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Obtaining 70% cocoa dark chocolate enriched with ginger oleoresin obtained via supercritical CO_2 extraction

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***Abstract*—The development of innovative products based on cocoa and natural additives with functional properties that benefit health has been studied in recent years. The use of natural extracts obtained with green technology is a sustainable alternative to be considered part of the chocolate production chain. Previous studies have shown that ginger oleoresin has a high antioxidant capacity; thus, its use in small quantities as a replacement for cocoa butter can improve the properties of chocolate. In this study, three formulations of 70% cocoa dark chocolate were developed with 0.026%, 0.078%, and 0.129% ginger oleoresin. The viscosity, texture, thermal properties, color, oxidative stability index, sensory properties, total polyphenol content, and antioxidant capacity were measured. The results revealed that the viscosity, melting point, and antioxidant capacity of the evaluated chocolates increased with increasing ginger oleoresin content, indicating that the thermal properties of the enriched chocolates improved. Furthermore, the color and whiteness index properties were not altered. On the other hand, the chocolate with 0.026% ginger oleoresin was the only formulation with sensory acceptability similar to that of the control; thus, it can be considered a technological prototype for industrial scaling.**

Keywords: Ginger oleoresin, chocolate, supercritical fluid, antioxidant, cocoa.

I. INTRODUCTION

Chocolate making is a traditional process carried out by many Peruvian microentrepreneurs and cocoa producers. In the Amazonas region of Peru, we can find a fine aroma cocoa called Criollo cocoa, which is used in the artisanal process of chocolate production [1]. There are different types of chocolate, ranging from white chocolate and milk chocolate to dark chocolate, among others. Many consumers prefer to consume 70% cocoa dark chocolate because of its beneficial antioxidant properties, such as its ability to protect the cardiovascular system [2]. Efforts to develop innovative chocolates that are well-received on the market have led to the use of many ingredients different from those currently used in chocolate. In this context, chocolates that incorporate extracts of exotic fruits, freeze-dried berry peels, vegetable oils, and other similar components have been developed [3-5]. However, the incorporation of these new ingredients results in variations in the quality parameters of the chocolates, such as melting and crystallization temperatures, texture, rheology, and oxidative stability. Studies conducted on the formulation of functional chocolates with Peruvian Criollo cocoa, which incorporate freeze-dried berries and sacha inchi oil, have shown an improvement in the antioxidant properties of the final product [5-6]. In this study, ginger oleoresin was used as an innovative ingredient to produce dark chocolate with 70% cocoa, and its quality properties were verified. Cocoa paste was mixed with cocoa butter, sugar, and ginger oleoresin at three different concentrations (0.026%, 0.078%, and 0.129%). A control chocolate mixture without ginger oleoresin was also prepared. The aim of this procedure was to obtain chocolate with good sensory acceptability, heat resistance, oxidation resistance, and improved bioactive properties. This prototype is intended to generate innovative proposals for the chocolate industry.

II. METHODOLOGY

A. Development of 70% cocoa dark chocolate based on Peruvian Criollo cocoa fortified with ginger oleoresin obtained by supercritical CO_2 extraction

1. Ingredients

For chocolate production, Criollo cocoa beans were provided by the Aprocam Cooperative in Bagua, Amazonas, Perú. Ginger oleoresin was obtained via supercritical CO_2 extraction at 50 °C and 250 bar following the methodology described by Reference [7].

2. Chocolate Production

The chocolate manufacturing phases included selection, roasting, winnowing, grinding, blending, conching, refining, tempering, molding, and packaging.

2.1. Selection

For this process, cocoa beans with an average diameter of 1.5 cm were selected (Fig. 1).

Fig 1. Selection of cocoa beans.

2.2. Roasting

The beans were roasted at 110 °C for 1 h in a tray dryer (Fischer Agro, Peru) and finally cooled (Fig. 2).

Fig. 2. Roasted cocoa beans.

2.3. Winnowing

The husks, which constitute the outer covering of the cocoa beans, were removed using a sheller (Imsa, DC-C, Peru) to obtain cocoa nibs (Fig. 3).

