Rozalli et al., 2015; Tanti et al., 2016) and sunflower tahini (Mureşan, 2018), multimodal PSD have been reported by different authors.

The mean size of the particles in cocoa hazelnut spread samples ranged from 0.448 to 74.308 µm. The specific surface area (SSA, m^2 g^{-1}), surface weighted mean ($D_{[3,2]}$, µm), volume weighted mean ($D_{[4,3]}$, µm), $Dv_{0.5}$, $Dv_{0.9}$ and span values were statistically similar for all samples ($p < 0.05$) (Table 3). Only the $Dv_{0.1}$ value, which corresponded to the smallest particles, obtained for IS100 sample was significantly different than those obtained for the other samples ($p < 0.01$). Compared to the other samples, IS100 had a lower volume fraction of particles with sizes below 0.89 µm. All samples included small proportion of particles with size >50 µm. This size of particles was found in slightly higher amount in control sample. This may be related to the agglomeration of sucrose particles in control

TABLE 3 Particle size distribution (PSD) properties of the cocoa hazelnut spread samples.

	Formulations			
PSD	IS0	IS60	IS80	IS100
SSA, m^2 g^{-1}	$2.57 \pm 0.19^{A*}$	2.42 ± 0.23^{A}	2.71 ± 0.11^{A}	2.13 ± 0.04^{A}
$D_{[3,2]}$, µm	2.35 ± 0.17^{A}	2.50 ± 0.24^{A}	2.22 ± 0.09^{A}	2.83 ± 0.05^{A}
$D_{[4,3]}$, µm	7.29 ± 2.50^{A}	6.41 ± 1.25^{A}	5.73 ± 0.81^{A}	6.43 ± 0.32^{A}
$Dv_{0.1}$, µm	$0.83 \pm 0.18^{B*}$	0.81 ± 0.10^{B}	0.76 ± 0.09^{B}	1.33 ± 0.08^{A}
$Dv_{0.5}$, µm	4.00 ± 0.23^{A}	4.38 ± 0.71^{A}	3.83 ± 0.92^{A}	4.22 ± 0.90^{A}
$Dv_{0.9}$, µm	16.99 ± 7.14^{A}	13.70 ± 2.22^{A}	12.88 ± 1.01^{A}	13.51 ± 0.10^{A}
Span	3.99 ± 1.60^{A}	2.94 ± 0.01^{A}	3.22 ± 0.49^{A}	2.96 ± 0.67^{A}

*Mean ± standard deviation within a row followed by different letters is significantly different ($p < 0.05$).

(Selvasekaran & Chidambaram, 2021). Large particle size might have caused the perception of graininess (Saputro et al., 2018). In a previous study, it was recorded that, particles larger than 30–35 μm in chocolate might cause sandy/gritty feeling (Afoakwa et al., 2007). In their study, Van de Walle et al. (2018) noted profound agglomeration in refined fat-based suspensions prepared with *Orafti HP inulin* (PD ≥23) but not in the ones prepared with sucrose or *Orafti HSI* (high soluble inulin).

3.4 | Textural analysis

The spreadability, firmness, cohesiveness, and adhesiveness values of the samples are presented in Table 4. In cocoa hazelnut spread, sugar has important functions other than adding a sweet taste. It also acts as a texturizing agent, preservative, adds color, and flavor. Substitution of sugar with inulin/stevia exerted variable influence on the textural parameters. The sample prepared with 80% substitution of sugar (IS80) had similar textural parameters compared to the control. Increase in the level of inulin/stevia gave lower firmness, spreadability, adhesive force, and adhesiveness values. This means that, complete replacement of sugar gave the lowest values of the measured textural parameters while 60% substitution of sugar yielded a firmer, and stickier product with higher spreadability compared to the control. Büker et al. (2021) determined the firmness value of chocolate spread prepared with partial replacement of sucrose with dried apple pomace at concentrations ranging between 4 and 20 g/100 g. In their study, the lowest and highest firmness values were found for the samples containing 16 g/100 g and 8 g/100 g apple pomace. The sample with the lowest firmness also had the lowest spreadability value. The results obtained in our study agreed with the findings of their study.

