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# Development of the dairy products incorporated with co-product bioactive compounds-rich as an alternative ingredient in the food industry

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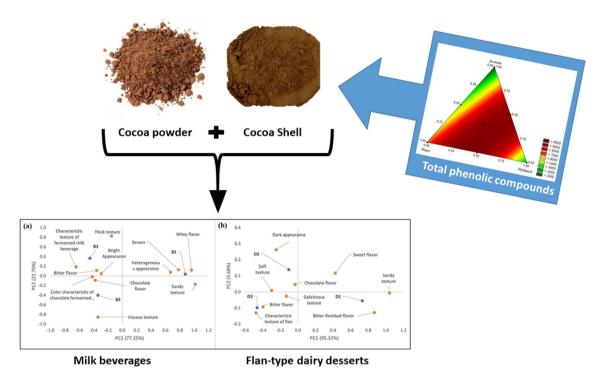
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**Abstract** The objective was to optimize the phenolic compounds extraction from cocoa shells using the simplex-centroid design with a mixture of solvents (water, methanol, and acetone) as its components, to prove the presence of these compounds and antioxidant activity. Also, the development of dairy products, such as milk beverages and dairy desserts, with bioactive compounds, through the replacement of cocoa powder by cocoa shell was studied and evaluated sensorially. The extraction optimization indicated that a solvent with 56.44% water, 23.77% methanol, and 19.80% acetone are ideal for maximizing the phenolic compounds. In addition, the cocoa shell showed a high antioxidant activity by the methods  $\beta$ -carotene/linoleic acid, FRAP, and phosphomolybdenum complex. The Check-All-That-Apply, Cochran's Q test, contingency analysis, and hierarchical cluster analysis allowed description characteristics of the dairy products and showed sensory differences between formulations with 100% cocoa shell and others. Both dairy products had good sensory acceptance in all attributes evaluated (appearance, flavor, texture, and overall impression), and their scores did not differ statistically by Tukey's test (p > 0.05). Thus, the cocoa shell is shown as an alternative substitute ingredient to be used in the dairy industry.

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## **Graphical abstract**



Keywords Agro-industrial co-product · Milk beverages · Flan-type dairy desserts · Sensory acceptance · CATA

# Introduction

The demand for food production has been promoting an increase in the generation of agro-industrial waste (Freitas et al. 2021). The seeds of the cacao (Theobroma cacao L.) tree, after going through the fermentation, drying, and roasting processes, are called almonds. The processes of peeling and fragmenting on the cocoa almonds separate the shell from the cotyledons, also called nibs (raw material for cocoa powder and its derivatives) (Okiyama et al. 2017; Soares and Oliveira 2022). Cocoa almond shell, commonly called cocoa shell, is a high-value and little-explored co-product with color and aroma like cocoa powder and contains several bioactive compounds with antioxidant activity, such as phenolic compounds (Martínez et al. 2012; Okiyama et al. 2017). It is a rich source of carbohydrates  $(178.0 \pm 0.9 \text{ g kg}^{-1})$ , dietary fibers (504  $\pm$  21 g kg<sup>-1</sup>), and proteins (150  $\pm$  2 g kg<sup>-1</sup>), showing a low lipid content  $(20.2 \pm 0.3 \text{ g kg}^{-1})$  (Martínez et al. 2012; Okiyama et al. 2017; Lessa et al. 2018; Barros et al. 2020; de Barros et al. 2021; Nogueira Soares Souza et al. 2022).

Due to its composition and technological potential, the cocoa shell has been used as an ingredient in food preparation. The cocoa shell was used as a substitute for wheat flour on chocolate cake, the development of cookies, food bars, chocolates, and chocolate muffins (Martínez-Cervera et al. 2011; Barros et al. 2020; de Barros et al. 2021; Barišić et al. 2022; Nogueira Soares Souza et al. 2022). The cocoa shell has been used in dairy industries as a food fortification/enrichment of milk and dairy products (Adinepour et al. 2022).