Fig. 3. Winnowing of roasted cocoa beans.

2.4. Grinding

The cocoa nibs were ground using a grinding machine (Prosol SAC, Tritur-50, Peru) until a cocoa paste was obtained. It was then packaged in bags and stored until it was mixed with the other ingredients.

2.5. Blending

Before all the ingredients were mixed, a premix was prepared containing 9 g of cocoa butter and 1.1654 g of ginger oleoresin. This premix was incorporated into the mixture of ingredients according to the experimental conditions of the formulas Ch1, Ch3, Ch5, and the control ChC. The ginger oleoresin obtained via supercritical CO_2 extraction and the premix of cocoa butter and ginger oleoresin (Formula 1) are shown in Fig. 4.

Fig. 4. Ginger oleoresin and premix of Formula 1

The melted cocoa paste was mixed with cocoa butter, sugar, and ginger oleoresin at three different concentrations (0.026%, 0.078%, and 0.129%). The proportions of these ingredients varied depending on the type of chocolate being manufactured. A control chocolate (ChC) mixture was also prepared without the addition of ginger oleoresin (Table 1). A single control formulation was used as a reference for comparison. The percentage of ginger oleoresin was selected to evaluate its effect on the physicochemical, bioactive, and sensory properties of chocolate. Reference [8] highlights that bioactive components in functional foods are effective at low concentrations and that their incorporation should consider the stability of the bioactive component, as well as the sensory properties of the product. Each formulation was prepared in a single batch because of the limited availability of ginger oleoresin obtained by supercritical fluid extraction. This work is an exploratory study evaluating the effects of the addition of ginger oleoresin as a bioactive component on the bioactive, physicochemical, and sensory properties of these compounds.

Table 1. Formulas of 70% cocoa dark chocolate with different percentages of ginger oleoresin

Formula	Cocoa paste g	Sugar g	Cocoa butter g	Ginger oleoresin g	Percentage of ginger oleoresin %
Ch1	350	125	24.8705	0.1295	0.026
Ch3	350	125	24.6115	0.3885	0.078
Ch5	350	125	24.3526	0.6474	0.129
ChC	350	125	25	0	0

Note: The codes used in the formula show the original experimental design; intermediate formulations Ch2 and Ch4 were not included.

2.6. Refining and conching

In total, 500 g of chocolate mass was refined in a conching-refining machine (Premier, PG508, India). During this process, the ingredients passed through stone rollers that completely refined the mixture for 24 hours. The temperature of the mixture reached 60 °C (Fig 5).

Fig. 5. Refining and conching.

2.7. Tempering

The chocolate temperature was reduced to 32 °C in a tempering machine (ChocoVision Corporation, Revolution V, New York, USA) to ensure the crystallization of the cocoa butter. It was then molded without exceeding 35 °C.

2.8. Molding

The liquid chocolate mass was poured into molds. The molds were placed in a low-temperature freezer where the chocolate hardened, acquiring its final shape (Fig. 6).

2.9. Packaging

The finished chocolates were wrapped in aluminum foil and stored under refrigeration (4 °C) until quality analysis was performed.

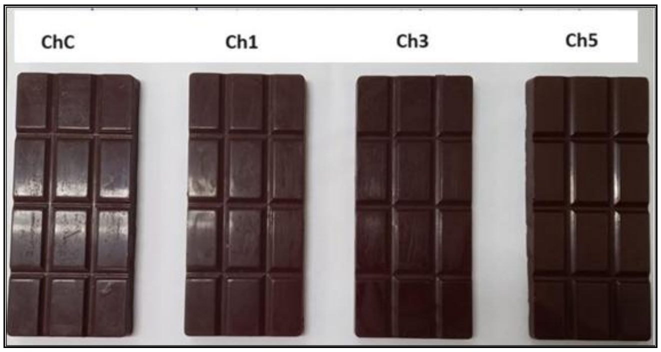

Fig. 6. Chocolates with the addition of ginger oleoresin and the control.