The differences in textural parameters were attributed to the differences in the particle size, moisture content, total solid content, and interactions of particles in the samples. In a study, Goktas et al. (2021) prepared compound chocolate by replacing sugar with inulin (6–12%) having different polymerization degrees (DP <10 and DP ≥23) and reported that the hardness of compound chocolate was affected by both the polymerization degree and the level of inulin in formulation. Sample containing 6% inulin with low DP had the lowest firmness while sample containing similar amount of inulin with high DP produced the highest firmness in their study. They stated that alteration of moisture content of samples caused by the different hygroscopicity of ingredients is an important factor influencing the hardness of samples. Inulin powder used in this study (Orafti HIS) has spherical particles with average particle size (D_{43}) of 89 μm while sucrose appears as angular shaped solids with a comparatively broad particle size distribution (Van de Walle et al., 2018). Sucrose may have fragmented easier than inulin due to its crystal structure during processing. Sucrose and other dense smaller particles will fill the void space between inulin particles which resulted in high solids packing intensity in the product. This might, at least partially, have contributed to the increase in firmness of inulin-stevia containing samples with increasing sugar content.

During storage, a decrease in textural parameters was observed for all samples. IS100 sample maintained its textural parameters better

TABLE 4 Textural properties of the cocoa hazelnut spread samples.

Texture parameter	Storage (month)	Formulation			
		IS0	IS60	IS80	IS100
Firmness (N)	0	$14.76 \pm 1.64^{B*a**}$	19.13 ± 0.50^{Aa}	13.83 ± 2.09^{Ba}	9.55 ± 1.40^{Ca}
	1	12.29 ± 0.51^{Ba}	14.44 ± 0.63^{Ab}	12.40 ± 1.46^{Ba}	8.70 ± 0.84^{Cab}
	2	9.54 ± 0.78^{Bb}	10.92 ± 0.98^{Ac}	8.47 ± 0.41^{BCb}	7.59 ± 0.47^{Cb}
	3	8.18 ± 2.42^{ABb}	8.74 ± 0.60^{Ad}	6.25 ± 0.41^{BCc}	6.03 ± 0.76^{Cc}
Spreadability (N s)	0	8.02 ± 1.04^{Ba}	10.40 ± 0.66^{Aa}	7.58 ± 1.41^{Ba}	5.08 ± 0.98^{Ca}
	1	7.05 ± 0.44^{Bab}	8.77 ± 0.51^{Ab}	7.10 ± 1.07^{Ba}	5.10 ± 0.65^{Ca}
	2	6.23 ± 0.66^{Bab}	7.11 ± 0.56^{Ac}	5.48 ± 0.33^{BCb}	5.09 ± 0.37^{Ca}
	3	5.45 ± 1.88^{ABb}	5.58 ± 0.20^{Ad}	3.89 ± 0.16^{Bc}	4.04 ± 0.65^{ABa}
Adhesive force (N)	0	-9.28 ± 0.72^{Bc}	-13.43 ± 0.37^{Cd}	-9.62 ± 1.68^{Bb}	-6.61 ± 0.92^{Ab}
	1	-7.78 ± 0.61^{Bb}	-9.59 ± 0.41^{Cc}	-8.53 ± 1.29^{BCb}	-5.66 ± 0.54^{Ab}
	2	-5.78 ± 0.51^{Ba}	-6.65 ± 0.81^{Cb}	-5.26 ± 0.28^{ABa}	-4.67 ± 0.28^{Aa}
	3	-4.71 ± 1.30^{ABa}	-5.01 ± 0.31^{Ba}	-3.83 ± 0.23^{Aa}	-3.74 ± 0.46^{Aa}
Adhesiveness (N s)	0	-4.67 ± 0.75^{Ba}	-7.26 ± 0.76^{Cc}	-4.93 ± 1.23^{Bb}	-3.10 ± 0.55^{Aa}
	1	-4.59 ± 0.26^{Ba}	-6.68 ± 0.70^{Cbc}	-4.88 ± 0.93^{Bb}	-3.26 ± 0.40^{Aa}
	2	-4.77 ± 0.68^{Aa}	-6.13 ± 0.84^{Bb}	-4.57 ± 0.22^{Ab}	-4.06 ± 0.33^{Ab}
	3	-4.20 ± 1.86^{Aa}	-4.68 ± 0.27^{Aa}	-3.22 ± 0.16^{Aa}	-3.25 ± 0.55^{Aa}

*For each texture parameter, capital letters in the same row compare the effect of the formulations for the given storage period ($p < 0.05$).
**For each texture parameter, lowercase letters in the same column compare the effect of storage period for the given formulation ($p < 0.05$).

during storage compared to other samples. No significant change occurred in spreadability and adhesiveness of IS 100 sample after 3 months of storage ($p > 0.05$). The % change in textural parameters during storage was higher in the samples prepared with partial substitution of sugar compared to control. After 3 months of storage, inulin-stevia containing samples had textural parameters similar to those of control. The only exception was the firmness of IS100 sample which was lower than the control.

