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Examining the Quality Enhancement and Antioxidant Properties of White Compound Chocolate Formulated with Pineapple Powder (*Ananas comosus* L.)

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Abstract

Efforts to enhance the antioxidant activity of white compound chocolate, a type of chocolate with the lowest phenolic content, can be achieved by incorporating additional fruit-based active ingredients. Pineapple powder is a promising ingredient for chocolate formulation due to its distinctive flavor characteristics and potential bioactive compounds. This study aimed to investigate the effects of varying pineapple powder concentrations and grinding durations on the physical and chemical properties of white compound chocolate formulated with pineapple powder. The study utilized a factorial completely randomized design (CRD) with 2 factors: pineapple powder concentration (15%, 20%, and 25%) and the grinding duration (2.5, 5, and 7.5 hours). The experiment was conducted in triplicate. The results indicated that L* was significantly reduced with the addition of 20% and 25% pineapple powder. At the same time, chocolate hardness was significantly higher than the control, especially with the 25% pineapple powder addition and grinding times of 5 and 7.5 hours. The incorporation of pineapple powder increased the moisture content of the chocolate from 1.3 to 2%. Volatile compounds, such as δ-Caprolactone, benzyl valerate, benzaldehyde, 2,4-dihydroxy-, γ-Decalactone, 5-hydroxymethylfurfural, were detected in the formulated chocolate. Incorporating 25% pineapple powder increased phenolic and flavonoid content by almost fourfold. Thus, it significantly improved the antioxidant activity of the chocolate in terms of DPPH and ferric reducing antioxidant power (FRAP). Variations in grinding duration did not significantly affect the physical and chemical properties. The results of this study create new opportunities for the food industry to develop innovative chocolate variants.

Keywords: flavor profile; melanger; phenolic improvement; quality attributes

INTRODUCTION

Among different types of chocolate, white chocolate has the lowest antioxidant and phenolic contents because it contains no cocoa solids, but only cocoa butter or cocoa butter substitutes, milk powder, sugar, lecithin, and vanilla, without cocoa mass (Muhammad et al., 2022). To enhance the antioxidant activity of white compound chocolate, additional ingredients can be used. The addition of cinnamon to white chocolate significantly increases antioxidant activity, but it may also have a drawback in aroma properties (Muhammad et al., 2020; 2021).

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It has been reported that total phenolic compounds and antioxidant activity of white compound chocolate substantially increase with the addition of fruits and vegetables, including cornelian cherries, spinach, pollen powder, matcha green tea powder, *Moringa oleifera* leaf powder, and blueberries (Jovanović et al., 2022; Poliński et al., 2022). As found in a previous experiment using cinnamon (Praseptiangga et al., 2019), fortification of chocolate using these vegetal substances changes its characteristics and, thus, may reduce consumer acceptance. Chocolate can be enriched using a popular plant-based extract to overcome this issue.

Pineapple (*Ananas comosus* L.), a tropical fruit widely cultivated in many countries, is used as a flavoring agent in various foods. Hence, pineapple can potentially enrich not only bioactive compounds but also the flavor of the chocolate. However, to date, no literature has discussed the use of pineapple in chocolate formulation. Pineapple has been reported to contain bioactive compounds, including vitamin C, phenolic compounds, and β-carotene, which act as natural antioxidants (Gardner et al., 2000; Du et al., 2016). The phenolic and flavonoid contents of pineapple were reported at 33.5 mg GAE 100 g^{-1} and 6.15 mg QE 100 g^{-1}, respectively (Nordin et al., 2023). Furthermore, pineapple has been reported to have DPPH radical-scavenging activity with an IC50 value of 24.13 μg ml^{-1} (Abbas et al., 2021). Various pineapple varieties have been reported to contain vitamin C (35.88 to 62.11 mg 100 g^{-1}) and phenolic compounds (71.07 to 126.95 mg gallic acid 100 g^{-1}), and thus exhibit antioxidant activity (Ferreira et al., 2016).

In addition, pineapple contains secondary metabolites, including aromatic volatile compounds. More than 290 volatiles, including methyl and ethyl esters of saturated and unsaturated fatty acids, acetates, terpenes, alcohols, aldehydes, 2-ketones, free fatty acids, and miscellaneous γ- and δ-lactones, have been reported by Steingass et al. (2015). The presence of aromatic compounds potentially improves the chocolate's consumer acceptance and sensory properties. Thus, there are 2 main reasons for using pineapple powder in this study: potential bioactivity improvement and potential flavor enhancement.

Furthermore, in a bean-to-bar technique of making chocolate, grinding using a melanger, so-called *melanging*, is important to achieve the desired texture and avoid a gritty mouthfeel. Chocolate grinding is crucial to the mouthfeel characteristic of the chocolate, as grittiness can be perceived when particles are larger than 30 μm (Hinneh et al., 2019a). Extended grinding time results in smaller particle sizes. It was reported that the particle size was > 50, 20-25, 15-20, and 10-15 μm after 0, 8, 16, and 26 hours of grinding, respectively (Clark et al., 2020).

Grinding duration using a melanger has been reported to affect the quality attributes of chocolate, including rheology, moisture content, and flavor profile (Hinneh et al., 2019a; 2019b). Thus, this study aimed to examine the effect of varying concentrations of pineapple powder and grinding durations on physical (color, hardness, and rheology) and chemical properties (moisture content, flavonoid content, total phenols, antioxidant activity, and aromatic volatile compounds). It was anticipated that adding pineapple powder could enhance the antioxidant activity of white compound chocolate with desired aromatic properties and acceptable textural and mouthfeel characteristics. The information obtained from this study may be useful for the chocolate industry to formulate new chocolate variants.

MATERIALS AND METHOD

Preparation of chocolate samples

White compound chocolate (Tulip, White Compound, PT Freyabadi Indotama, Indonesia) was melted in a choco-melter (Mol d'Art Chocolate Melter, Belgium) at a temperature of 45 °C. After obtaining molten white compound chocolate, pineapple powder (CV Seduh Tisane Nusantara, Indonesia) was added at varying concentrations (15%, 20%, and 25% w/w). The next process was the grinding of white compound chocolate with pineapple powder samples using a melanger (CocoaTown, ECGC-12SLTA, USA). Grinding was carried out for 3 different time variations; namely, 2.5, 5, and 7.5 hours. Afterward, the chocolate samples were molded and stored properly prior to analysis.