B. Physicochemical, bioactive and sensorial characterization of 70% cocoa dark chocolate based on Peruvian Criollo cocoa fortified with ginger oleoresin obtained by supercritical CO_2 extraction

1. Materials and Reagents

All the reagents were of analytical grade and were purchased from Sigma Aldrich, USA.

2. Viscosity

The viscosity of the chocolates was measured using a rheometer (Anton Paar, Model MCR 92, Austria) equipped with a concentric cylinder. The samples were first melted at 50 °C for 60 minutes and placed in a beaker. Rheological measurements were taken at 40 °C. The measurement cycle began with preconditioning at 40 °C for 60 s. The measurements were processed using the instrument's software (RheoCompass, version 1.33.540), which is based on the Casson model [6]. Analyses were performed in triplicate.

3. Texture

The texture was determined using a texture analyzer (Brookfield, CT3 Texture Analyzer, USA) equipped with a 25 kg load cell and a TA2/1000 stainless steel probe. All chocolates were kept at room temperature (20 °C) for 1 h before measurement. The following parameters were used: pretest speed, 0.5 mm/s; test speed, 2.0 mm/s; product volume, 0.7 mm × 55 mm × 50 mm; penetration depth, 3 mm; time set, 1–2 min; test speed, 50 mm/s; load cell, 25 kg; and trigger force, 20 g [6]. The analyses were performed in triplicate.

4. Thermal Properties

To obtain the crystallization and melting temperatures of the cocoa butter and its mixtures with ginger oleoresin, the procedure of Reference [9] was used with some modifications. The crystallization and melting temperatures of the samples were determined using a differential scanning calorimeter (DSC) (TA Instruments, Discovery DSC 2500, New Castle, USA). A sample measuring approximately 5 mg was placed in a low-mass Tzero aluminum container and hermetically sealed with a sample press. An empty container was used as a reference to obtain the initial configuration. To eliminate any crystalline memory and ensure homogeneity, the samples were heated from room temperature (16 °C) to 60 °C and held at that temperature for 10 minutes. The sample was then cooled at 10 °C/min to -35 °C and held at that temperature for 10 min, followed by heating at 10 °C/min to 60 °C. Using Trios software, version 5.1, the melting (TFus) and crystallization (TCrist) temperatures were determined, corresponding to the crystallization (exothermic) and melting (endothermic) peak temperatures of each thermogram (heat flow vs. temperature). The initial (TInicio) and final (TFin) temperatures of each peak were also determined. The reference plot determined the enthalpy of each process (crystallization or melting) as the peak area [6]. Analyses were performed in triplicate.

5. Color

The color parameters of the chocolates were measured in triplicate using a Minolta CR-400 colorimeter (Minolta Co. Ltd., Japan), with D65 illumination and a standard observer angle of 2°. The measurement was performed by placing the colorimeter's aperture on the chocolate tablet. The L*, a*, and b* values (Cielab) were recorded directly from the instrument. The whiteness index (WI) values were calculated [10]. Analyses were performed in triplicate.

6. Oxidative Stability

The Rancimat method was used. Three grams of 99.9% polyethylene glycol (Sigma Aldrich, USA) and one gram of chocolate were mixed in the reaction vessel of the instrument. The vessel was placed in a Rancimat 892 Professional instrument (Metrohm, Switzerland). The following parameters were programmed (Metrohm, 2024): 1 g of sample weight, 60 mL of the measuring solution, 110 °C temperature, autotemperature correction, 20 L/h air flow, and 300 µS/cm conductivity, with endpoints. The oxidative stability index (OSI) is expressed as the ″induction time (IP)″, which is the time at which the chocolate reaches the maximum oxidation rate, indicated by an increase in the conductivity of deionized water, when dry air passes through the chocolate [11]. All analyses were performed in triplicate.

7. Sensory Analysis

In Reference [6], a sensory profile of chocolate was developed to determine consumer preference for all the chocolates compared to the control. Sensory analysis was conducted with voluntary participants. To obtain the panelists' consent to participate in the sensory tests, they were informed about the research objectives and the composition of the chocolate through an introductory talk held before the test.