3.5 | Color

The color parameters of the samples measured immediately after production and during storage are presented in Table 5. At the beginning and end of the storage, the L*, a*, and b* values of the control sample were significantly higher compared to the ones prepared with stevia and inulin ($p < 0.05$). This means that replacement of sucrose with stevia and inulin resulted in darker color. This result is in accordance with the findings of Shah et al. (2010) where sucrose free chocolate was produced and replacement of sucrose with stevia, inulin and polydextrose resulted in lower L*, a*, and b* values. They attributed this, at least partially, to the higher surface roughness of sucrose-free chocolates. Shourideh et al. (2012) similarly reported that dark chocolate made with inulin had lower L*, a*, and b* values compared to the one made with sucrose. They stated that inulin absorbs water and the chocolate made with inulin scatter less light, resulting in a darker color. Aidoo et al. (2014, 2015) reported similar results during production of sugar-free chocolate including inulin and polydextrose mixtures in its formulation.

PSD and the processing parameters may also influence the color parameters of chocolate. Afoakwa, Paterson, Fowler, and Vieira (2008) noted an inverse relationship between the $Dv_{0.9}$ values of the chocolate and the lightness (L*) values. In this study, $Dv_{0.9}$ values of the spreads were statistically similar ($p > 0.05$), so the difference in color is attributed to the compositional variation, rather than particle size effects. Among the inulin-stevia containing samples, IS100 had lower b* values compared to IS60 immediately after production. Apart from that, there was no significant difference ($p > 0.05$) between the color parameters of the samples which indicated that increasing the level of stevia and inulin in formulation did not produce a visible change in color.

At the end of storage period, an increase in b* values of the samples were recorded. Except the control, 3 months storage yielded an increase also in a* values. Storage did not create a significant influence on the L* values.

3.6 | Melting profile by DSC

DSC thermograms of the formulations and the raw materials are given in Figure 2. In the thermogram of inulin, a peak at around 60°C was observed. Blecker et al. (2003) reported that the peak coming from inulin at that temperature is because of the removal of the moisture from the inulin. Since the pans for DSC analysis were not covered hermetically, the evaporation of water at that temperature is an expected phenomenon. In the thermogram of palm oil, two different peaks were observed. de Almeida et al. (2021) examined the thermal behavior of the palm oil and a similar trend was observed. Since the crystal structures differ depending on the temperature of the medium, it was thought that the larger crystals shrank below 20°C and all crystals were liquefied above 20°C. Similar case about change in crystal size but by cooling was observed in the study by Berk et al. (2021) on the crystallization of honey. Hazelnut oil and puree showed one distinct peak below 0°C. Tan and Man (2012) measured the melting

TABLE 5 Color parameters of the cocoa hazelnut spread samples during storage.

Color parameter	Storage (month)	Formulation			
		IS0	IS60	IS80	IS100
L*	0	34.36* ± 1.53^{A}**a***	31.18 ± 1.98^{Ba}	30.70 ± 1.56^{Bb}	30.64 ± 0.83^{Bab}
	1	29.94 ± 1.70^{Bb}	32.60 ± 0.91^{Aa}	30.15 ± 1.40^{Bb}	30.26 ± 1.93^{Bb}
	2	32.95 ± 1.36^{Aa}	31.98 ± 1.46^{ABa}	30.99 ± 1.16^{Bb}	29.20 ± 1.41^{Cb}
	3	34.35 ± 1.20^{Aa}	32.90 ± 1.36^{Ba}	32.73 ± 0.61^{Ba}	32.29 ± 1.30^{Ba}
a*	0	7.29 ± 0.53^{Aa}	6.18 ± 0.49^{Bc}	6.00 ± 0.52^{Bbc}	5.89 ± 0.30^{Bb}
	1	6.21 ± 0.46^{ABb}	6.66 ± 0.32^{Abc}	5.84 ± 0.43^{Bc}	5.89 ± 0.62^{Bb}
	2	7.29 ± 0.36^{Aa}	6.84 ± 0.44^{ABab}	6.47 ± 0.39^{BCb}	6.20 ± 0.65^{Cb}
	3	7.76 ± 0.50^{Aa}	7.30 ± 0.47^{ABa}	7.20 ± 0.40^{Ba}	7.06 ± 0.33^{Ba}
b*	0	5.78 ± 0.62^{Ab}	4.09 ± 0.56^{Bb}	3.64 ± 0.62^{BCc}	3.26 ± 0.34^{Cbc}
	1	4.42 ± 0.55^{Ac}	4.57 ± 0.36^{Ab}	3.45 ± 0.47^{Bc}	3.21 ± 0.57^{Bc}
	2	6.15 ± 0.44^{Aab}	5.24 ± 0.55^{Ba}	4.60 ± 0.43^{Cb}	3.80 ± 0.52 ^{Db}
	3	6.76 ± 0.57^{Aa}	5.80 ± 0.53^{Ba}	5.33 ± 0.44^{BCa}	5.18 ± 0.35^{Ca}

