

ISSN 1678-3921

Journal homepage: www.embrapa.br/pab

For manuscript submission and journal contents,
access: www.scielo.br/pab

Technological and nutritional aspects of dark chocolate with added coffee husk flour











Abstract – The objective of this work was to produce dark chocolates with the addition of coffee husk flour (CHF) and to evaluate the generated effects on their physical, physicochemical, microbiological, textural, and rheological characteristics. Husks of the Pacamara coffee (*Coffea arabica*) variety, produced under organic management, were used. The samples were previously dried, ground, and sieved at 0.250 mm. Five chocolate formulations were previously standardized at 55% content of cocoa solids (mass and cocoa butter) and at 0.4% soy lecithin. The addition of CHF was tested at the concentrations of 0, 2.5, 5.0, 7.5, and 10%, using a completely randomized design and three replicates. The addition of CHF up to 10% alters the physicochemical, textural, and rheological properties of the chocolate formulations, but without compromising their composition and quality. The tested formulations only differed regarding hardness and cohesiveness, evaluated in the texture profile. The hardness of the chocolate formulations increases as CHF is added.

Index terms: *Coffea arabica*, by-product, Casson model, color, rotational test, sustainability.

Aspectos tecnológicos e nutricionais de chocolate amargo com adição de farinha de casca de café

Resumo – O objetivo deste trabalho foi produzir chocolates amargos com adição de farinha de casca de café (CHF) e avaliar os efeitos gerados nas suas características físicas, físico-químicas, microbiológicas, de textura e reológicas. Foram utilizadas cascas da variedade de café (*Coffea arabica*) Pacamara, produzida com manejo orgânico. As amostras foram previamente secas, moídas e peneiradas a 0,250 mm. Cinco formulações de chocolates foram previamente padronizadas em 55% de teor de sólidos de cacau (massa e manteiga de cacau) e em 0,4% de lecitina de soja. A adição de CHF foi testada nas concentrações de 0, 2,5, 5,0, 7,5 e 10%, tendo-se utilizado delineamento inteiramente casualizado e três repetições. A adição de CHF até 10% altera as propriedades físico-químicas, de textura e reológicas das formulações de chocolate, mas sem comprometer sua composição e sua qualidade. As formulações testadas diferiram apenas quanto à dureza e à coesividade, avaliadas no perfil de textura. A dureza das formulações de chocolate aumenta à medida que a CHF é adicionada.

Termos para indexação: *Coffea arabica*, subproduto, modelo Casson, cor, teste rotacional, sustentabilidade.

Marília Viana Borges⁽¹⁾ ,
Cristina Xavier dos Santos Leite⁽¹⁾ ,
Ingrid Alves Santos⁽¹⁾ ,
Danilo Junqueira Leão⁽¹⁾ ,
Sibelli Passini Barbosa Ferrão⁽¹⁾ ,
Leandro Soares Santos⁽¹⁾ ,
Amanda Beatriz Sales de Lima⁽¹⁾ ,
Carmen Wobeto⁽²⁾ ,
Suzana Caetano da Silva Lannes⁽³⁾ ,
Marcondes Viana da Silva^{(1)✉} 

⁽¹⁾ Universidade Estadual do Sudoeste da Bahia, Praça Primavera, nº 40, Primavera, CEP 45700-000 Itapetinga, BA, Brazil. E-mail: marilia.engali@gmail.com, cristinaxsleite@gmail.com, ingridengali@gmail.com, daniolo7leao@yahoo.com.br, sferrao@uesb.edu.br, leosoaessantos@yahoo.com.br, amandaengalimentos@hotmail.com, marcondes.uesb@gmail.com

⁽²⁾ Universidade Federal de Mato Grosso, Avenida Alexandre Ferronato, nº 1.200, Setor Industrial, CEP 78550-728 Sinop, MT, Brazil. E-mail: carmenwobeto2014@gmail.com

⁽³⁾ Universidade de São Paulo, Avenida Professor Lineu Prestes, nº 580, Bloco 13-A, Cidade Universitária, CEP 05508-000 São Paulo, SP, Brazil. E-mail: scslan@usp.br

✉ Corresponding author

Received

August 15, 2023

Accepted

February 20, 2024

How to cite

BORGES, M.V.; LEITE, C.X. dos S.; SANTOS, I.A.; LEÃO, D.J.; FERRÃO, S.P.B.; SANTOS, L.S.; LIMA, A.B.S. de; WOBETO, C.; LANNES, S.C. da S.; SILVA, M.V. da. Technological and nutritional aspects of dark chocolate with added coffee husk flour.

Pesquisa Agropecuária Brasileira, v.59, e03484, 2024. DOI: <https://doi.org/10.1590/S1678-3921.pab2024.v59.03484>.

Introduction

The ingredients used in chocolate formulations are varied and depend on regional and cultural preferences and on the country's legislation regarding cocoa (*Theobroma cacao* L.) concentrations, milk solids, and permitted amounts and types of vegetable fats (Afoakwa, 2016).

Several studies available in the literature have been exploring the production of chocolates using unconventional ingredients, such as raspberry leaves (Barišić et al., 2020), blackberry juice encapsulate (Lončarević et al., 2018), grape pomace products (Bolenz & Glöde, 2021), and cinnamon (Muhammad et al., 2018), which are added as powder material, extract, or in encapsulated form, since they bring nutritional and functional enrichment to food products.

