Physicochemical characterization of national and commercial cocoa butter used in Brazil to make chocolate

Caracterização físico-química de manteigas de cacau nacional e comercial utilizadas no Brasil para fabricação de chocolate

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Abstract
Cocoa butter is one of the most important ingredients in chocolate production as it is responsible for important characteristics of chocolates such as hardness, snap, mold shrinkage, fat bloom stability and melting. It appears that factors such as the geographic area and climate where cocoa is grown directly affect the chemical composition of cocoa butter, so fruits grown in Africa and Asia have greater thermal stability. In this sense, two samples of cocoa butter, a Brazilian one from the south of Bahia and a commercial one (consisting of a mixture of butters from different origins) were studied and compared. The results obtained in this study showed that Brazilian cocoa butter had a higher content of Saturated Fatty Acids (SFA) and a higher content of unsaturated triacylglycerols compared to commercial cocoa butter. It also showed a faster polymorphic transition and a higher maximum solids content compared to commercial cocoa butter, indicated by isothermal crystallization analysis. Based on the analyzes carried out, it was found that this cocoa butter studied, from the Forasteiro amelonado species and coming from several farms in the south of Bahia, presented greater thermal stability in relation to commercial cocoa butter, i.e., different from that presented in other studies in the literature.

Keywords: Thermal behavior; Fatty acids; Triacylglycerols; Isothermal crystallization; Cooling curve; Solid curve.

Resumo
A manteiga de cacau é um dos ingredientes mais importantes na fabricação de chocolate, pois é responsável por características importantes desse produto como dureza, snap, contração na desmoldagem, estabilidade ao fat bloom e derretimento. Fatores como a área geográfica e o clima onde o cacau é cultivado afetam diretamente a composição química da manteiga de cacau, de modo que frutos cultivados na África e América apresentam maior estabilidade térmica. Nesse sentido, duas amostras de manteiga de cacau, uma brasileira proveniente do sul da Bahia e uma comercial (constituída por uma mistura de manteigas de diversas origens) foram estudadas e comparadas. Os resultados obtidos neste estudo mostraram que a manteiga de cacau brasileira apresentou maior teor de ácidos graxos saturados e maior teor de triacilgliceróis disaturados (S2U), em relação à manteiga comercial.
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HIGHLIGHTS:
• Oleic, stearic and palmitic acids are the most predominant fatty acids of cocoa butter
• The Brazilian sample presented characteristics close to the commercial sample
• It is not recommended to use only the solids curve for selecting cocoa butters for application in chocolates

1 Introduction

Cocoa butter is one of the most important raw materials used in chocolate production, being one of the ingredients with the highest value-added product, corresponding to 25 and 36%, or more, of the value of the final product. It is also one of the most important ingredients, where it represents the continuous phase of chocolate and acts as a dispersing matrix for cocoa, milk, and sugar solids. The importance of this ingredient is also related to the quality present in the final product, since it is responsible for the characteristics of hardness, snap (breakdown at room temperature), complete melting in the mouth, contraction during demolding and chocolate shine, in addition to the product stability to fat bloom (Suri & Basu, 2022).

Among the most predominant fatty acids in the structure of cocoa butter, oleic acid (C18:1), stearic acid (C18:0) and palmitic acid (C16:0) stood out (Jin et al., 2021). The literature has shown that factors such as geographic area and climatic conditions where cocoa is grown directly influence the composition of triacylglycerols (TAGs) and the fatty acid profile of cocoa butter, thus modifying its melting point and solids profile. According to Beckett (2008), the closer to the equator the cocoa was grown, the harder the butter would be. In addition, cocoa butter from fruits grown in South America contains a higher content of oleic acid than those from fruits grown in Africa and Asia (Beckett, 2008).