*Values are given as mean ± standard deviation ($n = 10$).

**For each color parameter, capital letters in the same row compare the effect of the formulations for the given storage period ($p < 0.05$).

***For each color parameter, lowercase letters in the same column compare the effect of storage period for the given formulation ($p < 0.05$).

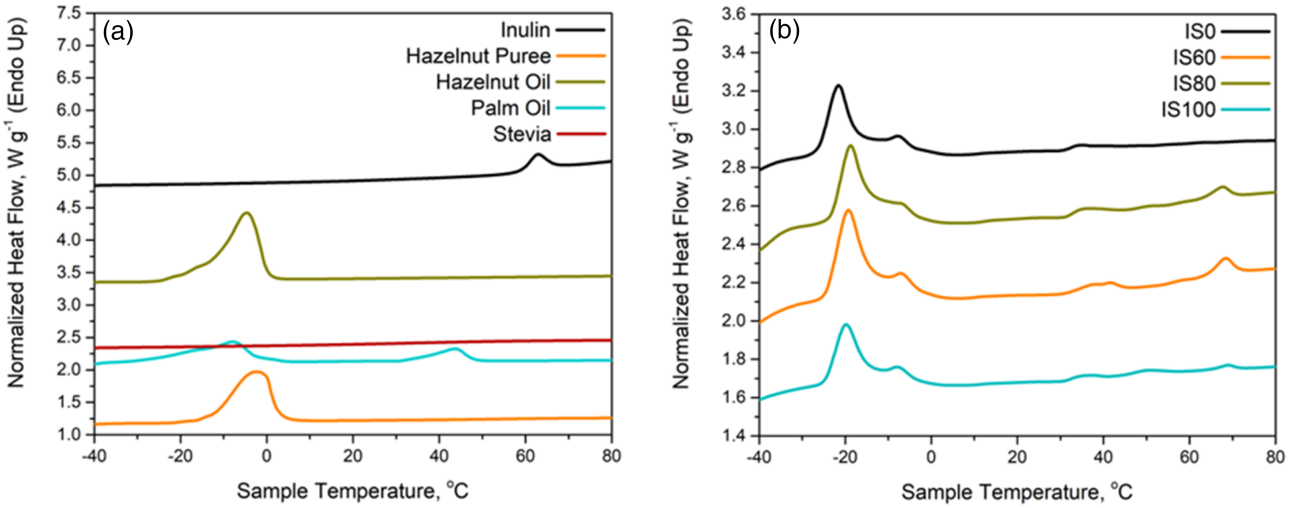

FIGURE 2 DSC thermograms of the (a) raw materials and (b) formulations after production.

TABLE 6 Enthalpy values (J g^{-1}) of the reactions occurred at specified temperatures.