The milk beverage and flan type dairy dessert are examples of dairy products widely consumed in all age groups due to their sensory voluptuousness and, with this, the better their nutritional formulations with the direct addition of agro-industrial co-products, such as cocoa shell, become an alternative that can be explored, besides adding value to the final products tied to sustainability (Özer and Kirmaci 2010). In addition, the consumer market's choice and acceptance of a food product are influenced by the functional properties, the sensory quality of the products, and the clean label policy, which values the preparation of healthier and more natural foods. Therefore, it becomes feasible to tie studied new natural sources of bioactive compounds, antioxidant activity, and dietary fibers to be inserted in dairy products to the acceptance test and descriptive analysis Table 1Experimentaldesign applied to optimizethe extraction of bioactivecompounds (contents of thetotal phenolic compound) fromthe cocoa shell using water (A),methanol (B), and acetone (C)

Assays	A% (mL)	B% (ml)	C % (mL)	Total phenolic (mg GAE 100 g <sup>-1</sup> )	
				Observed	Predicted
E1	100 (10)	0 (0)	0 (0)	$6630.00 \pm 1.20$	6543.96
E2	0 (0)	100 (10)	0 (0)	$3174.27 \pm 1.65$	3088.23
E3	0 (0)	0 (0)	100 (10)	$1868.60 \pm 1.03$	1785.17
E4	50 (5)	50 (5)	0 (0)	$8928.61 \pm 1.02$	9262.64
E5	0 (0)	50 (5)	50 (5)	$8717.52 \pm 1.25$	9061.68
E6	50 (5)	0 (0)	50 (5)	$4089.02 \pm 1.01$	4433.18
E7	33 (3.3)	33 (3.3)	33 (3.3)	$9069.34 \pm 1.24$	8816.34
E8	33 (3.3)	33 (3.3)	33 (3.3)	$9061.53 \pm 1.51$	8816.34
E9	33 (3.3)	33 (3.3)	33 (3.3)	$9084.98 \pm 1.61$	8816.34
Critical points	0.5644	0.2377	0.1980	_	_
Ideal concentrations (%)	56.44	23.77	19.80	_	_

Check-All-That-Apply (CATA), aiming to obtain greater perceptions about the tastes and descriptive perceptions of consumers (Burkert et al. 2012; Varela and Ares 2012; Cruz et al. 2013; Meyners et al. 2016).

Therefore, this study aimed to optimize the extraction of phenolic compounds, aiming to prove the antioxidant activity of cocoa shells. In addition to utilizing cocoa shell, in place of cocoa powder, in the development of dairy products (milk beverages and dairy desserts) with bioactive compounds, as well as evaluating them sensorially (acceptance and sensory description).

# Material and methods

#### Materials

The roasted cocoa shell with a granulometry of approximately 40 microns was supplied by chocolate industries located in Ilhéus—Bahia, Brazil. Pure cocoa powder and the other ingredients used in the formulations of food products were purchased at the local market in Lavras—Minas Gerais, Brazil. All raw materials were transported to the Fruit and Vegetable Post-Harvest Laboratory of the Federal University of Lavras, Lavras—Minas Gerais, Brazil, until further analysis and processing of dairy products.

# Extraction of the bioactive compounds from the cocoa shell

# Experimental design

The experimental design described by Scheffé (1963) called simplex-centroid was performed to evaluate the effect of solvents on the extraction of bioactive compounds (contents of the total phenolic compound—TPC) from cocoa shells (Table 1). The three components of the blend evaluated in this study were water  $(x_1)$ , methanol  $(x_2)$ , and acetone  $(x_3)$ . The proportions for each solvent were expressed as a fraction of the mixture, and for each treatment combination, the sum of the component proportions was equal to 100%, as shown in Eq. (1).

$$\Sigma X_i = x_1 + x_2 + x_3 = 100 \% = 10 \text{ mL}$$
(1)

The extract with the optimized concentrations of the solvents was submitted to antioxidant activity analyses ( $\beta$ -carotene/linoleic acid, FRAP, and phosphomolybdenum complex).