Determination of physical characteristics

Color

Color measurement of white compound chocolate samples was conducted using a portable colorimeter (WR-18 model, Guangdong Threenh

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Technology, Guangzhou, China). The values displayed on the colorimeter screen are L*, a*, and b*. These values were then used to determine the chroma (C*), whiteness index (WI), and color difference (ΔE) using Equations 1, 2, and 3, respectively.

$$C^{*}=\sqrt{a^{*2}+b^{*2}} \quad (1)$$

$$WI=100-\sqrt{\left(100-L^{*}\right)^{2}+a^{*2}+b^{*2}} \quad (2)$$

$$\Delta E=\sqrt{\left(L_1^{*}-L_2^{*}\right)^{2}+\left(a_1^{*}-a_2^{*}\right)^{2}+\left(b_1^{*}-b_2^{*}\right)^{2}} \quad (3)$$

Hardness

The hardness measurement was performed using a Universal Testing Machine Zwick/Roell BDO-FB0.5TS (Zwick GmbH & Co, Ulm, Germany) coupled with a probe (ϕ = 1 mm) following the standard penetration test.

Rheology

The rheological characteristics of spiced white compound chocolate were measured using a modular compact rheometer (MCR) (Physica MCR 302, Anton Paar, Graz, Austria). The sample was melted by incubating at a temperature of 40 ℃ before the measurement cycle began. Shear stress was then measured at a temperature of 40 ℃ as a function of shear rate over a range of 1 to 50 second^{-1} to examine the type of flow behavior. Subsequently, the apparent viscosity was reported at 50 second^{-1}, as this point represents the shear rate in the mouth (Muhammad et al., 2018).

Determination of antioxidant properties

Extraction of antioxidant compounds from white chocolate

Defatting was first conducted prior to the extraction process. Briefly, 20 g of the chocolate sample was weighed and transferred to a 250 ml beaker. Then, 100 ml of n-hexane was added. A magnetic stirrer was placed in the beaker, and the mixture was stirred for 10 minutes at a speed of 400 rpm. After stirring, the mixture was filtered using filter paper to separate the residue from the n-hexane containing the fat. This filtration process was repeated twice. The defatted sample was then weighed and stored in a dark room at room temperature for 24 hours.

For the extraction process, 8 g of the defatted sample was weighed and transferred into a 50 ml beaker. To this, 20 ml of a solvent mixture containing 70% acetone, 29.8% distilled water, and 0.2% acetic acid was added. A magnetic stirrer was inserted into the beaker, and the beaker was covered with aluminum foil. The sample was homogenized using the magnetic stirrer for 10 minutes. The mixture was then filtered using filter paper to separate the residue from the filtrate, which contained the antioxidants. This extraction procedure was repeated twice to ensure complete extraction. The volumes of the collected filtrates (filtrate 1 and filtrate 2) were recorded. Finally, the filtrates were stored in dark bottles and kept in cold storage (Muhammad et al., 2017).

Total phenolic content

The procedure for determining total phenolic content was as follows: 200 µl of Folin-Ciocalteu reagent and 1 ml of distilled water were added to each test tube. Subsequently, 200 µl of gallic acid solution from each dilution was added. The mixture was homogenized using a vortex for 20 seconds and then incubated for 6 minutes at room temperature in the dark. Following incubation, 2.5 ml of 7% Na_2CO_3 solution and 2.1 ml of distilled water were added to each test tube. The mixture was homogenized again for 20 seconds and incubated for an additional 90 minutes at room temperature in the dark. The absorbance was measured using a spectrophotometer (UVmini-1240, Shimadzu Corporation, Japan) at a wavelength of 760 nm (Muhammad et al., 2017).

Flavonoid content

The procedure for determining flavonoid content was as follows: 200 µl of sample solution was added to each test tube. Then, 5 ml of 0.1 M $AlCl_3$ solution was added. The mixture was homogenized using a vortex for 30 seconds and incubated for 40 minutes at room temperature in the dark. Absorbance was subsequently measured at a wavelength of 415 nm using a spectrophotometer (UVmini-1240, Shimadzu Corporation, Japan) (Muhammad et al., 2017).

DPPH-radical scavenging activity

The procedure for determining antioxidant activity using the 2,2-diphenylpicrylhydrazyl (DPPH) method was as follows: 0.1 ml of the sample was added to a test tube. Then, 4 ml of DPPH solution was added to each sample.

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The mixture was homogenized using a vortex for 30 seconds and incubated for 30 minutes at room temperature in the dark. Finally, absorbance was measured with a spectrophotometer (UVmini-1240, Shimadzu Corporation, Japan) at a wavelength of 517 nm (Muhammad et al., 2017).

Ferric reducing antioxidant power (FRAP)

The procedure for determining antioxidant activity using the FRAP method was carried out as follows: 2.5 ml of 0.2 M phosphate buffer (pH 7), 1 ml of the sample, and 2.5 ml of 1% potassium ferricyanide were mixed in a test tube. The mixture was homogenized using a vortex for 20 seconds and incubated at 50 °C for 30 minutes. Subsequently, 2.5 ml of 10% trichloroacetic acid (TCA) solution was added, and the mixture was homogenized again using a vortex for 20 seconds. From this solution, 2 ml was transferred into a new test tube, mixed with 2 ml of distilled water and 0.4 ml of 0.1% $FeCl_3$ solution. The mixture was then homogenized using a vortex, and the absorbance was measured using a spectrophotometer (UVmini-1240, Shimadzu Corporation, Japan) at a wavelength of 700 nm (Muhammad et al., 2017).

Determination of volatile compounds

The volatile aromatic compounds in white compound chocolate were analyzed using Gas Chromatography (GC) Agilent 7890A and Mass Spectrometry (MS) Agilent 5975C XL EI/CI (Santa Clara, California, USA) under specific conditions for extraction, injection, and temperature control. The sample (4 g) was first extracted in a 22 ml SPME vial. 2,4,6-Trimethylpyridine at 0.0001% (0.2 µl) was used as an internal standard. Extraction was conducted at 60 °C for 45 minutes using a SPME fiber DVB/CAR/PDMS (2 cm).

GC Agilent 7890A equipped with MS Agilent 5975C XL EI/CI and DB-Wax column (30 m × 250 µm × 0.25 µm) was used in this study. Tuning and calibration of the mass spectrometer were done prior to analysis. The extract was injected in splitless mode at 250 °C. Helium was used as the carrier gas at a flow rate of 0.8 ml $minute^{-1}$. The column temperature was held at 35 °C for 5 minutes, increased to 182 °C at a rate of 3 °C $minute^{-1}$, and continued to 240 °C at a rate of 6 °C $minute^{-1}$, and held for 7 minutes. The interface was set at 250 °C. The MS source and MS quadrupole were set at 230 °C and 150 °C, respectively. Scan mass was done at a range of 29-550 amu. A standard alkane mixture (C8-C40) was injected prior to analysis for peak identification. Library NIST14 was used to interpret the spectra.