A possible alternative ingredient is coffee husk flour, a by-product of coffee farming that has a high amount of antioxidant compounds, such as caffeine, chlorogenic acid, trigonelline, and diterpenes, but whose application is still limited (Lee et al., 2023). According to these authors, although the coffee (*Coffea arabica* L.) industry generates considerable amounts of by-products at all stages of its production chain, the husk is the most abundant, with an estimated 0.18 ton generated for each ton of dry-harvested coffee. In this scenario, researches on the exploitation of coffee by-products can contribute to reduce the environmental impacts caused by the disposal of these residues, in addition to generating income and jobs considering the high chocolate consumption worldwide, consequently improving the sustainability of the coffee production chain.

The objective of this work was to produce dark chocolates with the addition of coffee husk flour and to evaluate the generated effects on their physical, physicochemical, microbiological, textural, and rheological characteristics.

Materials and Methods

The used husks were from the Pacamara coffee (*C. arabica*) variety, produced under organic management at Fazenda Floresta, located in the municipality of Ibicoara, in the state of Bahia, in the Chapada Diamantina region, Brazil.

Before their physicochemical characterization, the coffee husks were crushed in the SL-031 Willey-knife

mill (Solab: Equipamentos para Laboratório, Piracicaba, SP, Brazil) and, then, sieved to 0.25 mm particles.

The following variables were determined according to Association of Official Analytical Chemists (AOAC, 2010): moisture by method 984.25; ash by method 925.51; titratable acidity by method 942.15; hydrogen potential (pH) by method 981.12; water activity (a_w), at $25\pm 1^\circ\text{C}$, using the AquaLab-4TE analyzer (Decagon Devices Inc, Fisher Scientific, Waltham, MA, USA); and total protein by method 920.152. Total lipids were obtained as described by Bligh & Dyer (1959). Reducing sugars were determined by the 3,5-dinitrosalicylic acid method (Miller, 1959), being neutralized with NaOH solution; this same analysis was used to obtain total sugars, but the samples were subjected to hydrolysis with concentrated HCl under heating before being neutralized. Non-reducing sugars were calculated by the difference between the contents of total sugars and reducing sugars, with results expressed in milligram of glucose per 100 milliliter.

The mineral composition of the coffee husks was determined using the Vista-PRO inductively coupled argon plasma optical emission spectrometer (Varian Australia Pty Ltd., Mulgrave, Australia), simultaneously with an axial array, Sturman-Master nebulization chamber, and V-groove nebulizer, equipped with a solid-state detector with charge-coupled device (CCD) arrangement. Argon with a 99.998% purity (White Martins Gases Industriais Ltda, Rio de Janeiro, RJ, Brazil) was used as the carrier gas. All determinations were performed with three replicates, expressed in milligrams of the corresponding mineral per kilogram of sample. The operating conditions of the equipment were: 40 MHz radiofrequency generator, 1.3 kW radiofrequency generator power, 1 s signal integration time, 15 L min^{-1} main argon flow, 1.5 L min^{-1} auxiliary argon flow, 0.8 L min^{-1} nebulization argon flow, 200 kPa nebulization pressure, 2.0 mL min^{-1} peristaltic pump speed, barium emission line CCD detector at 285.213 nm, carbon at 193.027 nm, calcium at 422.673 nm, copper at 324.754 nm, iron at 238.204 nm, potassium at 766.491 nm, magnesium at 285.213 nm, manganese at 257.610 nm, and sodium at 588.995 nm.

The evaluated chocolate formulations were previously standardized at 55% content of cocoa solids (mass and cocoa butter) and at 0.4% soy lecithin. Their

sugar content varied according to the addition of coffee husk flour at 0, 2.5, 5.0, 7.5, and 10% (Table 1).

The chocolate formulations were produced in the Spectra 11 Melanger stone mill (Spectra: Melangers & Equipment, Coimbatone, India), at an average temperature of $57\pm 2^\circ\text{C}$, where the conching and refining stages were carried out. Afterwards, the cocoa mass was added through homogenization for 1 hour. Sequentially, the sugar, the coffee husk flour, and part of the cocoa butter were slowly mixed together and kept under stirring for 21 hours. Afterwards, the remaining amount of cocoa butter was added and, lastly, lecithin, 30 min before the end of chocolate production, totaling 24 hours of processing.

The obtained mixture was subjected to a tempering step for 30 min at 45°C , under constant agitation, in the Mini Chocomachine tempering machine (Finamac, São Paulo, SP, Brazil). Then, the mixture was allowed to reach 30°C for molding in acrylic molds.

The molded chocolates were cooled to 4°C and, subsequently, packaged in coated paper and stored at room temperature ($25\pm 2^\circ\text{C}$) until the time of analysis (physicochemical, color, microbiological, granulometric, rheological, and texture).

The same physicochemical variables previously described were determined for the chocolate formulations.

Each formulation was subjected to instrumental color analysis using the ColorQuest XE spectrophotometer (HunterLab, Reston, VA, USA) and the three-parameter reading system, CIELAB, proposed by the International Commission on Illumination (Shanda, 2007). The L^* , a^* , and b^* parameters were provided by the Colorquest XE colorimeter (HunterLab, Reston, VA, USA), in which L^* defines luminosity ($L^* = 0$ black and $L^* = 100$ white), and the a^* and b^* coordinates are responsible for chromaticity ($+a^*$ red, $-a^*$ green, $+b^*$ yellow, and $-b^*$ blue). The whiteness index (WI) was

calculated as a function of the L^* , a^* , and b^* values, using the equation $(\text{WI}) = 100 - [(100 - L^*)^2 + (a^*)^2 + (b^*)^2]^{0.5}$, as proposed by Baycar et al. (2022).

The presence of *Salmonella*, total coliforms at 35°C , thermotolerant coliforms at 45°C , molds, and yeasts was investigated. All analyzes were performed in accordance with the Food and Drug Administration (FDA, 2024).