Formulation	Storage (month)	Enthalpy values (J g^{-1})			
		At − 21°C	At − 7°C	At 35°C	At 63°C
IS0	–	$8.777 \pm 0.206^{B*a**}$	$0.626 \pm 0.288^{AB a}$	0.401 ± 0.090^{Bb}	–
	1	3.851 ± 0.355^{Cc}	0.459 ± 0.051^{Ba}	1.044 ± 0.078^{Aa}	–
	2	1.339 ± 0.483^{Cd}	0.620 ± 0.130^{Ba}	0.930 ± 0.268^{Aa}	–
	3	6.838 ± 0.230^{Cb}	0.468 ± 0.071^{Ba}	0.958 ± 0.051^{Ba}	–
IS60	–	9.717 ± 0.392^{Aa}	0.580 ± 0.039^{Aba}	1.174 ± 0.041^{Aab}	1.458 ± 0.084^{Aa}
	1	8.176 ± 0.279^{Ba}	0.232 ± 0.028^{Cb}	1.119 ± 0.026^{Ab}	1.106 ± 0.074^{Bc}
	2	8.478 ± 1.183^{Ba}	0.412 ± 0.152^{Bab}	1.234 ± 0.048^{Aa}	1.225 ± 0.012^{Bbc}
	3	8.091 ± 0.098^{Ba}	0.336 ± 0.047^{Bb}	1.110 ± 0.028^{Ab}	1.284 ± 0.004^{Ab}
IS80	–	7.209 ± 0.450^{Cc}	0.227 ± 0.103^{Bb}	0.718 ± 0.030^{Bc}	0.875 ± 0.117^{Bb}
	1	8.622 ± 0.330^{Bb}	0.367 ± 0.035^{Bb}	1.082 ± 0.028^{Ab}	1.325 ± 0.070^{Aa}
	2	10.23 ± 0.101^{Aba}	0.975 ± 0.121^{Aa}	1.026 ± 0.042^{Ab}	1.431 ± 0.117^{Aa}
	3	8.184 ± 0.289^{Bb}	0.333 ± 0.059^{Bb}	1.357 ± 0.200^{Aa}	1.295 ± 0.017^{Aa}
IS100	–	10.46 ± 0.053^{Ab}	0.851 ± 0.057^{Ac}	0.530 ± 0.257^{Bb}	0.493 ± 0.110^{Ca}
	1	9.664 ± 0.526^{Ab}	1.316 ± 0.048^{Aa}	1.015 ± 0.035^{Aa}	0.534 ± 0.036^{Ca}
	2	11.56 ± 0.453^{Aa}	1.163 ± 0.063^{Ab}	1.211 ± 0.024^{Aa}	0.231 ± 0.068^{Cb}
	3	9.722 ± 0.067^{Ab}	1.032 ± 0.044^{Ab}	1.331 ± 0.023^{Aa}	0.620 ± 0.061^{Ba}

*For each parameter, capital letters in the same column compare the effect of the formulations for the given storage period ($p < 0.05$).
**For each parameter, lowercase letters in the same column compare the effect of storage period for the given formulation ($p < 0.05$).

temperature of hazelnut oil as −9.07°C and it was found very close to the measured values of hazelnut puree (−3°C) and hazelnut oil (−4.8°C). In the scanned temperature range, stevia did not give any thermal response.

In Figure 2b, major difference of the thermograms is coming from the peak that belongs to the inulin. IS0 sample has no peak at around 60°C due to the absence of inulin in the formulation. All formulations have distinct peaks at −21, −7 and 35°C. Enthalpy values of the reactions during thermal scanning are also given in Table 6.

The addition of stevia to the formulation decreased the area under the peak belonging to water evaporation from inulin due to the stevia. The steviol glycosides presenting in the inulin have hydrophilic nature and interact with the water (Moongngarm et al., 2022). The presence of stevia suppressed the removal of the water at elevated temperatures. de Souza Correia Cozentino et al. (2022) studied functional hazelnut spread and observed a similar melting trend having two different peaks, one below 0°C and one above 0°C. The peak coming at 35°C was attributed to the melting of all crystals in the

formulation which was the main change responsible for the mouth feel. The addition of stevia and inulin did not affect the energy required to melt the crystals significantly for IS80 and IS100 samples compared to the control ($p < 0.05$).

The melting enthalpies of the samples except IS60 showed a significant increase after 1 month of storage and then stayed nearly constant. The level of increase was the highest for the control sample. At the end of the first month of storage, all samples had similar melting enthalpies. For sample IS60, no significant change was observed in the melting enthalpy during storage.