Research design and statistical analysis

This study used a completely randomized design (CRD) consisting of 2 factors: pineapple powder concentration (15%, 20%, and 25%) and grinding duration (2.5, 5, and 7.5 hours). Data were obtained from 3 replications of independent samples. To compare the means, the research data were statistically analyzed using One-Way ANOVA at a significance level of $\alpha = 5\%$, particularly for the parameters of color, hardness, moisture content, antioxidant activity, total phenols, and total flavonoids. If significant differences were found, the analysis was followed by Duncan's multiple range test (DMRT).

RESULTS AND DISCUSSION

Antioxidant properties of pineapple powder

Table 1 shows that the pineapple powder had a total phenolic content of 0.567±0.012 mg GAE g^{-1} and a flavonoid content of 0.054±0.002 mg QE g^{-1}. This result is in accordance with previous studies reporting total phenolic content in pineapple powder ranging from 0.23 to 0.58 mg GAE g^{-1}, while fresh pineapple ranges from 0.51 to 0.71 mg GAE g^{-1} (Hashib et al., 2019). The phenolic content in pineapple varies depending on the part of the plant, with the peel and stem containing higher levels compared to the fruit flesh. Additionally, processing pineapple into powder reduces the phenol content compared to fresh fruit (Rasheed et al., 2012; Hashib et al., 2019).

Meanwhile, Vidinamo et al. (2022) found that flavonoids significantly decrease during pineapple powder production, particularly during the drying process. The antioxidant activity of pineapple powder, measured using the DPPH

Table 1. Characteristics of pineapple powder

Parameter	Value
Total phenol (mg GAE g^{-1})	0.567±0.012
Flavonoid content (mg QE g^{-1})	0.054±0.002
Antioxidant activity-DPPH (% inhibition)	29.92±2.100
Antioxidant activity-FRAP (mg AAE g^{-1})	0.068±0.002

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and FRAP methods, was about 29.92% inhibition and 0.068 mg AAE g^{-1} sample, respectively. Much literature shows that antioxidant activity is positively correlated with the phenolic content and FRAP value of the sample. In fresh pineapple, antioxidant activity ranges from 48 to 65% inhibition, while in pineapple powder, it ranges from 16 to 53% inhibition (Hashib et al., 2019; Poliński et al., 2022).

Physical characteristics

Color

The color analysis of white compound chocolate with varying concentrations of pineapple powder and grinding durations revealed several key findings. The L* value reflects color brightness, ranging from 0 (dark) to 100 (light) (Pack et al., 2015). The control sample (white compound chocolate) had an L* value of 100 (the highest brightness level). Adding 15% pineapple powder did not significantly change brightness, but additions of 20% and 25% significantly reduced it (Table 2). The decrease was attributed to non-enzymatic browning during spray drying of the pineapple powder, which affected the final color of the chocolate (Mala et al., 2024).

The a* value indicates the degree of redness (positive) to greenness (negative) (Pack et al., 2015). All samples exhibited negative a* values, indicating a greenish hue. Most samples differed significantly from the control, except those with 15% pineapple powder and 5-hour grinding. Similar greenish values have been reported in white chocolate (Muhammad et al., 2018) and pineapple juice products (Charoenphun, 2019; Quoc, 2020). The b* value indicates the degree of yellowness (positive) to blueness (negative) (Pack et al., 2015). All samples exhibited positive b* values, with pineapple powder increasing yellowness compared to the control. Significant increases were observed across different grinding times, depending on carotenoid content (Ferreira et al., 2016).

The C* measures color saturation or clarity (Genc Polat et al., 2020). Pineapple powder increased C* values, indicating more intense color saturation (Goktas et al., 2023). Grinding affected particle size, with smaller particles increasing C* due to larger surface area (Feichtinger et al., 2020). The WI reflects the degree of whiteness of a sample (Indiarto et al., 2024). The control had the highest WI, with significant decreases observed following the pineapple powder additions, especially at 2.5 and 7.5 hours of grinding. The reduction was attributed to the presence of yellow pigments in pineapple powder (Charoenphun, 2019). However, grinding duration did not significantly affect L*, a*, b*, C*, and WI. ΔE represents the color difference between a sample and the control. Higher ΔE values indicate perceptible differences. In this study, ΔE values increased with higher pineapple powder concentrations but were not significantly affected by grinding duration. ΔE values ranged from 2.18 to 4.41, indicating noticeable color differences from the control.

Hardness

As shown in Figure 1, the chocolates formulated with pineapple powder had a higher hardness level than the control chocolate. Also, there was a clear trend that the addition of pineapple powder increased the hardness of the chocolate, particularly noticeable at grinding times of 5 and 7.5 hours. As such, the hardness of the control chocolate was 85.57 N while that of

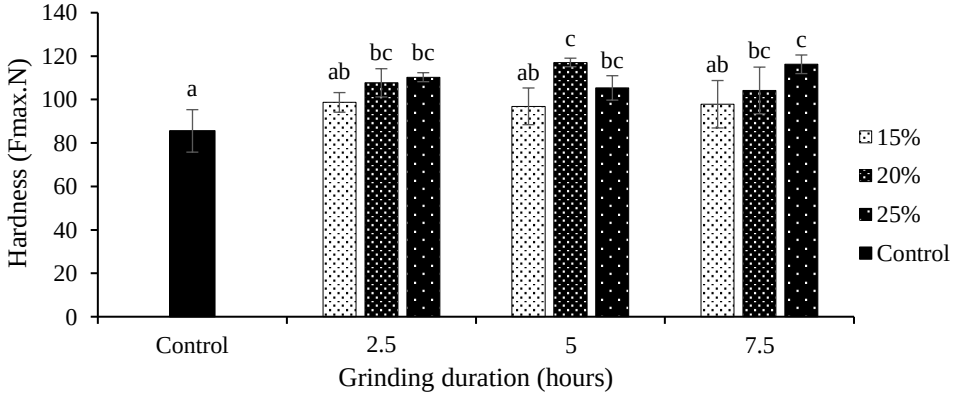

Figure 1. Hardness of chocolate formulated with pineapple powder
Note: Different superscripts indicate a significant difference ($p < 0.05$) among samples