The granulometry of the chocolate particles was determined using the Mitutoyo digital micrometer, with a scale from 0 to $25\ \mu\text{m}$ (Mitutoyo Sul Americana, Jundiaí, SP, Brazil). For this, a sample fraction of chocolate was previously melted on a metal plate, to which mineral oil was mixed with the aid of a spatula.

For the rheological analysis of the chocolate formulations, the rotational test was performed using the Haake-MARS II rheometer (Thermo Fisher Scientific, Waltham, MA, USA), with the unpolished C35/2Ti cone-plate sensor with a gap of 0.105 mm and a thermostatic bath at 40°C (Eischen & Windhab, 2002). The results were determined using the Rheowin software (Thermo Fisher Scientific, Waltham, MA, USA). The controlled shear rate test was conducted in the following three steps: 0.00 1/s to 65.00 1/s, $t = 180$ s; 65.00 1/s, $t = 60$ s; and 65.00 1/s to 0.00 1/s, $t = 180$ s. The obtained plastic viscosity and initial stress data were suitable for Casson's equation (Bourne, 2002).

For the texture analysis, the used samples were in the following format: 2.5 cm in height x 1.5 cm in diameter, with a mass of approximately 10 g. The chocolates were textured in the TA.HDplus texturometer, equipped with the Extended Craft Knife A/ECB probe (Stable Micro Systems Ltd, Surrey, United Kingdom), programmed to directly measure the following texture attributes: hardness, fracturability, adhesiveness, elasticity, cohesiveness, gumminess, and chewiness. The established parameters were: pre-test speed of $1.0\ \text{mm s}^{-1}$, test speed of $1.0\ \text{mm s}^{-1}$, and

Table 1. Formulations (F1–F5) of dark chocolate (55% total cocoa solids) with the addition of coffee (*Coffea arabica*) husk flour.

Ingredient	F1	F2	F3	F4	F5
Cocoa mass	49.00	49.00	49.00	49.00	49.0
Cocoa butter	6.00	6.00	6.00	6.00	6.0
Refined sugar	44.60	42.10	39.60	37.10	34.6
Soy lecithin	0.40	0.40	0.40	0.40	0.4
Coffee husk flour	0.00	2.50	5.00	7.50	10.0
Total	100	100	100	100	100

post-test speed of 2.0 mm s⁻¹, using a 50% strain rate and a data acquisition rate of 200 points per second. The texture attributes were read in ten replicates per repetition (Bourne, 2002).

To evaluate the breaking strength of the chocolates (force versus deformation curve), medal-shaped samples (5.0 cm in diameter and 0.3 cm in thickness) were subjected to a maximum breaking force, expressed in kilogram-force, using a strain rate of 2.0 mm s⁻¹, compression distance of 5.0 mm, and trigger of 0.09 N. The force values obtained in each test were divided by the cross-sectional area of each evaluated product, and rupture stress was expressed in kgf (cm²)⁻¹ and converted to Newton.

The experiments were conducted using a completely randomized design, with five formulations of dark chocolate with the addition of 0, 2.5, 5.0, 7.5, and 10% of coffee husk flour as a substitute to sucrose, in three replicates. The results were subjected to the regression analysis as a function of treatments, at 5% probability. Mathematical models were selected according to significant effects ($p < 0.05$), considering the lack of nonsignificant adjustment ($p > 0.05$) and coefficients of determination (R^2). All analyzes were performed using the SAS statistical software, version 9.0 (SAS Institute Inc., Cary, NC, USA).

Results and Discussion

The mineral composition of coffee husk flour (average levels) and its effect (percentage) on the mineral enrichment of dark-chocolate formulations are shown in Table 2. The variations observed in the mineral composition of coffee husks can be attributed to differences in harvest time, cultivation region, and percentage of parchment and pulp, among other factors (Arya et al., 2022).

Despite these differences, coffee husk flour, due to its mineral composition, increased the nutrient content of dark chocolate, which, consequently, becomes an alternative to improve the consumption of these nutrients and, when combined with other sources, also contributes to their minimum daily intake. However, further studies are needed on the bioaccessibility of these elements, covering risk assessments, nutritional evaluations, and how variations in gastrointestinal conditions could affect bioaccessibility (Peixoto et al., 2016).

As for granulometry, the particle size of the dark-chocolate formulations remained between 0.20 and 0.22 μm , with no significant difference ($p > 0.05$) due to the addition or not of coffee husk flour. Therefore, according to the granulometry analysis, when added at the evaluated concentrations, coffee husk flour did not affect this important quality parameter. Arya et al. (2022) highlighted that particle size influences the physical and sensory characteristics of chocolate, such as sandiness, perceived as rough or large particles in the product when melted in one's mouth. Böhme et al. (2016) recommend that the particles must be smaller than 30 μm , ideally between 20–25 μm , to be imperceptible to the consumer. In the present study, the size of the particles of the coffee husk flour added to the chocolate formulations was within the range considered ideal.

A linear increase in moisture from 1.12 to 1.86% was observed in the chocolate formulations, being attributed to the addition of coffee husk flour, which contained 9.5% moisture, compared with that of chocolates, which is less than 1.0% (Afoakwa, 2016). However, higher levels of moisture can result in the formation of sugar bloom, as well as increase the viscosity of the product (Beckett et al., 2017).

The increasing addition of coffee husk flour to the analyzed formulations resulted in a significant linear increase in ash content (Figure 1 A). Despite this, the average ash contents were lower than the maximum regulatory values of 2.5% (USDA, 2024).

Table 2. Mineral composition of coffee (*Coffea arabica*) husk flour (CHF) and dark chocolates enriched with proportions of 0 to 10% CHF.