#### Preparation of extracts

The extracts were prepared according to the methodology adapted from Larrauri et al (1997). Ten grams of cocoa shell and 10 mL of the solvent mixture equivalent to each treatment (Table 1) were packed in centrifuge tubes and vortexed for one minute. After 20 min of rest in a dark environment, the homogenate was centrifuged at 4 °C for 15 min at 21,952 g. Subsequently, the extract was filtered through Whatman paper Grade 1, a qualitative filter paper with pore size of 11  $\mu$ m, and store in amber glass bottles at -18 °C for further experiments.

Determination of the contents of total phenolic compounds and antioxidant activity

The Folin-Ciocalteu methodology (Waterhouse 2002) was used to determine the total phenolic content (TPC). Briefly, 0.5 mL of extract from each sample was added to tubes containing 2.5 mL of 10% Folin-Ciocalteu solution. Then, 2 mL of 4% sodium carbonate solution was mixed, followed by incubation for 2 h at room temperature. Finally, the absorbance was measured at 750 nm using a microplate

spectrophotometer (EZ Read 2000, Biochrom®— Cambridge, UK). The results were expressed as mg of gallic acid equivalent (GAE) per 100 g of sample.

The antioxidant activity by the method of  $\beta$ -carotene/ linoleic acid was performed according to the methodology described by Rufino et al (2010). Briefly, 50 µL of the  $\beta$ -carotene solution was mixed with 40 µL of linoleic acid, 530 µL of Tween 40, and 1 mL of chloroform, homogenized, and rotary evaporated to remove the chloroform. Then, under constant agitation, approximately 80 mL of distilled water saturated with oxygen was added to the mixture. In a test tube, 2.7 mL of this solution was added to 0.3 mL of extract from each sample, the absorbance was measured spectrophotometrically at 470 nm, the tubes were incubated in a water bath at 40 °C for 120 min, and then a new reading was performed. The results were expressed in the percentage of protection.

The Ferric Reducing Antioxidant Power (FRAP) assay was performed according to the methodology described by Rufino et al (2010). Briefly, 90  $\mu$ L of extract from each sample was homogenized with 270  $\mu$ L of distilled water and 2.7 mL of FRAP reagent and incubated in a water bath at 37 °C for 30 min. Then, the absorbance was measured spectrophotometrically at 470 nm, and the results were expressed in  $\mu$ M ferrous sulfate per g of sample.

The formation of the phosphomolybdenum complex was assessed according to the method described by Prieto et al (1999). Briefly, 0.3 mL of extract from each sample was added to a test tube with 3 mL of solution reagent (0.6 M sulfuric acid, 28 mM sodium phosphate, and 4 mM ammonium molybdate) and incubated in a water bath at 95 °C for 60 min. After cooling, the absorbance was measured at 695 nm, and the results were mg of ascorbic acid equivalent (AAE) per 100 g of sample.

### Formulations of the dairy products

#### Experimental design

For the development of dairy products (milk beverages and dairy desserts), the Entirely Randomized Design (ERD) was applied with three treatments, as described in Table 2. The sum of the proportion of cocoa shell and cocoa powder in the treatments totals 120 g for the dairy drink and 40 g for the dairy-fermented dessert (Table 2).

#### Milk beverage

 Table 2
 Experimental design applied to milk beverage (B) and Dairy dessert (D) developments

Treatment	Cocoa shell (%)	Cocoa powder (%)
B1 or D1	100	0
B2 or D2	50	50
B3 or D3	0	100

powder, and 15 g of neutral alloy stabilizer were used, plus equivalent amounts of cocoa shell and cocoa powder for each treatment (Table 2). All ingredients were mixed and taken for heat treatment at 90 °C for 5 min to obtain the milk beverage. Then, the developed products were cooled (between 5 and 10 °C), stored in plastic packaging, and kept under refrigeration at 5 °C until it was served at the sensorial analysis (approximately 24 h).