They were then given the questionnaire and wrote their name to indicate their consent to participate. The samples evaluated were safe for consumption and prepared under standard food safety conditions. Approximately 50 students (n=50) from the Faculty of Engineering and Agricultural Sciences (FICA) were selected as untrained panelists for the sensory test (30 men and 20 women). The panelists' ages ranged from 20 to 30 years. The chocolate samples were kept at room temperature (18 ± 2 °C) for 1 hour before evaluation. Each panelist then tasted all the fortified chocolates, including the control. For this purpose, four types of chocolate samples (2.5 × 1.3 × 0.7 cm each) were randomly distributed, with three copies of each. Each panelist completed a questionnaire to obtain a score ranging from 1 ("I dislike it a lot") to 9 ("I like it a lot") using hedonic scales for attributes such as flavor, color, shine, and acceptability.

8. Total phenolic compounds

The total phenolic content (TPC) in the chocolates was determined according to the procedure of Reference [6]. In total, 0.05 mL of diluted extract and 0.45 mL of distilled water (Milli-Q EQ 7000, Merck, France) were mixed with 2.5 mL of Folin–Ciocalteu phenol reagent diluted 1:10, followed by 2 mL of 7.5% (w/v) sodium carbonate. After 5 minutes at 50 °C, the absorbance was measured at 760 nm (Unico, S2100, USA). TPC was estimated from a gallic acid standard curve, and the results are expressed as mg gallic acid equivalents/g of sample (mg GAE/g). The samples were analyzed in triplicate.

9. Antioxidant capacity

The antioxidant capacity was determined according to Reference [6]. The samples were analyzed in triplicate.

10. Statistical Analysis

One-way analysis of variance with 5% significance (α=0.05) was used to determine the effect of incorporating ginger oleoresin at three concentrations. The ANOVA assumptions were evaluated through residual analysis. A representative example is shown for antioxidant capacity (DPPH) (Fig. 7). The residual plot indicates that the normality, homogeneity of variance, and independence of residuals are reasonably satisfactory. Tukey's test for mean difference was then used to determine significant differences. The software used was Minitab 17®.

III. DISCUSSION

Prototypes of 70% cocoa dark chocolate were successfully developed with the addition of ginger oleoresin, and their physicochemical and bioactive properties were evaluated. Ginger oleoresin was incorporated into the chocolates in three proportions 0.026%, 0.078%, and 0.129%, which were coded as Ch1, Ch3, and Ch5, respectively. A control chocolate (ChC) was also developed without the addition of ginger oleoresin, and served as a comparison standard. On the basis of the results shown in Table 2a, Table 2b, Table 3, and Table 4, the following conclusions can be drawn.

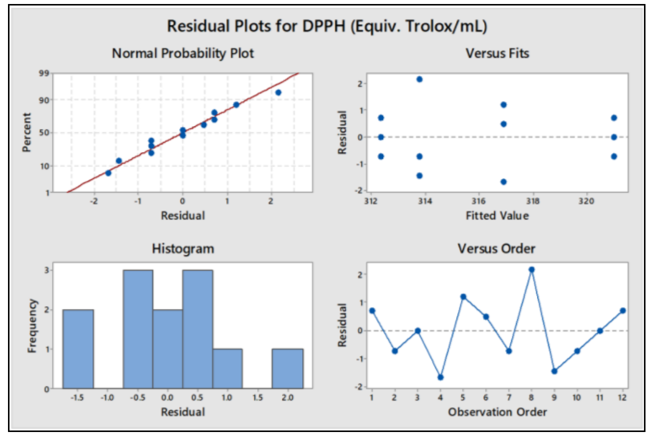

Fig. 7. Residual plots for antioxidant capacity (DPPH, expressed as Trolox Equiv./mL), including a normal probability plot, histogram, residuals versus fitted values, and residuals versus observation order.