Mineral (mg 100 g ⁻¹)	CHF ⁽¹⁾	Dark chocolates with CHF (minimum – maximum) ⁽²⁾
Barium	0.34±0.01	-
Calcium	241.24±27.06	6.03 – 24.12
Copper	0.67±0.01	-
Iron	30.48±2.45	0.76 – 3.05
Potassium	2,928.81±56.12	73.22 – 292.9
Magnesium	98.04±9.67	2.45 – 9.8
Manganese	0.72±0.01	0.018-0.07
Sodium	24.34±2.10	-
Phosphor	158.28±15.20	3.96 – 15.83
Zinc	0.75±0.05	-

⁽¹⁾Means ± standard deviation. ⁽²⁾Minimum values refer to chocolates with the addition of 0% CHF, and maximum values refer to chocolates with 10% CHF. -, not considered due to its low content.

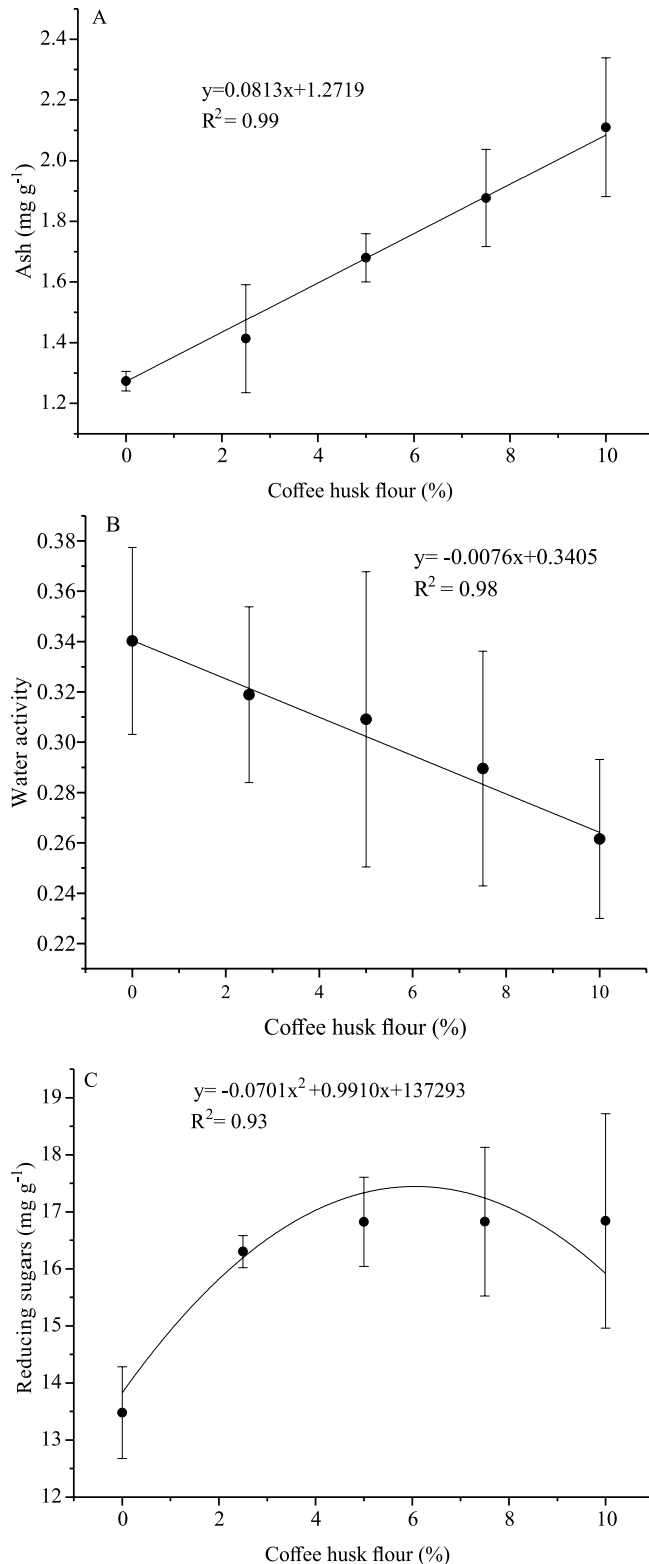


Figure 1. Impact of the addition of coffee (*Coffea arabica*) husk flour at 0, 2.5, 5, 7.5, and 10% to chocolate formulations on ash content (A), water activity (B), and reducing sugar content (C).

Acidity presented results that varied from 0.64 to 0.98 meq NaOH 100 g⁻¹, showing a significant increase ($p < 0.05$) with the addition of increased concentrations of coffee husk flour. De Vuyst & Weckx (2016) pointed out that, in chocolate formulations, acidity is influenced by fermentation steps, when the formation of acetic acid occurs, but that, during drying, roasting, and shelling, this acid is volatilized, which is important to avoid any negative influence on the formation of chocolate flavor. In the present work, however, acidity was only affected by the addition of coffee husk flour as the same cocoa mass and a standardized shelling process were used in all chocolate formulations.

Protein content also increased with increasing concentrations of coffee husk flour, with values ranging from 6.21 to 7.51 g 100 g⁻¹.

Compared with the other evaluated parameters (moisture, acidity, and protein), an opposite effect was observed for a_w , which decreased linearly as coffee husk flour was added (Figure 1 B). Overall, the a_w of the evaluated chocolate formulations presented lower levels than those reported in the literature. Beckett et al. (2017) found values from 0.4 to 0.5 for a_w , whereas Carvalho et al. (2018) obtained similar values of 0.519 for a_w when evaluating milk chocolates with 2.15% of freeze-dried grapes. However, Agibert & Lannes (2018) obtained a lower a_w of 0.358 when analyzing chocolates produced with encapsulated peanut oil.