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Table 2. Color analysis of white compound chocolate

Treatment		L*	a*	b*	C*	WI	ΔE
Control		100.00±0.00^{d}	-2.11±0.06^{a}	9.35±0.51^{a}	9.59±0.49^{a}	90.41±0.49^{d}	-
2.5 hours	15%	99.52±0.43^{cd}	-1.68±0.23^{bc}	11.41±0.30^{bc}	11.53±0.28^{bc}	88.45±0.30^{bc}	2.18±0.40^{ab}
	20%	98.67±0.47^{bc}	-1.46±0.14^{cd}	12.37±0.33^{d}	12.46±0.31^{d}	87.46±0.30^{ab}	3.39±0.33^{c}
	25%	97.03±0.73^{a}	-1.25±0.12^{de}	12.13±0.35^{cd}	12.20±0.34^{cd}	87.43±0.48^{a}	4.17±0.71^{d}
5 hours	15%	99.06±0.70^{bcd}	-1.92±0.05^{ab}	11.93±0.13^{cd}	12.08±0.13^{cd}	87.87±0.09^{ab}	2.81±0.18^{bc}
	20%	98.53±0.38^{b}	-1.70±0.07^{bc}	12.00±0.29^{cd}	12.12±0.29^{cd}	87.79±0.26^{ab}	3.08±0.19^{c}
	25%	96.96±0.43^{a}	-1.28±0.52^{de}	12.02±0.84^{cd}	12.09±0.88^{cd}	87.52±0.76^{a}	4.22±0.18^{d}
7.5 hours	15%	99.50±0.18^{cd}	-1.57±0.02^{bcd}	11.16±0.33^{b}	11.27±0.33^{b}	88.72±0.32^{c}	1.96±0.27^{a}
	20%	98.29±0.84^{b}	-1.35±0.18^{cde}	11.39±0.36^{bc}	11.47±0.34^{bc}	88.39±0.47^{bc}	2.79±0.83^{bc}
	25%	97.02±0.48^{a}	-1.02±0.21^{e}	12.40±0.30^{d}	12.45±0.30^{d}	87.20±0.33^{a}	4.41±0.44^{d}

Note: The results represented the means of 3 replicates of independent samples. Different superscripts in the same column indicate a significant difference ($p < 0.05$) among samples

chocolate formulated with 25% pineapple powder was in the range of 97.85 and 116.24 N. Chocolate hardness is influenced by particle interactions, ingredient composition, moisture content, and grinding duration (Muhammad et al., 2018; Feichtinger et al., 2020).

Figure 1 also shows that chocolate containing 20% pineapple powder and melanged about 5 hours exhibited the highest hardness value. However, the hardness was still not statistically different from that of the chocolate added with 25% pineapple powder ground for a similar duration. Adding other ingredients to white compound chocolate enhances the solid volume fraction, leading to greater particle interactions and a firmer texture. In this study, the increase in Fmax values in the textural property tests was later confirmed to be associated with increased moisture content. However, variations in grinding duration did not significantly affect the hardness of the chocolates. As grinding duration may affect chocolate particle size, this study hypothesizes that moisture content has a more pronounced effect on the chocolate hardness than particle size. Nevertheless, to prove this hypothesis, further research is required.

Rheology

Information regarding the rheological behavior of chocolate is important, particularly in terms of sensory characteristics (Vásquez et al., 2019) as well as material transfer and pumping during processing (Toker et al., 2023). Viscosity of both the control and pineapple powder-added white compound chocolate samples decreased with increasing shear rate, consistent with the behavior of chocolate as a shear-thinning (pseudoplastic) non-Newtonian fluid (Kumbár et al., 2021). The addition of pineapple powder and grinding duration were linearly related to the sample viscosity; as more powder was added and grinding time increased, the viscosity rose (Figure 2). The addition of solids causes particle interactions, leading to a thicker or more cohesive chocolate mixture. Also, longer grinding durations reduce particle size, increasing surface area and consequently enhancing chocolate viscosity (Feichtinger et al., 2020).

Chemical characteristics

Moisture content

Figure 3 illustrates that white compound chocolate samples with varying pineapple powder concentrations and grinding durations had higher

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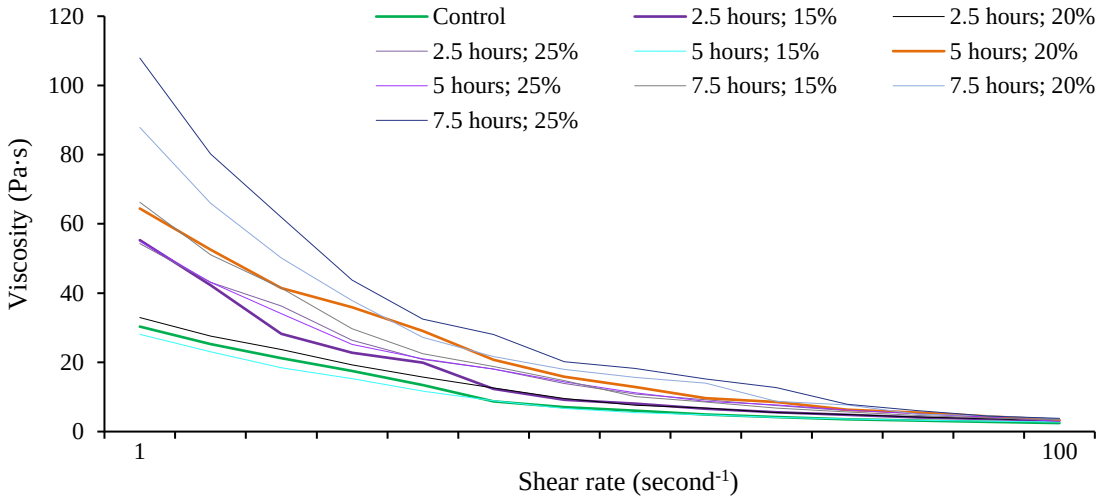

Figure 2. Rheological behavior of white compound chocolate with variations in pineapple powder concentration and grinding duration