The viscosity of formulations Ch1, Ch3, and Ch5 was higher than that of the control chocolate ChC, with the viscosity of chocolate (Ch3) at 0.078% being the highest. The opposite is true for texture, which was greater for the control chocolate. Therefore, ginger oleoresin produces more viscous and smoother chocolates. This texture increases with the amount of oleoresin used. These findings demonstrate that ginger oleoresin affects the physical properties of viscosity and texture.

Thermograms showing the melting points of formulations Ch1, Ch3, Ch5 and ChC are shown in Fig. 8. Compared with the prototypes, the control chocolate has a lower melting point. Of these, chocolate (Ch3) with 0.078% ginger oleoresin has the highest melting point, indicating that it is more heat-resistant. The opposite is true for the melting enthalpy, as the control chocolate has a higher enthalpy than the prototypes do, meaning that it absorbs more heat in less time, which is why it melts at a lower temperature.

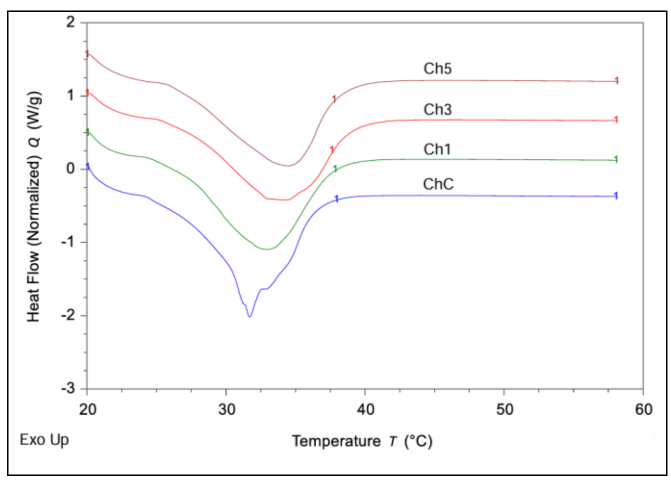

Fig. 8. Thermograms showing the melting point of 70% cocoa dark chocolates fortified with the addition of ginger oleoresin.

Table 2a. Physicochemical properties of 70% cocoa dark chocolates fortified with ginger oleoresin

Formula	Viscosity Pa.s	Texture N	Melting Temperature °C	Melting Enthalpy J/g	Crystallization Temperature °C	Crystallization Enthalpy J/g
Ch1	1.48 ± 0.04[ab]	20.71 ± 0.17[d]	32.56 ± 0.35[ab]	55.18 ± 0.74[ab]	10.45 ± 0.19[a]	32.05 ± 0.01[a]
Ch3	1.70 ± 0.06[a]	22.95 ± 0.55[c]	33.98 ± 0.91[a]	52.23 ± 0.53[b]	10.27 ± 0.74[a]	31.57 ± 0.43[a]
Ch5	1.43 ± 0.15[bc]	26.99 ± 1.45[b]	33.58 ± 0.76[a]	51.40 ± 1.38[b]	10.40 ± 0.62[a]	31.72 ± 0.63[a]
ChC	1.22 ± 0.04[c]	29.31 ± 0.36[a]	31.64 ± 0.06[b]	60.60 ± 4.69[a]	11.10 ± 0.00[a]	30.33 ± 0.00[b]

Note: Results from 3 replicates. The values are expressed as the mean ± SD. Different letters for each analysis indicate significant differences ($p < 0.05$).

Table 2b. Physicochemical properties of 70% cocoa dark chocolates fortified with ginger oleoresin

Formula	Color			Whiteness index	OSI (h)
	a	**c**	**L**		
Ch1	12.15 ± 0.39[a]	0.35 ± 0.33[a]	29.63 ± 0.07[b]	28.59 ± 0.11[b]	0.11 ± 0.01[a]
Ch3	12.13 ± 0.323[a]	0.69 ± 0.33[a]	29.68 ± 0.22[b]	28.63 ± 0.25[b]	0.07 ± 0.01[b]
Ch5	12.04 ± 0.261[a]	0.79 ± 0.30[a]	30.64 ± 0.31[ab]	29.59 ± 0.27[ab]	0.07 ± 0.01[b]
ChC	11.89 ± 0.14[a]	0.23 ± 0.17[a]	30.94 ± 0.86[a]	29.92 ± 0.87[a]	0.08 ± 0.02[ab]

Note: Results from 3 replicates. The values are expressed as the mean ± SD. Different letters for each analysis indicate significant differences ($p < 0.05$).