Reducing sugar content was the only parameter that presented a quadratic behavior between the formulations (Figure 1 C). Regarding sugar contents, the chocolate formulations with higher values did not show significant changes in color despite the effect of the concentration of sugars on the speed of the Maillard reaction. This result was possibly due to the used temperature of $57 \pm 2^\circ\text{C}$ in the production process, which likely influenced the homogeneous coloring in the different formulations.

In the evaluated chocolate formulations, the following variables did not show a significant linear or quadratic regression ($p > 0.05$): pH (5.12); lipids (40.72 g 100 g⁻¹); total sugars (57.38 g 100 g⁻¹); non-reducing sugars (41.59 g 100 g⁻¹); and color, i.e., L* (30.52), a* (6.60), b* (4.93), and the WI (29.62).

In an efficient conching process, the pH of chocolate can reach values close to 4.95 to 5.70, considered ideal for the product (Marchioretto et al., 2024). Afoakwa et al. (2008) concluded that a low pH and a high content

of organic acids, mainly acetic acid, resulting from the fermentation of cocoa beans, undesirably affect the flavor of chocolates.

Regarding the WI, only when visual fat reaches a relevant level of $WI > 35$, the color perception of the product is affected, and the defect called fat bloom can be identified (Son et al., 2018). The evaluated dark-chocolate formulations, mainly with a higher percentage of coffee husk flour, showed WI results well below the recommended, meaning there was no migration of fat to the surface of the chocolates.

As to their microbiological quality, all formulations of dark chocolate with the addition of coffee husk flour were safe for consumption as there was no contamination by *Salmonella* or by total and thermotolerant coliforms. According to the International Commission on Microbiological Specifications for Food (Campagnollo et al., 2020), the stages of mixing, refining, conching, and tempering of chocolate have a low influence on the final microbiota of the product; even under conching temperatures between 60–80°C, microorganisms hardly develop due to the low a_w and high fat content of chocolate.

The average hardness values related to the breaking strength of the coffee flour husk added to the chocolate formulations were: 28.52 ± 4.70 for the concentration of 0% (control), 28.50 ± 3.24 for that of 2.5%, 28.19 ± 5.63 for that of 5.0% 31.40 ± 5.47 for that of 7.5%, and 29.82 ± 5.40 for that of 10%. The evaluated formulations did not show a significant linear or quadratic regression ($p > 0.05$) (Figure 2 A), that is, the addition of coffee flour husk did not cause a greater or lower hardness in the evaluated dark-chocolate formulations.

Hardness is one of the main parameters used to determine the structural quality of chocolate. According to Ostrowska-Ligęza et al. (2019), the hardness of a finished product represents the physical structure of a material and its mechanical and surface properties, being understood as the force necessary to reach a certain deformation. Konar (2013) added that hardness in chocolate formulations is correlated with the type and amount of fat used, type of sugar used, particle size and distribution, coating conditions, and shell temperature. These authors also found that textural properties, such as snap, occur when solid particles are coated with fat during coating, shelling, and tempering (Konar et al., 2023). In the present study, the addition of coffee husk flour to the formulations

did not cause changes in the texture of the chocolate, such as the cited snap effect, most likely because the time and temperature of the shell and tempering steps were standardized for all evaluated formulations.

According to the obtained results, only the hardness and cohesiveness parameters of the texture profile of the dark-chocolate formulations were significantly affected ($p < 0.05$) by the addition of coffee husk flour. Hardness specifically presented a decreasing linear effect (Figure 2 A), with values ranging from 78,21.02 to 93,893.61 N. This lower hardness with the increasing addition of coffee husk flour may be related to the lower amount of added sugar, responsible for textural characteristics in chocolate formulations (Vásquez

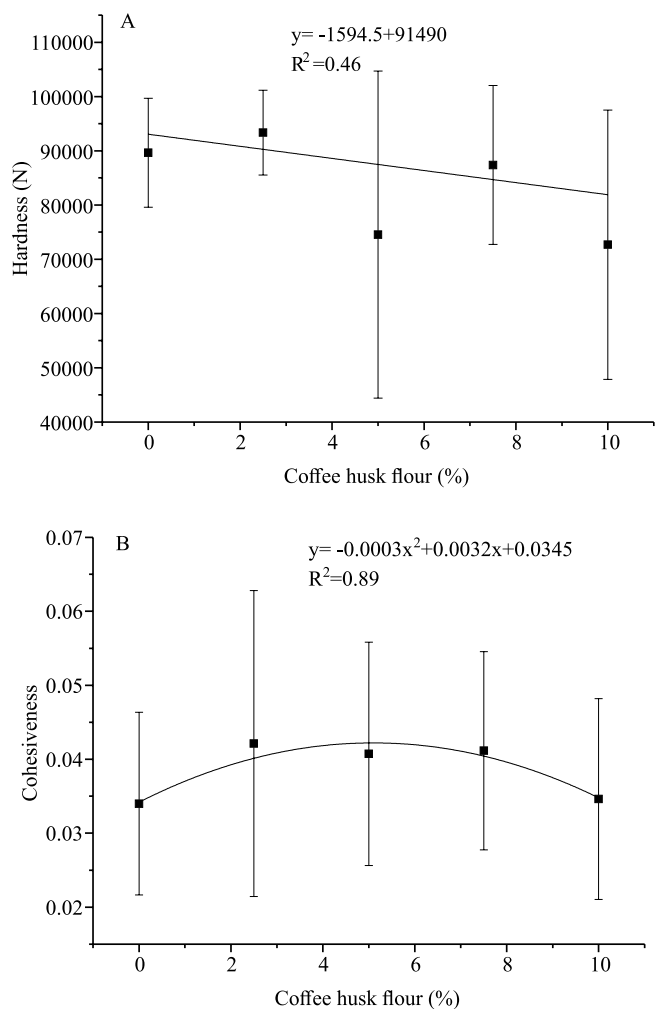


Figure 2. Linear behavior of hardness (A) and cohesiveness (B) among chocolate formulations with the addition of coffee (*Coffea arabica*) husk flour at 0, 2.5, 5, 7.5, and 10%.

et al., 2019). The greater softness of the chocolate as coffee husk flour was added may also be attributed to the latter's content of protein, a macromolecule related to the emulsifying activity that promotes the reduction of interfacial forces (Ashkezary et al., 2018).