## Dairy dessert

The formulation of dairy dessert was carried out according to Chandan and Kilara (2015), with modifications. To produce a batch, 500 mL of whole cow's milk, 57.5 g of refined sugar, 40 g of whole cow milk powder, 30 g of powdered neutral gelatin and 2.5 g of the neutral alloy were also used. To obtain flan-like dairy desserts, cocoa shell, and cocoa powder individually or mixed (proportions described in Table 2) have been added to the milk powder, sugar, and gelatin powder, forming a dry mixture. Then, this mixture was dissolved in the fluid milk at 40 °C and kept under agitation for the perfect dissolution of the ingredients. After complete homogenization of the ingredients, the mixture was heated to 65 °C for 30 min to promote the heat treatment of the product and hydration of the gelatin. Finally, the developed products were cooled (between 5 and 10 °C), stored in plastic packaging, and kept refrigerated at 5 °C until they were served at the sensorial analysis (approximately 24 h).

#### Sensorial analysis

A total of 95 and 92 volunteers, aged between 18 and 50 years, were screened at the Federal University of Lavras (UFLA) for the sensorial analysis of milk beverages (43 men and 52 women) and flan-type dairy desserts (39 men and 53 women), respectively. Frequent consumers of these two dairy products participated in the study and underwent the acceptance test and Check-All-That-Apply (CATA) contained in the same evaluation sheet. The panelists evaluated the milk beverage and flan-type dairy desserts in two

different sessions, with a sample of each treatment being presented, totaling 3 samples for each product. The presentation of the samples followed an experimental design of completely balanced blocks.

#### Acceptance test

For the evaluation of the samples regarding acceptance, the nine-point hedonic scale was used, with variation between 1 (I did not like) and 9 (I liked very much). For each sample, consumers evaluated the attributes "appearance", "flavor", "texture", and "overall impression." The experiments were carried out in random order.

# Check-all-that-apply (CATA)

For the evaluation, each attribute (Table 4) present in the form was presented to consumers before starting the analysis. Then, they were instructed to evaluate the samples and identify the characteristics in each one, as shown in Table 4, being able to mark as many alternatives as needed (Varela and Ares 2012). The characteristics presented to consumers were raised in a previous focus group of 12 assiduous evaluators who consume milk beverages and flan-type desserts.

#### Statistical analysis

The results of the contents of total phenolic compounds and total flavonoids of the cocoa shell were used to obtain a contour plot and the equation of the statistical model that best fit. The simplex-centroid design generating mathematical models was submitted to ANOVA (p < 0.05) using the Statistica 12.0 software (Statsoft USA). The statistical parameters evaluated were the validity of the models, the significance of lack of fit and regression, and the coefficient of determination ( $\mathbb{R}^2$ ). On the other hand, data related to the determinations of  $\beta$ -carotene/linoleic acid, FRAP, and phosphomolybdenum complex were obtained from the extract with the concentrations of optimized solvents in three replications and triplicate as a form of characterization of the antioxidant activity of cocoa shells.

The acceptance test scores (appearance, taste, texture, and general impression) were submitted to the Tukey mean test (p < 0.05). In the analysis of CATA data, the frequency with which consumers observed each characteristic in the different samples evaluated sensorially was accounted for, thus originating the contingency matrix. The Cochran's Q test was used to verify significant differences between the samples for each characteristic, thus indicating which ones characterized the samples (Meyners et al. 2013). The significant characteristics were submitted to Correspondence Analysis (CA) and Hierarchical Cluster Analysis (HCA) using the program R version 4.0.2 and the SensoMineR and FactoMineR packages.

# **Results and discussion**

# Total phenolic compound content and antioxidant activity

The recovered extracts were analyzed according to the total phenolic compounds (TPC), and the results are shown in Table 1. A regression analysis was applied to evaluate the significant interactions ( $p \le 0.05$ ). Thus, it was possible to observe that regarding the TPC, the independent variables were water (A), methanol (B), and acetone (C). Even with the application of ANOVA, it was found that the regression model for the TPC was significant in the 95% confidence interval ( $p \le 0.05$ ), with determination coefficients ( $\mathbb{R}^2$ ) equal to 0.9917. The experimental design analysis obtained