Table 3. Bioactive properties of 70% cocoa dark chocolates fortified with ginger oleoresin

Formula	TPC mg GAE/mg sample	DPPH Trolox Equiv./mL	ABTS Trolox Equiv./mL
Ch1	20.69 ± 0.27[b]	316.91 ± 1.50[b]	373.72 ± 2.60[b]
Ch3	20.28 ± 0.09[bc]	313.79 ± 1.91[bc]	377.81 ± 7.23[ab]
Ch5	19.58 ± 0.71[c]	321.01 ± 0.72[a]	390.57 ± 5.42[a]
ChC	21.98 ± 0.26[a]	312.34 ± 0.72[c]	365.54 ± 3.26[b]

Note: Results from 3 replicates. The values are expressed as the mean ± SD. Different letters for each analysis indicate significant differences ($p < 0.05$).

Table 4. Sensory properties of 70% cocoa dark chocolates fortified with ginger oleoresin

Formula	Color	Taste	Odor	Brigthness	Overall Acceptability
Ch1	6.44 ± 1.37[a]	5.46 ± 1.88[a]	6.04 ± 1.39[ab]	6.32 ± 1.47[a]	5.80 ± 1.80[ab]
Ch3	6.76 ± 1.38[a]	5.00 ± 1.97[a]	5.64 ± 1.60[b]	6.34 ± 1.80[a]	5.08 ± 1.77[b]
Ch5	6.78 ± 1.20[a]	4.82 ± 2.00[a]	5.74 ± 1.76[ab]	6.34 ± 1.85[a]	4.94 ± 1.81[b]
ChC	6.54 ± 1.34[a]	5.84 ± 2.07[a]	6.50 ± 1.19[a]	6.24 ± 1.77[a]	6.02 ± 1.63[a]

Note: Results from 50 replicates. The values are expressed as the mean ± SD. Different letters for each analysis indicate significant differences ($p < 0.05$).

The crystallization temperatures of the control chocolate and the prototypes Ch1, Ch3, and Ch5 were the same, indicating that they crystallized at the same temperature. Conversely, these prototypes have different enthalpies of crystallization, with the control sample having the highest value, meaning that it releases more heat than the others do during crystallization. This translates to a shorter tempering time and therefore lower energy consumption at this stage.

Both the control chocolate and Ch1 have the same oxidation times, and these oxidation times are even longer than those of the Ch3 and Ch5 chocolates, which indicates that the latter have greater oxidation stability than the others do. Therefore, ginger oleoresin does not improve the oxidative stability of chocolates.

With respect to the sensory analysis, there were no significant differences between the colors of the prototype and control chocolates, indicating that ginger oleoresin did not modify the color of any of the chocolates. The color attribute is related to brightness, which is why oleoresin also did not modify this parameter. Similarly, the flavor was not altered by ginger oleoresin. The opposite occurred with the aroma, as the control chocolate (ChC) and the chocolate with the lowest percentage of oleoresin (Ch1) shared a similar aroma. A graphical summary of the results is presented in Fig. 9.

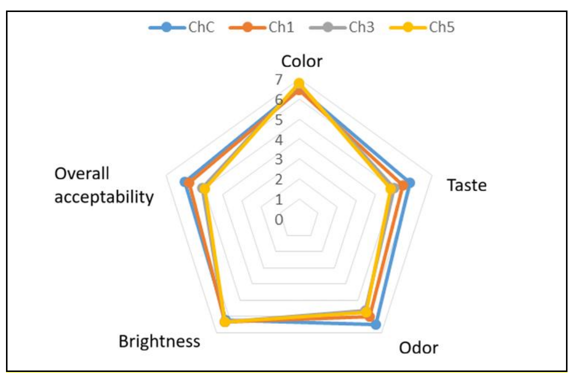

Fig. 9. Sensory quality parameters of 70% cocoa dark chocolates with the incorporation of ginger oleoresin.