Cohesiveness showed a quadratic effect, with values ranging from 0.034 to 0.042 (Figure 2 B). Cohesive forces are responsible for the tension of a surface, whose tendency is to resist rupture when placed under tension or stress, corresponding to the extent to which a material can be deformed before fracture (Ostrowska-Ligeza & Lenart, 2015). The parameters chewability (0.56 J) and adhesiveness (3.21 N), which represent the work required to overcome the force of attraction between the surfaces of the food and the one it is in contact with, did not present a significant linear or quadratic model ($p > 0.05$) for the evaluated formulations, that is, the addition of coffee husk flour did not promote a greater or lower chewability and adhesion between formulations.

The partial replacement of refined sugar by coffee husk flour affected the rheological parameters Casson plastic viscosity and Casson initial stress of the studied formulations, in addition to the linear correlation coefficients (R^2) and thixotropy (Table 3). The correlation coefficients indicated a good fit of the experimental data to the Casson model. Moreover, rotational test parameters, initial stress, plastic viscosity, and thixotropy did not show a significant linear or quadratic regression ($p > 0.05$).

According to the rheological analysis, the addition of coffee husk flour did not significantly interfere in the rheological properties of the produced chocolate. Glicerina et al. (2013) highlighted that dark chocolate presents a very complex behavior related to rheology, i.e., it has an apparent elastic limit and a plastic

viscosity strictly dependent on its manufacturing process. Although the values obtained in the present study are adequate for that parameter, the stress values are below the limit considered ideal for chocolate formulations, which must be between 10–200 Pa (Beckett et al., 2017).

Lončarević et al. (2018), investigating the effects of the addition of encapsulated blackberry extract on the rheological parameters of chocolate formulations, verified that there was a significant increase ($p < 0.05$) in the three evaluated parameters (viscosity, tension, and thixotropy). The authors obtained values that varied from 0.63 to 1.22 Pa.s for Casson viscosity and from 4.11 to 6.05 Pa for initial tension, results very close to those found in the present work.

Product's with lower tension values show a greater fluidity. According to Konar et al. (2014), the initial tension is related to the contact surface of the particles, meaning that, the smaller the particle size, the greater the interaction between them, increasing their surface area, which increases the liquid flow of chocolate. This easiness in flow observed in the present study may be attributed to the fact that the granulometry of the chocolate particles was within the ideal range of 20–25 μm (Böhme et al., 2016).

From the point of view of the energy requirements for chocolate processing, viscosity and thixotropy are relevant. The greater the thixotropy of a fluid, the easier it becomes to pump (Ardakani et al., 2014). Therefore, optimal values of thixotropy, together with an ideal yield-point value, are an indicative that coffee husk flour is a promising substitute for sucrose. Moreover, the functional characteristics of this by-product add value to chocolate, whose process conditions and stability maintenance are favored by the addition of

Table 3. Rheological parameters of chocolate formulations (F1–F5) with the addition of coffee (*Coffea arabica*) husk flour (CHF)⁽¹⁾.

Formulation	τ_{ca} (Pa)	η_{ca} (Pa.s)	R^2	Thixotropy (Pa. s ⁻¹)
F1	4.37±0.14	1.19±0.17	0.9942	4645.33±492.03
F2	3.51±0.46	1.08±0.21	0.9946	4064.80±670.48
F3	5.55±0.97	1.11±0.29	0.9919	4741.00±415.38
F4	6.74±0.84	1.84±0.56	0.9926	5234.33±253.98
F5	5.50±0.87	1.15±0.18	0.9942	4799.00±793.23
Model	-	-	-	-

⁽¹⁾F1, formulation with 0% CHF (control); F2, formulation with 2.5% CHF; F3, formulation with 5.0% CHF; F4, formulation with 7.5% CHF; F5, formulation with 10% CHF; τ_{ca} , initial Casson stress; η_{ca} , Casson's plastic viscosity; and R^2 , coefficient of determination (correlation coefficient). -, no significant regression parameter ($p > 0.05$) for the linear or quadratic model.

coffee husk flour, showing the positive effects of this material as a new ingredient in the chocolate industry.

Conclusions

1. The addition of up to 10% of coffee (*Coffea arabica*) husk flour promotes changes in the physicochemical, microbiological, textural, and rheological properties of the evaluated chocolate formulations, but without compromising their composition and quality.

2. The addition of coffee husk flour significantly increases moisture, ash, protein, and acidity, but decreases water activity in the chocolate formulations, which, despite these changes, remain in compliance with the standards established by the Brazilian legislation and the Codex Alimentarius.

3. Regarding the texture profile of the dark-chocolate formulations, the addition of coffee husk flour at 0.0, 2.5, 5.0, 7.5, and 10.0% only affects hardness and cohesiveness, with the softness of the chocolate formulations increasing as the coffee husk flour is added.

Acknowledgments

To Fundação de Amparo à Pesquisa do Estado da Bahia (FAPESB), for financial support (grant number DTE0004/2016); to Conselho Nacional de Desenvolvimento Científico e Tecnológico (CNPq), for doctoral scholarship; and to Professor Daniel and Ibicoara farm, for the used coffee husks.