Studies have been shown that Brazilian cocoa butter had a lower content of monounsaturated triacylglycerols and a higher content of di and polyunsaturated triacylglycerols (Ribeiro et al., 2012). In this way, it has a softer texture, which interferes with the thermal stability of chocolates produced with this raw material and, consequently, contributes to a reduction in its commercial value. According to Beckett (2008), while the cocoa butter from Malaysia had a Solid Fat Content (SFC) of 81%, the Brazilian one had only approximately 65% of this content. For this reason, most of the cocoa butter used by Brazilian industries in chocolate making is imported, as they are more resistant to heat, but more expensive. As an alternative to this issue, many cocoa processors in Brazil sell a blend of cocoa butters from different origins, but this blend has a percentage of only 20 to 30% of national butter in the mixture, which is considered a low value. In this way, this work aimed to evaluate the physicochemical characteristics of Brazilian cocoa butter sample, from the south of Bahia and commercial one (blend of cocoa butter from different origins).
2 Material and methods

2.1 Material

The Brazilian cocoa butter used in this study was granted by the Cocoa Innovation Center (CIC), being extracted from cocoa beans of the Forasteiro amelonado species, from several farms located in the south of Bahia. Commercial cocoa butter was obtained from the company Olam Cocoa, Brazil, that it was a commercial cocoa butter, made up of a mixture of butters from different origins, the proportion of which was not known.

2.2 Physicochemical characterization

2.2.1 Fatty acids composition

The fatty acid composition was determined according to the AOCS Ce 2-66 method (American Oil Chemists' Society, 2009). A Capillary Gas Chromatography (CGC) model Agilent 6850 Series GC System and an Agilent DB-23 capillary column (50% cyanopropyl) - methylpolysiloxane, with dimensions of 60 m, internal diameter of 0.25 mm and 0.25 μm thickness of movie were used. The identification of fatty acids was performed by comparing the retention time obtained with the respective standards. The analysis was performed in triplicate. The conditions used for chromatograph operation were: column flow = 1.0 mL/min.; linear velocity = 24 cm/sec; detector temperature: 280°C; injector temperature: 250 °C; oven temperature: 110 °C for 5 min, 110 – 215 °C (5 °C/min), 215 °C for 24 min.; carrier gas: Helium; and injected volume: 1.0 μL.

The identification of the fatty acids present in the sample was carried out by comparing the retention times of the peaks with those of the respective fatty acid standards. The quantification of fatty acids was determined through the normalization of the peak areas obtained by the chromatogram, expressed in mass percentage. The analysis was carried out in triplicate.

2.2.2 Triacylglycerol composition

The triacylglycerol composition was determined according to the AOCS Ce 5-86 methodology (American Oil Chemists’ Society, 2009). A CGC model Agilent 6850 Series Gc System and capillary column DB-17 HT Agilent Catalog: 122-1811 (50% phenyl – ethylpolysiloxane) with dimensions of 10 m, internal diameter of 0.25 mm and 0.15 μm film were used. The chromatograph operating conditions were: column flow = 1.0 mL/min.; linear velocity = 40 cm/sec; detector temperature: 375 °C; injector temperature: 360 °C; Oven temperature: 250 – 350 °C (5 °C/min), 350 °C for 20 minutes; carrier gas: Helium; injected volume: 1.0 μL, split 1:100; and sample concentration: 10 mg/mL tetrahydrofuran (THF). TAGs were identified through the comparison of retention times, according to the procedures of Antoniosi Filho et al. (1995). The quantity was determined by peak area normalization. The analysis was carried out in triplicate.

2.2.3 Solid Fat Content (%SFC)

The SFC of cocoa butters was determined according to the AOCS Cd 16b-93 method, using a Bruker pC120 Minispec Nuclear Magnetic Resonance Spectroscopy (NMR Spectroscopy) (Silberstreifen, Rheinstetten, Germany) (American Oil Chemists’ Society, 2009). First, the sample was tempered using Duratech Tcon 2000 dry baths (Carmel, USA), where it was subjected to 0 °C for 90 min, 26 °C for 40 h and, again, 0 °C for 90 min. After this preparation, the sample was kept for 1 h at the temperatures analyzed (10, 20, 25, 30 and 35 °C) before taking the reading. The analysis was carried out in triplicate.
2.2.4 Melting point

The melting point of the samples was obtained through the solids curve, corresponding to the temperature at which the sample presented 4% of solids.

2.2.5 Isothermal crystallization

The sample was melted and kept at 70 °C for 1 h in a TCON 2000 dry bath, with a temperature range of 0 to 70 °C (Duratech, USA), to erase its crystalline history. After this period, the sample was kept in a thermal bath at 17.5 °C ± 0.5 °C and readings were taken every 1 min in a Bruker pc120 Minispec NMR Spectroscopy (Rheinstetten, Germany), until reaching equilibrium (250 min). The crystallization kinetics was characterized according to the induction period data, which corresponds to the time required for the beginning of the formation of a stable nucleus and maximum SFC (SFCmax). The methodology used for the isothermal crystallization analysis was proposed by Ribeiro et al. (2009). The analysis was carried out in duplicate.