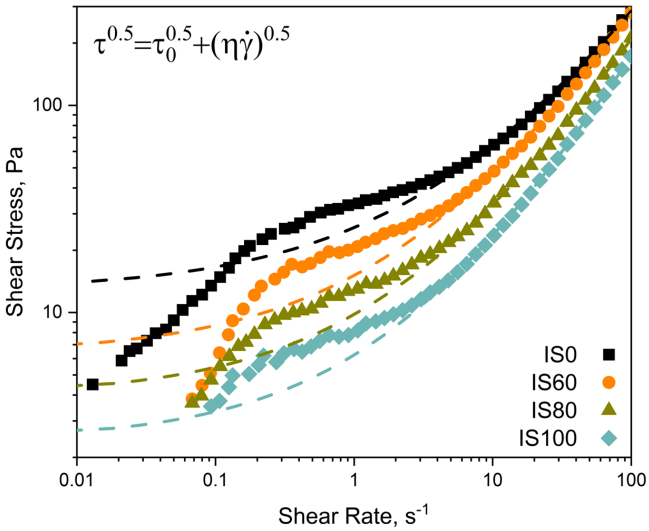

FIGURE 3 Rheograms and Casson model fittings of the formulations after production.

3.7 | Rheological analysis

Casson model is a widely used model to describe the flow behavior of chocolate (Selvasekaran & Chidambaram, 2021). Principato et al., (2022) used Casson model to describe the flow behavior of commercial hazelnut/cocoa spread samples and obtained a good fit of the experimental data to the Casson model. Experimental shear rate and stress data obtained in this study were well-fitted to the Casson model with the lowest R^2 value of 0.990. Rheograms of the formulations obtained after production and Casson model fittings are given in Figure 3.

For each sample, the Casson model parameters obtained during storage are presented in Table 7. After production, the Casson plastic viscosity (η) of the control sample was 2.737 Pa s. Inulin and stevia-containing samples had lower η values compared to the control. Aidoo et al. (2014) reported that chocolate samples prepared with variable levels of inulin and polydextrose had higher Casson plastic viscosity compared to the one prepared with sucrose. In their study, a rise in inulin level with a simultaneous decrease in polydextrose level caused higher viscosity values. Aidoo et al. (2015) obtained higher Casson plastic viscosity values in chocolate made with stevia/inulin and polydextrose compared to the one prepared with sucrose. These findings differ from the ones obtained in this study. This may be caused by the differences in formulation and the type of inulin used. In the production of cocoa hazelnut spread, inulin with high solubility was used. Shah et al. (2010) prepared sucrose-free milk chocolate using stevia and different types of inulin and found that the inulin with a lower polymerization degree (PD) gave a lower Casson viscosity value. It was also thought that higher internal friction caused by the

TABLE 7 Casson model fitting parameters.

Formulation	Storage (month)	η, (Pa.s)	τ_0, (Pa)	R^2	RMSE
IS0	–	2.737* ± 0.218^{A**a***}	12.55 ± 2.440^{Aa}	0.990	6.368
	1	2.093 ± 0.410^{Aa}	10.96 ± 4.020^{Aa}	0.991	6.755
	2	2.390 ± 0.335^{Aa}	8.690 ± 2.760^{Aa}	0.994	5.794
	3	1.702 ± 0.105^{Aa}	6.650 ± 4.050^{Aa}	0.990	5.704
IS60	–	2.196 ± 0.296^{ABa}	6.145 ± 0.084^{Ba}	0.995	4.519
	1	1.940 ± 0.514^{Aa}	5.847 ± 0.293^{ABa}	0.995	3.986
	2	1.974 ± 0.122^{Aa}	4.651 ± 0.629^{ABa}	0.995	3.988
	3	2.221 ± 0.325^{Aa}	4.648 ± 0.919^{Aa}	0.996	3.764
IS80	–	1.656 ± 0.105^{Ba}	4.044 ± 1.008^{Ba}	0.996	3.135
	1	1.950 ± 0.563^{Aa}	3.459 ± 0.075^{ABa}	0.996	3.439
	2	2.047 ± 0.100^{Aa}	4.275 ± 0.167^{ABa}	0.996	3.824
	3	1.724 ± 0.176^{Aa}	3.498 ± 0.537^{Aa}	0.996	3.253
IS100	–	1.534 ± 0.194^{Ba}	1.428 ± 0.027^{Ba}	0.997	2.275
	1	1.577 ± 0.003^{Aa}	1.821 ± 0.269^{Ba}	0.996	2.503
	2	1.535 ± 0.220^{Aa}	1.788 ± 0.081^{Ba}	0.996	2.406
	3	1.537 ± 0.040^{Aa}	1.227 ± 0.284^{Aa}	0.997	2.150

*Values are given as mean ± standard deviation ($n = 10$).
**For each parameter, capital letters in the same column compare the effect of the formulations for the given storage period ($p < 0.05$).
***For each parameter, lowercase letters in the same column compare the effect of storage period for the given formulation ($p < 0.05$).

agglomeration of sucrose particles in the presence of a small amount of water may be, at least partially, responsible for the higher Casson plastic viscosity of the control (Selvasekaran & Chidambaram, 2021).