References

- AFOAKWA, E.O. **Chocolate science and technology**. 2nd ed. Oxford: Wiley Blackwell, 2016. 507p. DOI: <https://doi.org/10.1002/9781118913758>.
- AFOAKWA, E.O.; PATERSON, A.; FOWLER, M.; RYAN, A. Flavor formation and character in cocoa and chocolate: a critical review. **Critical Reviews in Food Science and Nutrition**, v.48, p.840-857, 2008. DOI: <https://doi.org/10.1080/10408390701719272>.
- AGIBERT, S.A.C.; LANNES, S.C. da S. Dark chocolate added with high oleic peanut oil microcapsule. **Journal of the Science of Food and Agriculture**, v.98, p.5591-5597, 2018. DOI: <https://doi.org/10.1002/jsfa.9102>.
- AOAC. Association of Official Analytical Chemists. **Official Methods of Analysis of AOAC International**. 18th ed. Washington, 2010. 1v.
- ARDAKANI, H.A.; MITSOULIS, E.; HATZIKIRIAKOS, S.G. Capillary flow of milk chocolate. **Journal of Non-Newtonian Fluid Mechanics**, v.210, p.56-65, 2014. DOI: <https://doi.org/10.1016/J.JNNFM.2014.06.001>.
- ARYA, S.S.; VENKATRAM, R.; MORE, P.R.; VIJAYAN, P. The wastes of coffee bean processing for utilization in food: a review. **Journal of Food Science and Technology**, v.59, p.429-444, 2022. DOI: <https://doi.org/10.1007/S13197-021-05032-5>.
- ASHKEZARY, M.R.; YEGANEHZAD, S.; VATANKHAH, H.; TODARO, A.; MAGHSOUDLOU, Y. Effects of different emulsifiers and refining time on rheological and textural characteristics of compound chocolate. **Italian Journal of Food Science**, v.30, p.26-36, 2018. DOI: <https://doi.org/10.14674/IJFS-759>.
- BARIŠIĆ, V.; STOKANOVIĆ, M.C.; FLANJAK, I.; DOKO, K.; JOZINOVIĆ, A.; BABIĆ, J.; ŠUBARIĆ, D.; MILIČEVIĆ, B.; CINDRIĆ, I.; AČKAR, Đ. Cocoa shell as a step forward to functional chocolates-bioactive components in chocolates with different composition. **Molecules**, v.25, art.5470, 2020. DOI: <https://doi.org/10.3390/MOLECULES25225470>.
- BAYCAR, A.; KONAR, N.; GOKTAS, H.; SAGDIC, O.; POLAT, D.G. The effects of beetroot powder as a colorant on the color stability and product quality of white compound chocolate and chocolate spread. **Food Science and Technology**, v.42, e66220, 2022. DOI: <https://doi.org/10.1590/FST.66220>.
- BECKETT, S.T.; FOWLER, M.S.; ZIEGLER, G.R. (Ed.). **Beckett's industrial chocolate manufacture and use**. 5th ed. New Jersey: John Wiley & Sons, 2017. 760p. DOI: <https://doi.org/10.1002/9781118923597>.
- BLIGH, E.G.; DYER, W.J. A rapid method of total lipid extraction and purification. **Canadian Journal of Biochemistry and Physiology**, v.37, p.911-917, 1959. DOI: <https://doi.org/10.1139/o59-099>.
- BÖHME, B.; SYMMANK, C.; ROHM, H. Physical and sensory properties of chocolate made with lecithin of different origin. **European Journal of Lipid Science and Technology**, v.118, p.1839-1845, 2016. DOI: <https://doi.org/10.1002/EJLT.201600201>.
- BOLENZ, S.; GLÖDE, L. Technological and nutritional aspects of milk chocolate enriched with grape pomace products. **European Food Research and Technology**, v.247, p.623-636, 2021. DOI: <https://doi.org/10.1007/s00217-020-03651-4>.
- BOURNE, M.C. Sensory methods of texture and viscosity measurement. In: BOURNE, M.C. **Food texture and viscosity: concept and measurement**. 2nd ed. Cambridge: Academic Press, 2002. p.257-291. DOI: <https://doi.org/10.1016/b978-012119062-0/50007-3>.
- CAMPAGNOLLO, F.B.; FURTADO, M.M.; SILVA, B.S.; MARGALHO, L.P.; CARMINATI, J.A.; SANT'ANA, A.S.; NASCIMENTO, M.S. A quantitative risk assessment model for salmonellosis due to milk chocolate consumption in Brazil. **Food Control**, v.107, art.106804, 2020. DOI: <https://doi.org/10.1016/J.FOODCONT.2019.106804>.
- CARVALHO, J.C.S.; ROMOFF, P.; LANNES, S.C. da S. Improvement of nutritional and physicochemical proprieties of milk chocolates enriched with kale (*Brassica oleracea* var. *acephala*) and grape (*Vitis vinifera*). **Food Science and**