2.2.6 Cooling curve

The cooling curves of the cocoa butter samples were obtained on a Multitherm TC temperature meter (Buhler, Switzerland). The sample was heated to 70 °C and packed in aluminum capsules, specific to the equipment. The equipment's cooling compartment was kept at a temperature of 17.6 °C for the analysis. The result was evaluated using the Buhler Crystallization Index (BCI) (Zeng et al., 2021).

2.3 Statistical analysis

The results obtained were submitted to the t test for two samples to determine significant differences in the means at a significance level of 5% ($p \leq 0.05$), using the Minitab 19 software.

3 Results and discussion

3.1 Fatty acids composition

The results presented in Table 1 indicate that the fatty acids present in greater amounts in cocoa butter are palmitic (C16:0), stearic (C18:0) and oleic (C18:1) acids, which represent more than 90% of the fatty acids in the sample. The levels obtained for these fatty acids are close to the values found in the literature, which vary from 24 to 27% for palmitic acid, 33 to 36% for stearic acid and 32 to 35% for oleic acid (Ribeiro et al., 2012). According to Beckett (2008), the fact that cocoa butter is relatively simple in relation to the main fatty acids makes this fat have very interesting physical properties, such as the fact that it melts in a small temperature range, and melts quickly in the mouth, besides being responsible for the glow and snap of chocolates.

It was also verified that the national cocoa butter had a higher content of Saturated Fatty Acids (SFA) and a lower content of Unsaturated Fatty Acids (UFAs) in relation to commercial cocoa butter. The literature has shown that factors such as geographic area and climatic conditions where cocoa is grown directly influence the composition of TAGs and the fatty acid profile of cocoa butter, modifying its melting point and solid profile. According to Beckett (2008), the closer to the equator the cocoa was grown, the harder the butter would be. In addition, cocoa butter from fruits grown in South America contains a higher content of oleic acid than those from fruits grown in Africa and Asia (Beckett, 2008).
Table 1. Fatty acids composition (%) present in commercial and national cocoa butter (CB).

| Fatty acids     | National CB     | Commercial CB  |
|-----------------|-----------------|----------------|
| C14:0 – myristic| 0.1 ± 0.00      | 0.1 ± 0.02     |
| C16:0 – palmitic| 26.7 ± 0.02     | 25.1 ± 0.01    |
| C16:1 – palmitoleic| 0.3 ± 0.03   | 0.3 ± 0.01     |
| C17:0 – margaric| 0.3 ± 0.00      | 0.3 ± 0.00     |
| C18:0 – stearic | 33.6 ± 0.03     | 33.3 ± 0.24    |
| C18:1 – oleic   | 34.3 ± 0.03     | 35.1 ± 0.10    |
| C18:2 – linoleic| 2.9 ± 0.02      | 4.1 ± 0.26     |
| C18:3 – linolenic| 0.2 ± 0.00   | 0.3 ± 0.02     |
| C20:0 – arachidic| 1.2 ± 0.00    | 1.1 ± 0.01     |
| C20:1 – gadoleic| 0.1 ± 0.01      | 0.1 ± 0.00     |
| C22:0 – behenic | 0.2 ± 0.00      | 0.2 ± 0.00     |
| C24:0 – lignoceric| 0.1 ± 0.00  | 0.0 ± 0.02     |
| ∑ Saturated     | 62.2 ± 0.05     | 60.2 ± 0.21    |
| ∑ Unsaturated   | 37.8 ± 0.09     | 39.8 ± 0.14    |

3.2 TAG composition

The TAGs are the major components of oils and fats, and they are responsible for the properties of these lipids, such as spreadability, melting in the mouth, crystallization profile and type of polymorph. Table 2 present the TAG composition of South of Bahia and commercial cocoa butter. For both butters, it was possible to obtain 14 types of TAGs. The TAGs of the types Palmitic-Oleic-Palmitic (POP), Palmitic-Oleic-Stearic (POSt) and Stearic-Oleic-Stearic (StOSt) were the predominant ones, where they presented a total of 84.5% in national butter and 78.5% in commercial butter. These values agree with the range of these triglycerides reported in the literature, which varies from 72 to 93% (Jin et al., 2021).