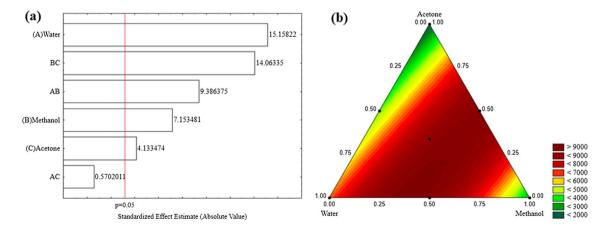


Fig. 1 Pareto chart **a** and contour curves **b** illustrate the combined effects of the water, methanol, and acetone on the total phenolic compounds extracted from cocoa shell

the predictive model (Eq. 2), respecting the significative interactions shown in Fig. 1a. The values of the critical points and, consequently, of the ideal concentrations of the solvents tested for greater extraction of bioactives compounds are found in Table 1 and were obtained from Eq. (2).

$$TPC = 6555.62A + 3097.56 B + 1791.89 C + 17635.44 AB + 19402.39 AC + 7768.44 BC (2)$$

Figure 1b shows the contour curves, where the dark red area is considered the most representative point, thus allowing the visualization of the interactions of the TPC regarding the concentrations (%) of the water, methanol, and acetone. When the phenolic compounds were extracted in water, the cocoa shell presented higher values, indicant that the phenolic compounds of this species are water-soluble (Leite et al. 2021). It was founded that TPC values varied from 2210 to 7330 mg 100 g<sup>-1</sup> in the cocoa shell extracted in water at different conditions of temperature ranging from 30 to 100 °C, time 5 to 90 min, acidity 0–2%, and solid-to-liquid ratio 0.02 to 0.05 g mL<sup>-1</sup> (Rebollo-Hernanz et al. 2021).

From the optimized concentrations were obtained results of  $\beta$ -carotene/linoleic acid (% protection), ABTS<sup>\*+</sup> (µg of Trolox g<sup>-1</sup>), FRAP (µM of ferrous sulfate g<sup>-1</sup>), and Phosphomolybdenum Complex (mg AAE 100 g<sup>-1</sup>) equal to 99.03 ± 0.26, 90.02 ± 0.49, 366.34 ± 0.84, and 180.29 ± 7.86, respectively. The results showed strong antioxidant potential. High antioxidant activities of the extracts are also attributed to the roasting cocoa process, i.e., Maillard reaction products lead to the formation of high molecular weight polymers or co-polymers, which present strong antioxidant activity (Ali et al. 2015; Mazzutti et al. 2018). Thus, justifying the direct addition of cocoa shell enriches food formulations with functional aspects of bioactive compounds and antioxidant activity.

#### Sensorial analysis

Tables 3 and 4 describe the frequency of consumer perception of the sensory characteristics and Cochran's Q test of each sample of milk beverages and flan-type dairy dessert, respectively, in addition to presenting the respective average scores for the attributes of appearance, flavor, texture and overall impression of the acceptance test.

#### Acceptance test

The application of sensorial analysis using the affective acceptance test through the hedonic scale aimed to evaluate the acceptance of two dairy products developed regarding appearance, flavor, texture, and overall impression. According to the scores obtained from the milk beverages (Table 3), sample B1 (100% cocoa shell) differed statistically by the Tukey test ( $p \le 0.05$ ) from samples B3 (100% cocoa powder) and B2 (50% cocoa shell and 50% cocoa powder) in all attributes studied. Samples B2 and B3 were statistically equal for almost all attributes (p > 0.05) except for texture. For flan-type dairy dessert, it was also observed that the formulation with 100% cocoa shell (D1) differed ( $p \le 0.05$ ) from the other samples (D2 and D3), which did not differ statistically from each other for all attributes, as described in Table 4.

About all attributes evaluated (Tables 3 and 4), the average scores obtained by the formulations with 100% cocoa shell of the milk beverage (B1) and the flan-type dairy dessert (D1) were in the range of 3.05 (texture) to 4.78 (appearance) and 5.72 (texture) to 7.05 (appearance), respectively. In the case of formulations with 100% cocoa powder (B3 and D3), the average scores were higher than 7 for both developed products. For products made with 50% cocoa shell and 50% cocoa powder of the milk beverage (B2), the scores ranged between 6.80 (texture) and 7.65 (appearance), and those of the milk dessert (D2) were between 6.88 (flavor) and 7.90 (appearance).