The increasing level of inulin/stevia in formulation produced a slight but not statistically significant reduction in η value. One reason of this slight decrease may be the reduction in the interaction of solid particles with decreasing levels of sugar in the complex system. Principato et al., (2022) investigated the rheological properties of five different commercial hazelnut-based spreads and reported a reduction in the Casson plastic viscosity of samples with an increasing fat/sugar ratio. Moreover, it was thought that the sugar interacts with lecithin more than inulin, which may contribute to the retardation of the flow of the medium. Bonarius et al. (2014) concluded this kind of behavior in fiber-added lipid-based foods to the fact that hydrophilic materials form water bridges while fiber facilitates the flowing. In addition, this phenomenon may cause the insufficient emulsification. The increase in the inulin/stevia content decreased the η and increased the T_2 values that has a correlation with η value ($r = -0.843$ for T_{2a}, $r = -0.916$ for T_{2b} and $r = -0.935$ for T_{2c}, $p < 0.05$).

It has been stated by different authors that the Casson yield stress is influenced greatly by particle-particle interactions which depend on the composition of ingredients (Aidoo et al., 2014; Afoakwa, Paterson, & Fowler, 2008; Shah et al., 2010). In this study, the replacement of sugar with inulin/stevia yielded a significant decrease in Casson yield stress (τ_0). The samples with a higher level of inulin/stevia had lower values of Casson yield stress. Different levels of particle–particle interaction caused by the different types and concentrations of the ingredients in cocoa hazelnut spread samples may create variations in flow properties. Aidoo et al. (2014) obtained lower Casson yield stress values in chocolate made with inulin and polydextrose compared to the one prepared with sucrose. They attributed this to the differences in PSD which influences inter-particle contact. In their study, the formulation with 100% inulin which contained large crystals with more void spaces compared to other formulations had the lowest yield stress. This finding agrees with that obtained in this study. In the case of IS 100 sample, the fat may fill the voids between particles easily and reduce the particle-particle interaction which contributes to the reduction of yield stress (Afoakwa, Paterson, & Fowler, 2008). Flow properties of spreads did not change significantly during the three-month storage period. Besides internal dynamics of the formulations, the processing conditions such as pump and pipe design to transport the spread from the tank to filling are affecting from the rheological behavior. The differences between formulations provide knowledge about these design parameters. Lower Casson plastic viscosity facilitates the pumpability of the product inside the pipeline.

4 | CONCLUSION

Cocoa hazelnut spread is a popular food consumed by every aged people. The controversiality of this food is mostly due to its high sugar content. As a solution to this problem, sweetener addition accompanied by fiber fortification is an option. In this study, stevia and inulin-added cocoa hazelnut spread formulations were prepared and characterized. In general, the replacement of sugar with inulin and stevia yielded products with a darker color, lower Casson viscosity, and quite similar particle size distribution compared to the control. The investigated properties of the products were influenced by the level of inulin and stevia. Complete removal of sugar resulted in low spreadability. 80% replacement of sugar with inulin and stevia yielded a product with similar textural parameters and fat-melting mouth feeling compared to the control. Cocoa hazelnut spreads prepared with inulin and stevia showed good storage stability. The findings of this study can guide food manufacturers in formulating products that meet both health-conscious consumer preferences and quality expectations. Overall, this research has the potential to inspire the food industry to explore and adopt innovative approaches in sugar reduction without compromising product quality. As a further study, there is need for a sensory analysis to make sure that the sugar-reduced formulations are acceptable based on consumer attitude and perception.

AUTHOR CONTRIBUTIONS

Berkay Berk: Formal analysis; writing – original draft; methodology; writing – review and editing. **Sumeyye Cosar:** Formal analysis. **Bekir G. Maz:** Writing – original draft; writing – review and editing; supervision; resources; methodology; formal analysis; investigation. **Mecit Halil Oztop:** Resources; supervision; writing – original draft.

CONFLICT OF INTEREST STATEMENT

The authors declare that they do not have any conflict of interest.

DATA AVAILABILITY STATEMENT

The data that support the findings of this study are available from the corresponding author upon reasonable request.

ETHICS STATEMENT

This study does not involve any human or animal testing.

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