However, contrary to what was expected, commercial cocoa butter had a higher content (9.8%) of TAGs of the types Palmitic-Oleic-Oleic (POO) and Stearic-Oleic-Oleic (StOO) compared to national butter (6.6%). In a study carried out by Ribeiro et al. (2012), the authors found a higher percentage of POO and StOO in Bahia (8.5%) and Espirito Santo (9.3%) butters in relation to the commercial butters evaluated (around 5%). Furthermore, national cocoa butter presented lower content of TAGs trisaturated (0.9%) compared to commercial cocoa butter (1.3%). These TAGs have a higher melting point and can accelerate the fat crystallization.

Table 2. Triacylglycerol composition present in commercial and national cocoa butter (CB).

| TAG (%)     | National CB     | Commercial CB  |
|-------------|-----------------|----------------|
| PPP         | 0.2 ± 0.0       | 0.2 ± 0.1      |
| PPSi        | 0.4 ± 0.2       | 0.5 ± 0.1      |
| POP         | 20.6 ± 0.2      | 17.6 ± 0.2     |
| PLP         | 1.9 ± 0.2       | 2.1 ± 0.0      |
| POSt        | 40.6 ± 0.8      | 37.4 ± 0.9     |
| POO         | 3.3 ± 0.3       | 4.5 ± 0.0      |
| PLSt        | 2.7 ± 0.2       | 3.5 ± 0.4      |
| PLO         | 0.4 ± 0.1       | 0.7 ± 0.4      |
| PStSt       | 0.3 ± 0.5       | 0.6 ± 0.3      |
| StOSt       | 23.3 ± 0.2      | 23.5 ± 0.4     |
| StOO        | 3.3 ± 0.4       | 5.3 ± 0.1      |
| SILO        | 1.5 ± 0.0       | 2.0 ± 0.0      |
| OOO         | 0.2 ± 0.3       | 0.9 ± 0.2      |
| StoA        | 1.3 ± 0.0       | 1.3 ± 0.4      |
| ΣS3         | 0.9 ± 0.7       | 1.3 ± 0.5      |
| ΣS2U        | 90.4 ± 1.5      | 85.3 ± 2.3     |
| ΣU2S        | 8.5 ± 0.8       | 12.5 ± 0.4     |

P: Palmitic (saturated); St: Stearic (saturated); O: Oleic (monounsaturated); A: Arachidic (saturated); L: Linoleic (diunsaturated).
Triacylglycerols: S₃ = trisaturated, S₂U = disaturated–monounsaturated and U₂S = diunsaturated–monosaturated.
3.3 Solid Fat Content (%SFC)

One of the indications of the fat melting profile is through the solids curve, which relates the solid fraction of fat in relation to its total mass (SFC) when the sample is subjected to different temperatures. The solids curves obtained for the national CB and commercial CB are shown in Figure 1. At all temperatures evaluated, Bahia cocoa butter presented a lower solid fat content in relation to commercial cocoa butter, presenting a similar behavior to that obtained by Ribeiro et al. (2012). The authors evaluated the SFC of different commercial cocoa butters, where, at 10 °C, this content ranged from 83.9 to 85.4%, while for national butter, the value obtained by the authors was 77.4%. In the present work, at the same temperature, it was possible to obtain a value of 80% for national butter and 86.4% for commercial butter.

In a study carried out by Shukla (2006), the authors obtained a variation in SFC from 74.8 to 83.7%, at a temperature of 25 °C, for butters from Ghana, India, Nigeria, Malaysia, and Sri Lanka. In the present work, at 25 °C, a value of 57.5% was obtained for national butter and 65.4% for commercial butter, lower values compared to imported butters. Ribeiro et al. (2012) also obtained values like those obtained in this study for Bahia and commercial butter. The authors stated that national cocoa butters, especially those obtained in Espírito Santo (Brazil), have greater softness in relation to imported cocoa butters and, therefore, to be used in tropical regions, they need to be mixed with butters from foreign origins to obtain higher levels of SFC at various temperatures. Importantly, other analyses, such as isothermal crystallization and thermal behavior, must also be considered before determining whether one cocoa butter is harder than the other.