Therefore, it can also be inferred that the presence of 100% cocoa shells in the elaborated products (formulations B1 and D1) promoted the reduction of the scores referring to the attributes, but mainly in the texture, resulting in low overall impression scores for the milk beverage  $(3.73 \pm 1.96)$  and the flan-type dairy dessert  $(6.17 \pm 1.61)$ , resulting in the lower acceptance between the formulations. On the other hand, the formulations with 100% cocoa powder (B3 and D3) obtained high overall impression scores  $(7.55 \pm 1.22 \text{ and } 7.41 \pm 1.21$ , respectively), resulting in the best acceptance between the formulations in the case of dairy products formulated with cocoa shell and cocoa powder mixed in equal proportions (B2 and D2), they also presented good acceptance with overall impression scores close to those of samples B3 and D3 ( $7.09 \pm 1.52$  and  $7.25 \pm 1.27$ , respectively).

Thus, it is recommended that consumption be based on the objectives, sensory and functional aspects that consumers seek in products since cocoa powder has sensory acceptance and cocoa shells can provide nutritional enrichment with dietary fibers and bioactive compounds with antioxidant activity, a fact observed in other studies when they used this co-product in formulations of various food products (Martínez-Cervera et al. 2011; Barros et al. 2020; de Barros et al. 2021; Barišić et al. 2022; Nogueira Soares Souza et al. 2022).

#### Check-all-that-apply (CATA)

CATA is a simple method that allows collecting information about the perception of consumers about the characteristics Table 3 Contingency table of the CATA sensory evaluation of dairy beverages formulated with cocoa shell (B1), cocoa powder (B3), and a mixture of both (B2), the *p* value obtained in the Cochran Q test for the characteristics and the acceptance test scores (appearance, flavor, texture, and overall impression), following the nine-point hedonic scale, 1 (I did not like) to 9 (I liked very much)

Characteristic	Samples				
	B1 B2		B3	(Cochran's Q test)	
Heterogeneous appearance	66	19	21	< 0.001	
Dark appearance	30	45	36	0.099*	
Bright appearance	21	45	52	< 0.001	
Light brown	25	5	7	< 0.001	
Characteristic color of chocolate milk beverage	17	63	65	< 0.001	
Chocolate flavor	21	72	64	< 0.001	
Sweet flavor	26	34	36	0.301*	
Acid flavor	4	1	2	0.368*	
Bitter flavor	9	22	29	0.001	
Whey flavor	44	6	8	< 0.001	
Bitter residual flavor	22	18	20	0.783*	
Sandy texture	88	25	1	< 0.001	
Viscous texture	9	57	7	< 0.001	
Thin texture	16	1	42	< 0.001	
Characteristic texture of milk beverage	2	38	57	< 0.001	
Acceptance test scores**					
Appearance	$4.78 \pm 2.04^a$	$7.65 \pm 1.06^{\rm b}$	$7.77 \pm 1.13^{b}$		
Flavor	$4.05\pm2.24^{\rm a}$	$7.26 \pm 1.61^{b}$	$7.55 \pm 1.37^{\rm b}$		
Texture	$3.05 \pm 1.89^{\rm a}$	$6.80 \pm 1.65^{\mathrm{b}}$	$7.34 \pm 1.56^{\circ}$		
Overall impression	$3.73 \pm 1.96^{a}$	$7.09 \pm 1.52^{b}$	$7.55 \pm 1.22^{\circ}$		

\*Non-significant Characteristic by the Cochran Q test at 5% significance

\*\*Scores means followed by the same letters in the same row do not differ from each other by the Tukey test at 5% significance