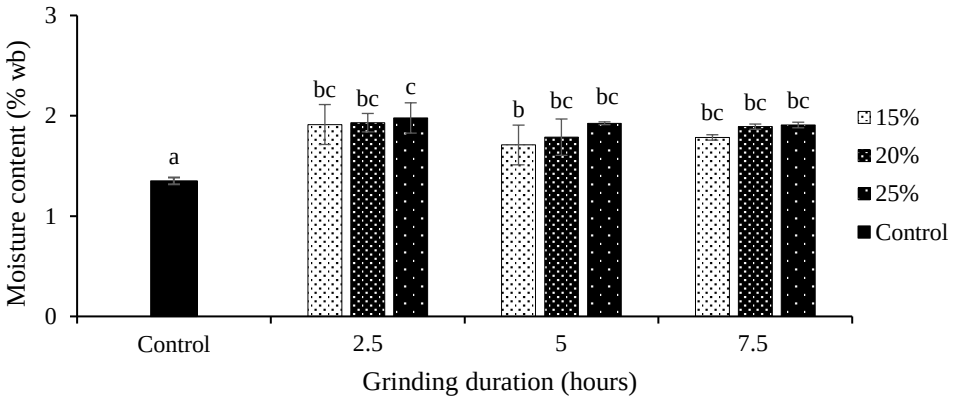

Figure 3. Moisture content of chocolate formulated with pineapple powder
Note: Different superscripts indicate a significant difference ($p < 0.05$) among samples

moisture content (1.71 to 1.98%) compared to the control sample (1.35%). Pineapple powder has a higher moisture content than chocolate; therefore, a higher proportion of pineapple powder formulated in the chocolate resulted in the higher moisture content of the chocolate. Also, an increase in the moisture content is attributed to the hygroscopic nature of pineapple powder, which absorbs moisture from the environment (Juarez-Enriquez et al., 2017; Malini et al., 2024).

Despite the increased concentration of pineapple powder and variations in grinding duration (2.5 to 7.5 hours), there were no significant differences in moisture content. The grinding durations with the melanger had no effect in reducing moisture content (Hinneh et al., 2019a), as its main purpose is to reduce the particle size of the chocolate. Grinding for up to 7.5 hours did not significantly reduce moisture in the formulated chocolate. Increased moisture content due to the higher proportion of pineapple powder was also correlated with the hardness of the material, as moisture content relates to sugar network formation, strengthens particle network systems, and thus increases the hardness of the chocolates (Muhammad et al., 2018).

Total phenolic and flavonoid content

Figure 4 shows that white compound chocolate samples with varying pineapple powder concentrations and grinding durations had higher total phenolic content compared to the control sample (0.15±0.01 mg GAE g^{-1}). Statistical analysis confirmed that the phenolic contents of chocolates with pineapple powder were significantly different. Total phenolic content increased with higher pineapple powder concentrations. Phenolic compounds in pineapple, such as myricetin and tannic acid,

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exhibit antioxidant, antimicrobial, and anti-inflammatory properties (Rasheed et al., 2012). However, prolonged grinding tended to reduce phenolic content. The reduction may be attributed to the oxidation and degradation of phenols caused by exposure to oxygen and light during extended grinding (Medina-Mendoza et al., 2023).

A similar trend was shown in the parameter of the total flavonoid content. It was shown that white compound chocolate samples with varying concentrations and grinding durations had higher flavonoid content compared to the control sample (0.019±0.003 mg QE g^{-1}), with statistically significant differences. Flavonoid content increased with the addition of pineapple powder, especially at a concentration of 25%, across all grinding durations of 2.5, 5, and 7.5 hours. Flavonoids are antioxidant compounds belonging to the phenolic group. Extended grinding times (from 5 to 7.5 hours) resulted in a significant reduction in flavonoid content (with 15% and 20% pineapple powder additions). This reduction may also be attributed to prolonged exposure to oxygen, which can lead to flavonoid degradation (Medina-Mendoza et al., 2023).

Antioxidant activity

Antioxidant activity was assessed using the DPPH and FRAP methods (Figure 5). White compound chocolate samples with varying concentrations and grinding durations exhibited higher antioxidant activity than the control. Antioxidant activity, measured by the DPPH-radical scavenging activity method, increased with the addition of pineapple powder, with significant increases observed at 15% and 25% powder additions. A similar phenomenon was shown using the FRAP test, which also demonstrated increased antioxidant activity with the addition of pineapple powder. This finding supports previous research that adding phenolic-rich material, such as cinnamon, can enhance phenolic content and antioxidant activity in white chocolate (Muhammad et al., 2018).

Another research demonstrated that the addition of pineapple peel powder to crackers resulted in higher and significantly different FRAP values compared with the control crackers (Mala et al., 2024). Regarding variations in grinding duration, a decrease in DPPH-radical scavenging activity was observed from 5 to 7.5 hours for the 15% addition of pineapple powder. In contrast, the FRAP values did not show significant reductions. The rise of antioxidant activity was attributed to pineapple powder. Pineapple powder has been proven to contain vitamin C, phenolic compounds, and β-carotene, which are natural antioxidants (Gardner et al., 2000). In this study, it was clearly shown that pineapple powder had a total phenol content of 0.567±0.012 mg GAE g^{-1} and a flavonoid content of 0.054±0.002 mg QE g^{-1}. It also exhibited DPPH-radical scavenging and FRAP activities.

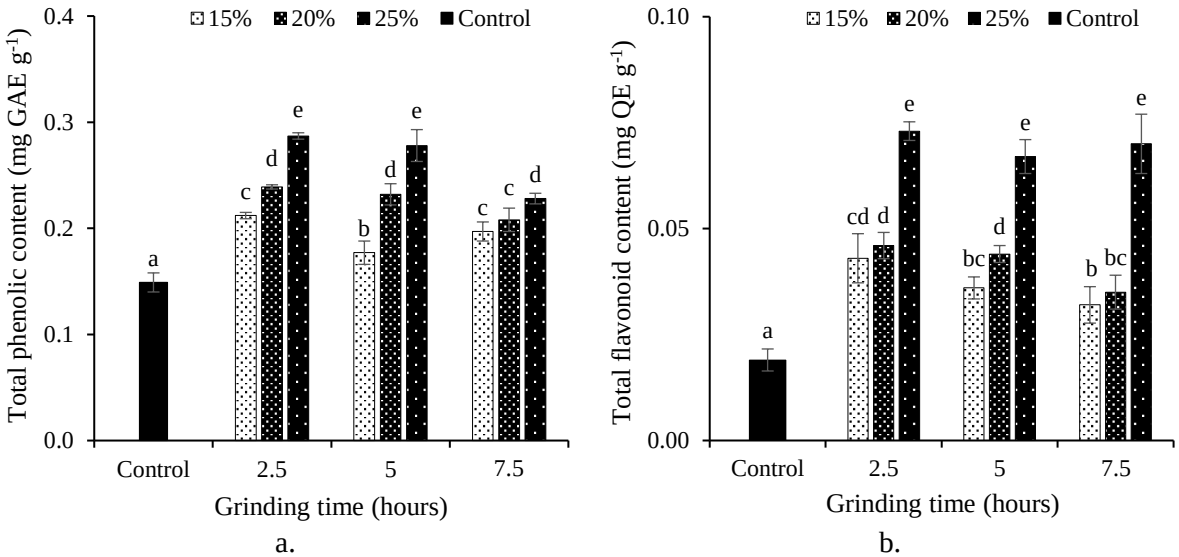

Figure 4. Total phenolic content (a) and total flavonoid content (b) of chocolate formulated with pineapple powder