![Figure 1. Solid Fat Content (SFC) of national cocoa butter and commercial cocoa butter (CB).](image)

Through the solids curve, it was possible to quantify several physical parameters of fats. A very important parameter that can be extracted from the solids curve is the variation of the solid fat content from 25 to 35 °C, called ∆S. This parameter is widely used in the food industry to assess the quality of cocoa butter (Luccas, 2001). It is desirable that fats used for chocolate production have high values of ∆S, thus indicating that the fat has good melting properties in the mouth, associated with the release of flavor and sensation of freshness. The solid fat content at 25 °C represents the hardness of the fat. Fats with higher values of S25 °C and lower values of ∆S25 °C – 35 °C are characterized by being harder and with greater resistance to heat, respectively. The presence of solid fat at temperatures above 35 °C is undesirable, since it causes a sensory perception of waxy fat, and is easily perceptible during tasting (Luccas, 2001). For cocoa butter to be used in the manufacture of chocolates, it must have a solid fat content above 50% at 25 °C and must not have high solids content at 35 °C (Luccas, 2001).

The values obtained for S25 °C, ∆S25 °C – 35 °C, and S35 °C are shown in Table 3. The cocoa butters showed significant differences (p < 0.05) concerning the fat content solid at 25 °C and 35 °C, where commercial butter showed higher SFC values than Bahia cocoa butter. Regarding the ∆S values, it was found that commercial cocoa butter presented a higher value compared to Bahia cocoa butter, with a significant difference between the samples at a significance level of 5%. Both butters had low solids content at 35 °C, as expected for cocoa butter samples.
Table 3. Comparison between the physical characteristics of national cocoa butter (Bahia) and commercial cocoa butter (CB).

| Samples           | $\Delta S_{25 ^\circ C}$ | $\Delta S_{25 ^\circ C} - 35 ^\circ C$ | $S_{35 ^\circ C}$ |
|-------------------|--------------------------|----------------------------------------|------------------|
| National CB (Bahia) | 57.51 ± 0.04b            | 57.30 ± 0.16h                         | 0.21 ± 0.17b     |
| Commercial CB     | 65.38 ± 0.19a            | 63.96 ± 0.34a                         | 1.42 ± 0.15a     |

Values with different letters in the same column are significantly different ($p \leq 0.05$).

3.4 Melting point

From the solids curve, it was also possible to obtain the value of the melting temperature of the samples, which corresponded to the temperature at which the solid fat content of the sample was equivalent to 4%. It is considered that at this value the fat is completely in the liquid state, with no residual fraction of solids (Ribeiro et al., 2012). The melting temperature obtained for each of the butters evaluated is shown in Table 4. According to Shukla (2006), the melting temperature of cocoa butter could vary in a range of 32 to 35 °C. This variation in temperature is not relevant because cocoa butter has a relatively uniform chemical composition, since the predominant TAGs in cocoa butter (POP, POS and SOS) have a uniform chemical structure, where oleic acid occupies the second position (Suri & Basu, 2022). The values determined for the national CB and commercial CB are within this temperature range, and both values were very close.

Table 4. Melting point of national and commercial cocoa butter (CB).

| Samples       | Melting point (°C) |
|---------------|--------------------|
| National CB   | 34.40 ± 0.04       |
| Commercial CB | 34.66 ± 0.02       |

3.5 Isothermal crystallization

The graph of Figure 2 shows the curves obtained from the isothermal crystallization of the analyzed cocoa butters. The isothermal crystallization analysis was performed at 17.5 °C, according to Marangoni & McGauley (2003). It is possible to verify that, in both cocoa butters, it was possible to obtain a crystallization with an induction period called “$\tau_1$” and in a second step, a second induction period called “$\tau_2$” was obtained. In addition, the crystallization curve showed a sigmoidal shape. Ribeiro et al. (2012) also observed this behavior of cocoa butter. It follows that the first induction time ($\tau_1$) is related to the time required for the formation of a crystal nucleus. The presence of this plateau in the cocoa butter isothermal crystallization is related to its polymorphic transition, where the transition from a less stable to a more stable form occurs.
Table 5 presents the results of the two induction periods ($\tau_1$ and $\tau_2$) and the maximum solids content obtained for both butters. It is found that national cocoa butter had a higher $\tau_1$ value than commercial one. This behavior may be related to the TAG composition of the fat. According to Suri & Basu (2022), different types of cocoa butter submitted to the same crystallization condition may have different crystallization rates. This fact can be attributed to the minority components present in cocoa butter, such as glycolipids, phospholipids and tri-saturated TAGs, which can act as “seeds” or crystallization nuclei and accelerate the crystallization of fat (Suri & Basu, 2022). Based on the data of the TAG composition of the cocoa butters in this study, it was possible to note that commercial cocoa butter had a TAG content (S3) of approximately 40% more than national cocoa butter (Bahia). Such TAGs had a higher melting point, thus being the first to crystallize, therefore the commercial butter started to crystallize first.