Characteristic	Samples			
	D1	D2	D3	(Cochran's Q test)
Heterogeneous appearance	49	49	56	0.459*
Brown	56	42	42	0.068*
Dark appearance	11	41	29	< 0.001
Characteristic color of chocolate flan	40	49	51	0.263*
Chocolate flavor	40	60	55	0.012
Sweet flavor	23	20	9	0.015
Bitter flavor	18	51	77	< 0.001
Acid flavor	3	1	1	0.449*
Residual flavor	40	14	6	< 0.001
Sandy texture	65	25	0	< 0.001
Gelatinous texture	34	54	62	< 0.001
Soft texture	15	39	46	< 0.001
Characteristic texture of flan	7	25	42	< 0.001
Acceptance test scores**				
Appearance	$7.05 \pm 1.59^{\rm a}$	$7.90\pm0.97^{\rm b}$	$7.90 \pm 1.16^{\rm b}$	
Flavor	$5.88 \pm 1.92^{\rm a}$	$6.88 \pm 1.52^{\rm b}$	$7.00 \pm 1.60^{\rm b}$	
Texture	$5.72 \pm 1.85^{\rm a}$	$7.07 \pm 1.53^{\rm b}$	$7.61 \pm 1.33^{b}$	
Overall impression	$6.17 \pm 1.61^{a}$	$7.25 \pm 1.27^{b}$	$7.41 \pm 1.21^{b}$	

\*Non-significant Characteristic by the Cochran Q test at 5% significance

\*\*Scores means followed by the same letters in the same row do not differ from each other by the Tukey test at 5% significance

Table 4Contingency table ofthe CATA sensory evaluationof flan type dairy dessertformulated with cocoa shell(D1), cocoa powder (D3), anda mixture of both (D2), thep-value obtained in the CochranQ test for the characteristicsand the acceptance test scores(appearance, flavor, texture andoverall impression), followingthe nine-point hedonic scale, 1(I did not like) to 9 (I liked verymuch)

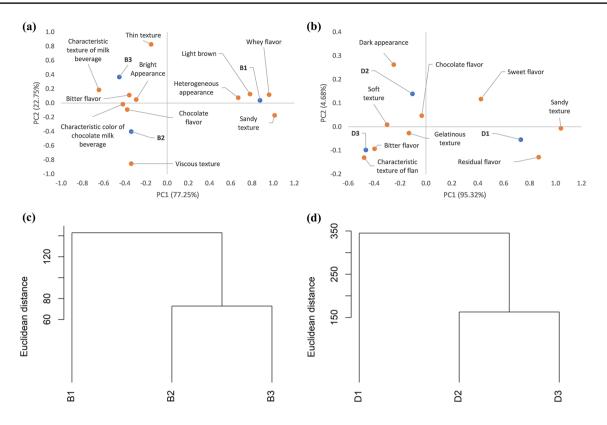


Fig. 2 Correspondence Analysis (CA)  $\mathbf{a}$ ,  $\mathbf{b}$  and the Hierarchical Cluster Analysis (HCA)  $\mathbf{c}$ ,  $\mathbf{d}$  used in the CATA analysis of the milk beverage (B1, B2, and B3) and the flan type milk dessert (D1, D2,

and D3), respectively, formulated with cocoa shell (B1 or D1), cocoa powder (B3 or D3) and a mixture of both (B2 or D2)

of the products developed. Thus, the p-value obtained from Cochran's Q test aimed to identify significant differences ( $p \le 0.05$ ) between the characteristics and, therefore, which may have impacted the acceptance test for the different dairy treatments and proceed to the correspondence analysis (CA).

First, the data related to s contingency tables (Tables 3 and 4) were submitted to the chi-square test ( $X^2$ ), which indicated that the associations between the samples and the characteristics were significant, thus validating the CATA analysis. According to Cochran's *Q* test, the characteristics of dark appearance and sweet, acid, and bitter residual flavors were not significant for the sensory description of the milk beverage (p > 0.05), as shown in Table 3. The characteristics not significant for flan-type dairy desserts were heterogeneous appearance, brown, characteristic color of chocolate flan, and acid flavor because the values obtained were higher than 0.05 (Table 4). Based on this, the Correspondence Analysis (CA) did not consider these characteristics.

Figure 2a, b represent the two-dimensional maps of Correspondence Analysis (CA) of beverages and dairy desserts, respectively, which were used to explain contingency tables (Tables 3 and 4). According to CA, it is observed that the product space presents low dimensionality, considering that most of the data of the milk beverage and dairy dessert were represented, respectively, by dimensions 1 (x-axis), 77.25, and 95.32%, while dimensions 2 (y-axis) represented only 22.75 and 4.68%, respectively, totaling 100% of the variability of the data for both dairy products.