Note: Different superscripts indicate a significant difference ($p < 0.05$) among samples

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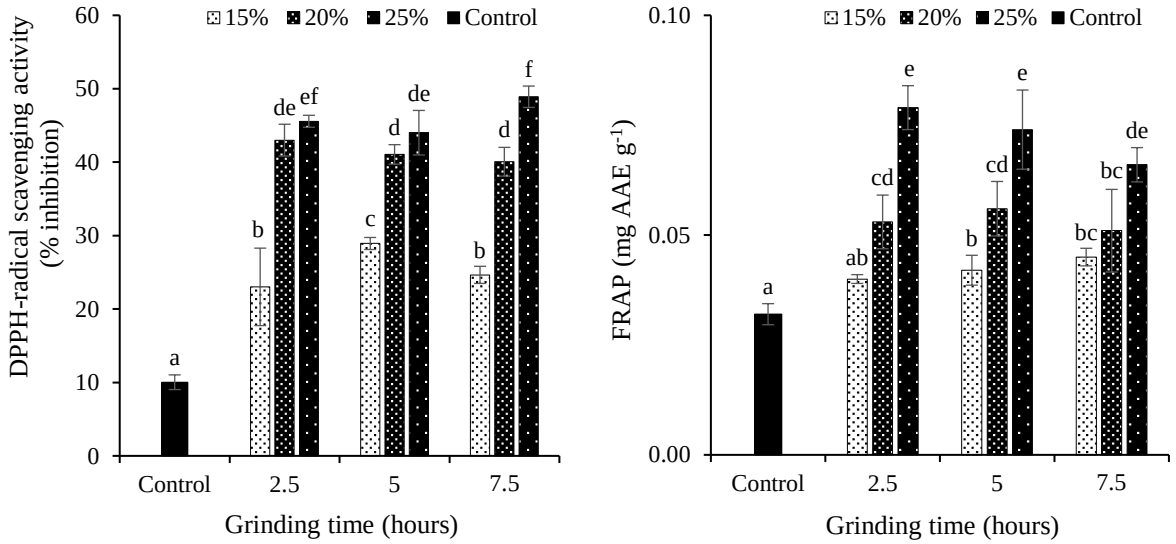

Figure 5. Antioxidant activity of chocolate formulated with pineapple powder tested using the DPPH method (a) and the FRAP method (b)

Note: Different superscripts indicate a significant difference ($p < 0.05$) among samples

Volatile aromatic compound

As shown in Table 3, about 125 compounds were detected in the GC-MS analysis. Furan, tetrahydro-3-methyl-, was predominantly detected in the chocolate control and in chocolate formulated with pineapple, at 29.6680 and 15.9933 ppb, respectively. Vanillin was also the major compound, with levels of 10.9013 and 20.1860 ppb in the chocolate control and chocolate formulated with pineapple, respectively. Some compounds initially found in the chocolate control, such as 2-Butyloctanol, Octane, 2-methyl-, Decyl octyl ether, and Hemellitol, were not detected in the chocolate formulated with pineapple. This may be because volatile compounds evaporated or their proportion in the chocolate decreased during the process. Meanwhile, some new compounds were detected after pineapple supplementation: δ-Caprolactone; Benzyl valerate; Benzaldehyde, 2,4-dihydroxy-; and γ-Decalactone, at about 0.0137; 0.0460; 0.5784 and 0.3571 ppb, respectively.

Several compounds were detected in both samples, but were present at higher concentrations in the white compound chocolate sample with 25% pineapple powder and a grinding duration of 7.5 hours. This means that these compounds naturally exist in both white chocolate and pineapple powder. These compounds included allyl caproate, acetic acid, propanoic acid, benzyl alcohol, ethyl maltol, caprylic acid, and lauric acid. The concentration of allyl caproate in the sample was 10.64 ppb, significantly higher than the concentration in the control sample (0.371 ppb). Allyl caproate has a pineapple and fruity odor (Zhu and Yu, 2010).

The concentration of acetic acid was also higher in the chocolate sample with 25% pineapple powder (3.21 ppb) and imparts a sour, vinegar-like, and astringent aroma (Aprotosoaie et al., 2016), contributing to a stronger aroma and sour taste (Tanamool et al., 2020). Propanoic acid was found at 3.00 ppb, which also contributed to the sour aroma. The thresholds for acetic acid and propanoic acid range from 0.03 to 1.00 ppm and 0.026 to 0.100 ppm, respectively. At a similar concentration, the compound with a lower threshold has a higher contribution to the odor of a product. Benzyl alcohol, which was also present at a higher concentration in the sample with 25% pineapple powder, has a sweet and floral aroma. Overall, the compounds detected at higher concentrations in the sample with 25% pineapple powder exhibited fruity, sweet, and pineapple-like aromas.

In the white compound chocolate sample with 25% pineapple powder and a grinding duration of 7.5 hours, several volatile compounds not found in the control sample were identified. These compounds included benzyl valerate (sweet, fruity, floral aroma), benzaldehyde, 2,4-dihydroxy- (sweet, floral, almond aroma),

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Table 3. Volatile aromatic compounds in white compound chocolate