Regarding the values of $\tau_2$, it appears that the national cocoa butter showed a faster polymorphic transition than the commercial one. This fact can be attributed to the TAG composition of cocoa butters, since national cocoa butter had a higher content of symmetrical TAGs (90.4%) in relation to commercial cocoa butter (85.3%), and these TAGs are responsible for accelerating the polymorphic transition of these fats. Regarding the maximum solids content, it appears that, although the national cocoa butter has presented itself as softer by the solids curve analysis, when evaluated at a temperature of 17.5 °C, it could reach equilibrium with a higher solids content when compared to commercial cocoa butter.

| Samples        | $\tau_1$ (min) | $\tau_2$ (min) | SFCmax (%) |
|----------------|----------------|----------------|------------|
| National CB    | $22.00 \pm 0.71^a$ | $55.00 \pm 1.41^b$ | $73.77 \pm 0.42^a$ |
| Commercial CB  | $14.75 \pm 0.35^b$ | $62.00 \pm 5.60^b$ | $59.00 \pm 0.71^b$ |

Values with different letters in the same column are significantly different ($p \leq 0.05$).

### 3.6 Cooling curve

The cooling curve provides information on the crystallization of cocoa butter, making it possible to assume whether it has the necessary or desired hardness for making chocolates. The analysis is based on a calorimetric principle, where heat is released from cocoa butter during cooling. The analysis is performed at a temperature of 17.6 °C to maximize the expansion of the cocoa butter exothermic reaction. The BCI index was created by Buhler (manufacturer of the equipment used) and evaluates the cooling curve of cocoa butter as a whole, considering the nucleation and crystals growth. Through this value it is possible to know the crystallization of the raw material used is suitable for chocolate production. According to the manufacturer, fats with indexes above 4.0 are recommended for chocolate production, as they have high hardness and rapid crystallization. However, some companies use cocoa butter to make chocolates with BCI values up to 3.5.

Table 6 presents the values determined for the cocoa butters in this study. It is verified that national cocoa butter (Bahia) presented a BCI value of 5.4, which is characterized as a very hard fat, with rapid crystallization. The commercial cocoa butter evaluated had an index of 3.5, thus characterizing a butter with moderate crystallization capacity (Zeng et al., 2021). Furthermore, the data obtained for crystallization start temperatures also showed that domestic cocoa butter started to crystallize before commercial cocoa butter, suggesting it is a harder cocoa butter.

| Samples       | $T_n$ (°C) | $t_n$ (min) | $Q$ (°C/min) | BCI |
|---------------|------------|-------------|--------------|-----|
| National CB   | 18.8       | 16.7        | 0.22         | 5.4 |
| Commercial CB | 20.2       | 9.2         | 0.12         | 3.5 |

$T_n$ corresponds to the temperature at the beginning of crystallization; $t_n$ corresponds to the time of start of crystallization; $Q$ corresponds to the crystallization rate.
4 Conclusion

The results obtained in this study showed that Brazilian cocoa butter had a higher content of SFA and a higher content of unsaturated TAGs compared to commercial cocoa butter. In addition, national cocoa butter also showed a faster polymorphic transition and a higher SFCmax compared to commercial cocoa butter. Although Brazilian cocoa butters are characterized by being softer when compared to butters originated from countries such as Ghana and Malaysia, it could be noted in the sample studied, Forasteiro amelonado species, which came from several farms located in the South of Bahia, that this species presented physicochemical characteristics close to the commercial sample, used by national chocolate industries, unlike the results presented in other studies. This indicated that, probably, the South region of Bahia has some species consisting of significant thermal resistance.

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