Overall acceptability is an attribute that reflects the overall perception of the product, which is more highly valued by consumers who are not chocolate experts, but who do or are willing to consume chocolate. Therefore, individuals with this profile were selected. The control chocolate had better overall acceptability, suggesting that ginger oleoresin imparts an astringent flavor to chocolates. However, it also increases their melting point, making them more resistant to melting. Therefore, its use in smaller proportions would be advisable. Compared with the control chocolate, all the prototypes had a lower overall acceptability score because of the addition of ginger oleoresin. The overall acceptability of chocolate with the lowest concentration of ginger oleoresin (Ch1, 0.026%) did not differ significantly from that of the control; however, its antioxidant capacity was better. Therefore, this chocolate would be suitable as a reference for potential scaling up. Moreover, with a small amount of ginger oleoresin it is possible to obtain a favorable effect on the antioxidant capacity of chocolate.

Among the chocolates. the chocolate with 0.129% ginger oleoresin (Ch5) had the highest antioxidant capacity because of the higher concentration of ginger oleoresin added. There is an inverse relationship between the antioxidant capacity of the chocolates and their overall acceptability, since a higher concentration of ginger oleoresin increases the antioxidant capacity but reduces its acceptability. Compared with the control, chocolate with 0.026% oleoresin resulted in a suitable balance between functional improvement and sensory acceptance, with no significant differences. This should be considered when formulating chocolates for commercialization.

The observed increase in antioxidant capacity with increasing oleoresin concentration is consistent with the findings of previous studies on functional chocolates enriched with natural bioactive compounds [12,13]. The presence of gingerols and shogaols in ginger oleoresin contributes to the underlying antioxidant mechanism. However, as reported in similar systems, high concentrations can negatively impact sensory properties, making it important to determine the appropriate dosage [14]. These findings demonstrate that low concentrations of bioactive additives can improve functional properties while maintaining the quality of the final product.

IV. CONCLUSION

In this study, 70% cocoa dark chocolate prototypes made with Peruvian Criollo cocoa were enriched with ginger oleoresin obtained through supercritical carbon dioxide extraction. The addition of 0.026% oleoresin increased the antioxidant capacity (DPPH) from 312.34 ± 0.72 to 316.91 ± 1.50 compared with that of the control, while the sensory acceptability scores were maintained between 6.02 ± 1.63 and 5.80 ± 1.80. A higher concentration (0.129%) improved the antioxidant capacity (DPPH) but negatively affected the sensory properties. Additionally, an increase in the melting point from 31.64 ± 0.06 to 32.56 ± 0.35 suggests modifications in the chocolate matrix structure. This demonstrated the technological feasibility of using bioactive extracts as functional ingredients in chocolate lipid matrices. Compared with the control chocolate, the formulation with 0.026% oleoresin stood out as the most suitable in terms of overall acceptability also offered improvements in antioxidant capacity.

From a process engineering perspective, the color stability, low whiteness index, and adequate crystallization observed in all formulations indicate that the addition of oleoresin does not negatively interfere with the formation of the crystalline phases of cocoa butter, which is a key aspect for ensuring the physical and visual stability of the chocolate throughout its shelf life. The rheological and thermal results show that ginger oleoresin acts as a lipid matrix modifier, increasing the viscosity and melting point of fortified chocolates. This can improve product performance during transport, storage, and marketing in warm climates, reducing losses due to deformation or melting.

The results demonstrate that ginger oleoresin obtained using supercritical fluid technology is a potential ingredient for formulating value-added functional chocolates. The chocolate market can be enhanced by this type of development focused on fortified chocolates.

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