No.	Compounds	Choc. control (ppb)	Choc. with pineapple powder (ppb)	Odor descriptor
1	Furan, tetrahydro-3-methyl-	29.6680	15.9933	Ether-like, sweet, fruity
2	Ethyl formate	3.8762	4.0775	-
3	Ethyl alcohol	0.6992	2.7039	Sweet, alcoholic[1]
4	1,3-Propanediol	0.6727	0.3748	-
5	iso-Amylnitrate	0.3282	0.1097	-
6	Benzene, (2,2-dimethylbutyl)-	0.7141	0.2152	-
7	3-Octen-2-ol	0.7683	0.3122	-
8	Decyl butyrate	0.4280	0.1111	
9	2-Butyloctanol	0.1934	-	-
10	Dodecane, 2,6,10-trimethyl-	0.3857	0.1439	-
11	Oxalic acid, bis (6-ethyloct-3-yl) ester	0.0888	-	-
12	Octane, 2-methyl-	0.1484	0.1333	-
13	Decyl octyl ether	0.2879	-	-
14	2-Hexyl-1-octanol	0.4495	-	-
15	m-Xylene	0.4883	0.1017	-
16	Ethylbenzene	0.3124	0.0341	-
17	o-Xylene	0.1687	0.0279	-
18	1-Butanol, 2-methyl-, (S)-	0.3032	0.1091	-
19	5-Methyl-2-hexene	0.1494	0.0444	-
20	Hexyl octyl ether	0.0730	-	-
21	p-Xylene	0.1393	0.0196	-
22	Dodecane	0.2792	0.1782	-
23	Undecane, 2,8-dimethyl-	0.0428	-	-
24	Isobutyl isobutyrate	0.1136	-	-
25	δ-Octalactone	0.0476	0.1799	Fruity, sweet[2]
26	Undecane, 4-ethyl-	0.0553	0.0493	-
27	Decane, 2,3,5-trimethyl-	0.2165	0.1663	-
28	Styrene	0.1790	0.1033	Fruity, flowery[2]
29	Undecane, 3,9-dimethyl-	0.0743	0.0612	-
30	Undecane, 3,3-dimethyl-	0.0675	0.1135	-
31	Hemellitol	0.0694	-	-
32	Furan, tetrahydro-2,5-dimethyl-	0.0391	-	Sweet, caramel-like, cocoa-like[3]
33	Acetylcarbinol	0.0373	0.1168	Pungent, sweet-caramellic, slightly burning[3]
34	Decane, 3-methyl-	0.1072	0.1535	-
35	Undecane, 3,5-dimethyl-	0.0400	0.0297	-
36	Decane	0.0577	0.0891	Gasoline-like[4]
37	Methyl tuberate	0.0455	0.0369	-
38	Ethyl heptanoate	0.0316	0.2883	Sweet, pleasant, fruity[3]
39	1,3-Cyclobutanedicarbonitrile,cis-	0.0364	-	-
40	Decane, 5-ethyl-5-methyl-	0.0636	0.0320	-
41	Allyl caproate	0.3711	10.6401	Pungent, fatty-fruity[3]
42	Caprylic acid, α-methyl-	0.0773	0.1212	Punget, fatty odor, rancid[3]
43	Nonanal	0.1381	0.1120	Tallowy, soapy-fruity[5]
44	Undecane	0.0434	0.0544	-
45	3-Octen-2-one	0.0313	0.0314	-

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Table 3. *Continue*

No.	Compounds	Choc. control (ppb)	Choc. with pineapple powder (ppb)	Odor descriptor
46	Cyclodecane	0.0132	0.0316	-
47	Benzene, 1,2-dichloro-	0.4618	0.3209	-
48	2,5-Dimethylanisole	0.0110	-	-
49	Acetic acid	0.0912	3.2107	Sour, vinegar astringent[6,7]
50	1-Octen-3-ol	0.0173	-	-
51	Furfural	0.0552	0.7079	Almond-like, sweet, caramel, herby, nutty[8,9]
52	Pyrazine, tetramethyl-	0.0141	0.0077	Chocolate, cocoa, coffee[10]
53	Furan, 2-propyl-	0.0274	0.0098	-
54	2-Ethylhexanol	0.1558	0.0305	Camphoraceous, herbal, solvent-like[3]
55	Cyclododecanol	0.0168	0.0065	-
56	3-Furancarboxylic acid	0.0043	0.0197	Sweet, caramel-like, burnt sugar[3]
57	2-Acetylfuran	0.0029	0.0156	Pleasant, sweet, caramel-like[3]
58	Benzaldehyde	1.7431	0.7632	Bitter, nutty, almond, pleasant[1,4,6]
59	1,3-Dithiolane, 2,2-dimethyl-	0.0191	0.0143	-
60	2-Dodecenal	0.0086	0.0163	-
61	Propanoic acid	0.0933	3.0099	Pungent, rancid, soy[14]
62	2-Pyridinecarboxaldehyde	0.0209	0.0215	-
63	3-Carene	0.0072	0.0047	-
64	α-Methylene butyrolactone	0.0144	0.0218	-
65	3,5-Octadien-2-one, (E,E)-	0.0165	0.0281	-
66	Furfural, 5-methyl-	0.0065	0.0157	Caramellic, bready, coffe-like[11]
67	1,2-Propanediol, 1-acetate	0.0078	0.1017	-
68	Methyl (methylsulfinyl)acetate	0.0175	0.0316	-
69	Dimethyl sulfoxide	0.0117	0.0215	-
70	Cyclopent-4-ene-1,3-dione	0.0333	0.0187	-
71	Propylene glycol	0.0295	1.2738	Faintly sweet[3]
72	Isopropyl alcohol	0.0107	0.9409	Sharp, alcoholic, solvent-like[3]
73	2-Acetoxy-1-propanol	0.0062	0.0594	-
74	Butyric acid	0.5081	1.1518	Rancid[3]
75	2-Hydroxyethyl propionate	0.0044	0.1837	-
76	3-p-Menthol	0.0136	0.0140	-
77	Levomenthol	0.0516	0.0198	-
78	2-t-Butyl-4-(dimethylbenzyl)phenol	0.1117	0.1383	-
79	2-Furanmethanol	0.1124	2.5136	Sweet, bready, caramel-like[3]
80	2-Methybutyric acid	0.0475	0.3118	Pungent, sweaty, rancid[3]
81	Acetic acid, 1-(S)-phenylethyl ester	0.0307	-	-
82	Benzyl acetate	0.0046	0.5985	Floral, jasmine, rose, fresh[12]
83	Naphthalene	0.7599	0.4006	Mothball, tar-like[1]