The main description in the CA's (Fig. 2a, b for both types of products developed was the separation by the differences between the formulations with cocoa powder (B2, B3, D2, and D3) and those with the absence (B1 and D1). Thus, it is inferable that the partial addition of cocoa shell originated beverage (B2) and dairy dessert (D2) with the same characteristics identified for their respective formulations with 100% cocoa powder (B3 and D3) due to the proximity of the points in the x-axis mapping (Fig. 2 a and b). Therefore, consumers described samples B2 and B3 as having a bright appearance, characteristic color of chocolate milk beverage (Fig. 2a). Also, the sample B2 can be described with a viscous texture, and B3 with thin textures, due to

the distance of the points in the y-axis mapping. For the dairy dessert, samples D2 and D3 were described with dark appearance, chocolate, and bitter flavors, and with characteristic textures of flan, soft and gelatinous texture (Fig. 2b). In the case of products developed with 100% cocoa shell, sample B1 (beverage) was characterized by a heterogeneous and light brown appearance, sandy texture and whey flavor (Fig. 2a), and sample D1 was described with sandy texture and sweet and residual flavors (Fig. 2b).

The dietary fiber content of the cocoa shells explains that consumers have observed a sandy texture for both types of products developed with their highest concentration, which according to the scores referring to the texture of the acceptance test, is not well accepted at least. In addition, this coproduct also has color and flavor like that of cocoa powder, justifies the same characteristics conferred on all formulations with pure cocoa powder or mixed with cocoa shell, both for beverages (B2 and B3) and for the developed dairy desserts (D2 and D3) (Okiyama et al. 2017; Jan et al. 2018).

Hierarchical Cluster Analysis (HCA), widely known as dendogram, was applied in the CATA results of the milk beverage and dessert are represented in Fig. 2c, e, d, aiming to group the samples according to the similarities found in the descriptions of sensory characteristics obtained with input coordinates in the first and second dimensions of sensory maps. In addition, each cluster presented in Fig. 2c, d was formed by at least two treatments that are close to the sampling plan (Cruz et al. 2013; Barros et al. 2020). Based on this assumption and considering that the higher the line connecting two groups, the later its grouping was made and that the height of the line connecting two clusters is proportional to its distance, it is concluded that the samples of milk beverage B2 and B3 presented similar characteristics and were slow to be grouped with formulation B1 (Fig. 2c). The same behavior was observed for the flan-type dairy dessert, whose samples D2 and D3 belong to the same group, differing from the D1 sample.

From the above, it can be inferred that the HCAs (Fig. 2c, d) confirmed the data and the groups present in Fig. 2a, b, respectively, considering that there was a clear separation between the formulations with cocoa powder and without, besides corroborating the results and statistical differences observed in the acceptance test for all attributes studied (Tables 3 and 4).

# Conclusions

For the conditions evaluated in this study, the simplex-centroid design and the mixing surface effectively optimize the extraction of phenolic compounds with different solvents, thus obtaining their ideal concentrations. The optimized extract made it possible to conclude that the cocoa shell has high antioxidant activity and a significant content of total phenolic compounds. The sensory acceptance test allowed us to infer, for all the attributes studied, that despite the presence of 100% cocoa shell promoting low acceptance of the formulations of both elaborated dairy products, its mixture with cocoa powder obtained good acceptance, similarly observed for those with 100% cocoa powder. The CATA analysis allowed an effective sensory description of the developed milk beverages and flan-type dairy desserts, demonstrating the differences in characteristics between treatments with the presence and absence of cocoa powder. Therefore, it is concluded that the partial replacement of cocoa powder with cocoa shell in the development of dairy products is feasible, aiming at a possible nutritional enrichment with bioactive compounds and with antioxidant activity, in addition to adding value to the final product.

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**Data availability** The authors confirm that the data supporting the findings of this study are available within the article. The datasets generated during and/or analysed during the current study are available from the corresponding author on reasonable request.

#### Declarations

**Conflict of interest** The authors declare that they have no conflict of interest.

**Ethics approval** This article does not contain any studies with human or animal subjects.

**Consent to participate** All authors agree to participate.

**Consent for publication** All authors agree to publish.

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