- Technology**, v.38, p.551-560, 2018. DOI: <https://doi.org/10.1590/fst.15018>.
- DE VUYST, L.; WECKX, S. The cocoa bean fermentation process: from ecosystem analysis to starter culture development. **Journal of Applied Microbiology**, v.121, p.5-17, 2016. DOI: <https://doi.org/10.1111/JAM.13045>.
- EISCHEN, J.-C.; WINDHAB, E.J. Viscosity of cocoa and chocolate products. **Applied Rheology**, v.12, p.32-34, 2002. DOI: <https://doi.org/10.1515/arh-2002-0020>.
- FDA. Food and Drug Administration. **Bacteriological analytical manual: Salmonella**. Available at: <https://www.fda.gov/food/laboratory-methods-food/bacteriological-analytical-manual-bam>. Accessed on: Jan. 10 2024.
- GLICERINA, V.; BALESTRA, F.; ROSA, M.D.; ROMANI, S. Rheological, textural and calorimetric modifications of dark chocolate during process. **Journal of Food Engineering**, v.119, p.173-179, 2013. DOI: <https://doi.org/10.1016/J.JFOODENG.2013.05.012>.
- KONAR, N. Influence of conching temperature and some bulk sweeteners on physical and rheological properties of prebiotic milk chocolate containing inulin. **European Food Research and Technology**, v.236, p.135-143, 2013. DOI: <https://doi.org/10.1007/s00217-012-1873-x>.
- KONAR, N.; ÖZHAN, B.; ARTIK, N.; DALABASMAZ, S.; POYRAZOGLU, E.S. Rheological and physical properties of inulin-containing milk chocolate prepared at different process conditions. **CyTA - Journal of Food**, v.12, p.55-64, 2014. DOI: <https://doi.org/10.1080/19476337.2013.793214>.
- KONAR, N.; POLAT, D.G.; DALABASMAZ, S.; ERDOGAN, M.; SENNER, S.; SARIKAYA, E.K. Effects of various milk powders on main quality parameters of cocoa butter substitute-based chocolate. **International Dairy Journal**, v.139, art.105571, 2023. DOI: <https://doi.org/10.1016/J.IDAIRYJ.2022.105571>.
- LEE, Y.-G.; CHO, E.-J.; MASKEY, S.; NGUYEN, D.-T.; BAE, H.-J. Value-added products from coffee waste: a review. **Molecules**, v.28, art.3562, 2023. DOI: <https://doi.org/10.3390/MOLECULES28083562>.
- LONČAREVIĆ, I.; PAJIN, B.; FIŠTEŠ, A.; ŠAPONJAC, V.T.; PETROVIĆ, J.; JOVANOVIĆ, P.; VULIĆ, J.; ZARIĆ, D. Enrichment of white chocolate with blackberry juice encapsulate: impact on physical properties, sensory characteristics and polyphenol content. **LWT**, v.92, p.458-464, 2018. DOI: <https://doi.org/10.1016/J.LWT.2018.03.002>.
- MARCHIoretto, C.; LUCCAS, V.; GORUP, L.F.; BORGES GOMES, R.A.; SIMIONATTO, E.; OLIVEIRA, K.M.P. de; ARAÚJO, R.P. de; ALTEMIO, Â.D.C.; PORZANI, G.B.; MARTELLI, S.M.; ARRUDA, E.J. de. Nutritional value and acceptability of chocolate with high cocoa content and green banana biomass. **LWT**, v.191, art.115667, 2024. DOI: <https://doi.org/10.1016/j.lwt.2023.115667>.
- MILLER, G.L. Use of dinitrosalicylic acid reagent for determination of reducing sugar. **Analytical Chemistry**, v.31, p.426-428, 1959. DOI: <https://doi.org/10.1021/ac60147a030>.
- MUHAMMAD, D.R.A.; SAPUTRO, A.D.; ROTTIERS, H.; Van de WALLE, D.; DEWETTINCK, K. Physicochemical properties and antioxidant activities of chocolates enriched with engineered cinnamon nanoparticles. **European Food Research and Technology**, v.244, p.1185-1202, 2018. DOI: <https://doi.org/10.1007/s00217-018-3035-2>.
- OSTROWSKA-LIGEŻA, E.; LENART, A. Influence of water activity on the compressibility and mechanical properties of cocoa products. **LWT**, v.60, p. 1054-1060, 2015. DOI: <https://doi.org/10.1016/j.lwt.2014.10.040>.
- OSTROWSKA-LIGEŻA, E.; MARZEC, A.; GÓRSKA, A.; WIRKOWSKA-WOJDYŁA, M.; BRYŚ, J.; REJCH, A.; CZARKOWSKA, K. A comparative study of thermal and textural properties of milk, white and dark chocolates. **Thermochimica Acta**, v.671, p.60-69, 2019. DOI: <https://doi.org/10.1016/J.TCA.2018.11.005>.
- PEIXOTO, R.R.A.; DEVESA, V.; VÉLEZ, D.; CERVERA, M.L.; CADORE, S. Study of the factors influencing the bioaccessibility of 10 elements from chocolate drink powder. **Journal of Food Composition and Analysis**, v.48, p.41-47, 2016. DOI: <https://doi.org/10.1016/J.JFCA.2016.02.002>.
- SCHANDA, J. **Colorimetry: understanding the CIE system**. New Jersey: John Wiley & Sons, 2007. 496p. DOI: <https://doi.org/10.1002/9780470175637.fmatter>.
- SON, Y.-J.; CHOI, S.; YOO, K.-M.; LEE, K.-W.; LEE, S.-M.; HWANG, I.-K.; KIM, S. Anti-blooming effect of maltitol and tagatose as sugar substitutes for chocolate making. **LWT**, v.88, p.87-94, 2018. DOI: <https://doi.org/10.1016/j.lwt.2017.09.018>.
- USDA. United States Department of Agriculture. Agricultural Marketing Service. **CID bakery products**. Available at: <https://www.ams.usda.gov/grades-standards/cid/bakery-products>. Accessed on: Jan. 10 2024.
- VÁSQUEZ, Z.S.; CARVALHO NETO, D.P. de; PEREIRA, G.V.M.; VANDENBERGHE, L.P.S.; OLIVEIRA, P.Z. de; TIBURCIO, P.B.; ROGEZ, H.L.G.; GÓES NETO, A.; SOCCOL, C.R. Biotechnological approaches for cocoa waste management: a review. **Waste Management**, v.90, p.72-83, 2019. DOI: <https://doi.org/10.1016/j.wasman.2019.04.030>.