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Table 3. *Continue*

No.	Compounds	Choc. control (ppb)	Choc. with pineapple powder (ppb)	Odor descriptor
84	Valeric acid	0.0150	0.0268	sweat, acid, rancid[16]
85	2(5H)-Furanone	0.0136	0.0193	-
86	2-Decenal, (E)-	0.0065	0.0099	-
87	Acetamide	0.0133	0.0442	-
88	Oxime-, methoxy-phenyl-	0.1470	0.1549	-
89	δ-Caprolactone	-	0.0137	Sweet, creamy, lactonic, tobacco, and coumarin-like with traces of green coconut[13]
90	Benzyl propionate	0.0121	0.6324	Fruity, sweet, balsamic[3]
91	Allyl cyclohexylpropionate	0.0096	0.1345	Fruity, pineapple-like, sweet[3]
92	Methyl laurate	0.0104	0.1272	-
93	Isophthalaldehyde	0.1645	0.0364	Strong, sharp, almond-like[3]
94	4-Propylbenzaldehyde	0.0368	0.1942	sweet, fruity, floral[3]
95	Caproic acid	1.1898	2.0625	Punget, sickening, rancid, sour[11]
96	o-Guaiacol	0.0199	0.0402	Roasted, smoked, sweet[2]
97	Benzyl alcohol	0.1066	9.8611	Sweet, floral[6]
98	Dimethyl sulfone	1.0695	1.5454	-
99	2-Piperidinone	0.0118	0.0156	-
100	Butylated hydroxytoluene	0.1059	0.1061	-
101	Di-t-butyl-4-butylphenol	0.1518	0.1684	-
102	Enanthic acid	0.0450	0.0704	-
103	Maltol	0.0597	0.1196	Sweet, caramel-like[3]
104	2-(Hydroxyacetyl)furan	0.0102	0.0539	-
105	Ethyl maltol	0.0036	21.5097	Sweet sugary, caramelized, strawberry-like[3]
106	Caprylic acid	0.9069	11.1166	Slightly unpleasant, rancid-like smell
107	Benzyl valerate	-	0.0460	Fruity, floral[3]
108	Triacetin	0.0104	0.2384	Creamy, slightly acidic[14]
109	Benzaldehyde, 2,4-dihydroxy-	-	0.5784	Sweet, floral, almond[3]
110	γ-Decalactone	-	0.3571	Sweet-peach-like[3]
111	γ-Hydroxyisoeugenol	0.0316	0.0134	-
112	Eugenol	0.0102	-	Warm, clove-like, somewhat woody scent[15]
113	Pelargic acid	0.0397	0.4477	-
114	Caprolactam	0.0038	0.0095	-
115	δ-Octalactone	0.0212	0.0271	Fruity, sweet[2]
116	Piperonal	0.0163	0.0181	-
117	α-Monoacetin	0.0021	0.0484	-
118	Capric acid	0.0591	1.6504	-
119	Benzoic acid	0.0197	0.0427	Sour[17]
120	Lauric acid	0.0533	1.3867	Mild, fatty odor[3]

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Table 3. *Continue*

No.	Compounds	Choc. control (ppb)	Choc. with pineapple powder (ppb)	Odor descriptor
121	5-Hydroxymethylfurfural	-	0.0674	Caramellic, musty, buttery[3]
122	Vanillin	10.9013	20.1860	Vanilla, sweet, cocoa[2]
123	Piperonol	0.0291	0.0302	-
124	Myristic acid	0.0472	0.1000	Faintly fatty, soapy[3]
125	*Palmitic acid*	0.2367	0.6814	-

References: [1] = Ruth (1986); [2] = Deuscher et al. (2020); [3] = Arctander (2017); [4] = Guzmán Penella et al. (2023); [5] = Crafack et al. (2014); [6] = Aprotosoaie et al. (2016); [7] = Rodriguez-Campos et al. (2011); [8] = Starowicz (2021); [9] = Shi et al. (2013); [10] = Rottiers et al. (2019); [11] = Al Tamimi et al. (2023); [12] = Britto de Andrade et al. (2021); [13] = Chen et al. (2017); [14] = Michel et al. (2021); [15] = Zarzo et al. (2015); [16] = Magalhães da Veiga Moreira et al. (2017)

γ-decalactone (sweet, peach, fruity aroma), and 5-hydroxymethylfurfural (caramellic, musty, buttery aroma). Pineapple contains various aromatic volatile compounds, including esters, lactones, acids, and hydrocarbons (Wei et al., 2011). Over 130 volatile compounds in fresh pineapple and its processed products, including γ-decalactone, 5-hydroxymethylfurfural, benzaldehyde, furfural, acetic acid, and propanoic acid, have been identified in a previous report (Elss et al., 2005). This implies that pineapple powder can contribute to the aroma of chocolate, and thus, it can potentially be used to create a new variant of chocolate.

Table 3 also lists several volatile compounds that were uniquely detected in the control sample, including 2-butyloctanol, oxalic acid bis(6-ethyloct-3-yl) ester, decyl octyl ether, 2-hexyl-1-octanol, hexyl octyl ether, undecane, 2,8-dimethyl-; isobutyl isobutyrate, hemellitol, furan, tetrahydro-2,5-dimethyl-; 1,3-cyclobutanedicarbonitrile, *cis*-; 2,5-dimethylanisole, 1-octen-3-ol, and acetic acid, 1-(s)-phenylethyl ester. Chocolate samples contain 100 volatile compounds, including acids, alcohols, aldehydes, ketones, esters, furans, pyrazines, pyrroles, and pyridines (Cemin et al., 2022). Pervious research identified over 100 volatile compounds in dark chocolate, including acids (acetic acid, propanoic acid), alcohols and phenols (benzyl alcohol, guaiacol, 1-octanol), aldehydes and ketones (nonanal, heptanal), esters (tiacetin, ethyl benzoate), furans, furanones, pyranones (furfuryl alcohol, 2-pentylfuran), hydrocarbons (undecane), and lactones (δ-octalactone) (Michel et al., 2021).

Some compounds were present in both samples, but at higher concentrations in the control sample. This is likely due to the impact of grinding time, which can reduce or even eliminate volatile compounds. Certain volatile compounds, such as acids, pyrazines, phenols, aldehydes, and ketones, diminish during conching (Albak and Tekin, 2016).

CONCLUSIONS

The study found that varying pineapple powder concentration and grinding duration slightly affected physical traits like color, while hardness increased notably at 20 to 25% powder with 5 to 7.5 hours of grinding. Apparent viscosity also increased with higher powder levels and longer grinding. Pineapple addition enhanced total phenolics, flavonoids, and antioxidant activity (DPPH, FRAP), and introduced new volatiles (benzyl valerate, benzaldehyde, 2,4-dihydroxy-, γ-decalactone, 5-hydroxymethylfurfural). The research demonstrates the potential of pineapple powder in chocolate and offers opportunities for the development of novel tropical-flavored products. However, further studies are required to evaluate market competitiveness, including comparative consumer trials and shelf-life assessments for commercialization.

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