CELEBES Agricultural 6(2): 56 - 65 February (2026)

Research Article

Effect of Roasting Time on Total Phenolic Content and Antioxidant Activity of Cocoa Bean Shells

Pengaruh Waktu Sangrai terhadap Total Fenol dan Aktivitas Antioksidan Kulit Biji Kakao

Nadirah B Andi Pallawa[1*], Miming Berlian Hi. B Salam[1] , Laras Ayu[1]

[1] Study Program of Agro-Product Processing Technology, Politeknik Palu, Jl. Sinar Kemuning I No. 1A, Palu, Central Sulawesi 94119, Indonesia

*email: nadirahbap@gmail.com

Received: July, 2025
Accepted: April, 2026
Published: April, 2026

p-ISSN: 2723-7974
e-ISSN: 2723-7966
doi: 10.52045/jca.v6i2.1015

Website: https://ojs.untika.ac.id/index.php/faperta

Abstract: Cocoa bean shells are a by-product of cocoa processing that contain bioactive compounds, particularly phenolics, with potential antioxidant activity. However, information on the effect of roasting time on the total phenolic content and antioxidant activity of fermented cocoa bean shells remains limited. Therefore, this study evaluated the effect of roasting time on the total phenolic content and antioxidant activity of fermented cocoa bean shells derived from Forastero cocoa beans (BR 25 variety) from Bolang Village, Enrekang Regency. A laboratory experiment was conducted using a drum roaster with a charge temperature of 135°C and roasting times of 10, 14, 18, and 22 min. Roasting time significantly affected total phenolic content and antioxidant activity. Roasting for 14 min produced the highest mean total phenolic content (30.1425 mg GAE/g extract) and the lowest IC50 value (83.69 ppm), although these results were not significantly different from those at 18 min. In contrast, the 10 and 22 min treatments showed lower total phenolic content and weaker antioxidant activity. These findings suggest that moderate roasting durations, particularly 14–18 min, were more favorable for maintaining the bioactive properties of cocoa bean shells.

Keywords: Antioxidant Activity, Cocoa Bean Shells, IC50, Phenolic Compounds, Roasting Time

Abstrak: Kulit biji kakao merupakan produk samping pengolahan kakao yang mengandung senyawa bioaktif, terutama fenolik, dan berpotensi sebagai sumber antioksidan alami. Namun, informasi mengenai pengaruh waktu penyangraian terhadap kandungan total fenol dan aktivitas antioksidan pada kulit biji kakao fermentasi masih terbatas. Oleh karena itu, penelitian ini bertujuan untuk mengevaluasi pengaruh waktu penyangraian terhadap kandungan total fenol dan aktivitas antioksidan kulit biji kakao fermentasi yang berasal dari biji kakao Forastero varietas BR 25 dari Desa Bolang, Kabupaten Enrekang. Penelitian dilakukan secara eksperimen laboratorium menggunakan drum roaster dengan charge temperature 135°C dan waktu penyangraian 10, 14, 18, dan 22 menit. Waktu penyangraian berpengaruh nyata terhadap kandungan total fenol dan aktivitas antioksidan. Penyangraian selama 14 menit menghasilkan rerata kandungan total fenol tertinggi (30,1425 mg GAE/g ekstrak) dan nilai IC50 terendah (83,69 ppm), meskipun hasil tersebut tidak berbeda nyata dengan penyangraian selama 18 menit. Sebaliknya, perlakuan 10 dan 22 menit menunjukkan kandungan total fenol yang lebih rendah dan aktivitas antioksidan yang lebih lemah. Temuan ini menunjukkan bahwa durasi penyangraian sedang, khususnya 14–18 menit, lebih baik dalam mempertahankan sifat bioaktif kulit biji kakao.

Kata kunci: Aktivitas antioksidan, IC50, Kandungan fenolik, Kulit biji kakao, Waktu penyangraian

Citation:
Pallawa NBA, Salam MBHB, Ayu L. 2026. Effect of Roasting Time on Total Phenolic Content and Antioxidant Activity of Cocoa Bean Shells. *CELEBES Agricultural*. 6(2): 56-65. doi: 10.52045/jca.v6i2.1015

INTRODUCTION

Cocoa bean shells are a promising source of bioactive compounds, particularly phenolic compounds and antioxidants, with potential for food and health-related applications. They are also generated in large quantities as a by-product of chocolate processing, creating opportunities for their utilization as value-added products (Lembong et al., 2021; Barbosa-Pereira et al., 2021; Sánchez et al., 2023). However, their functional potential may be influenced by cocoa processing steps, particularly fermentation and roasting, which can alter their phenolic content and antioxidant-related bioactive composition (Agustriana et al., 2023; Lembong et al., 2021).

Roasting induces chemical reactions that may modify phenolic compounds and antioxidant activity in cocoa-derived materials. Previous studies have shown that variations in roasting temperature and time can influence bioactive compounds and antioxidant capacity. Moderate roasting may enhance the extractability of phenolic compounds and antioxidant activity, whereas excessive roasting may reduce them because of the thermal degradation of heat-sensitive compounds. Cortez et al. (2024) found that roasting was one of the postharvest stages that most strongly affected phenolic compounds in cocoa, particularly epicatechin, catechin, gallic acid, and caffeic acid. Similar trends have been reported in other roasted plant materials, where intermediate roasting conditions preserved or enhanced bioactive compounds and antioxidant properties, while more intense roasting caused significant losses (Shawky et al., 2024; Liu et al., 2024; Spychaj et al., 2025).

These findings suggest that roasting conditions play an important role in determining the functional quality of cocoa-derived materials. However, only a limited number of studies have specifically addressed polyphenol content and antioxidant activity in cocoa bean shells after roasting. Although Pallawa et al. (2025) examined roasting time, the focus was on cocoa nibs rather than cocoa bean shells. Therefore, the effect of roasting time on the total phenolic content and antioxidant activity of fermented cocoa bean shells remains unclear.

This study aimed to determine how different roasting times influence these parameters to identify roasting conditions that may preserve or enhance the functional value of cocoa bean shells and support their potential application in value-added food products.

MATERIALS AND METHODS

Equipment and Materials

The equipment used in this study comprised a drum roaster with a 1 kg capacity per batch. It is a cylindrical, horizontally oriented device that is rotated manually and equipped with a digital thermometer to monitor the drum temperature. Additional equipment comprised an analytical balance, 10 mL volumetric flasks, volumetric pipettes, a UV-Vis spectrophotometer, quartz or glass cuvettes, and test tubes. The laboratory setup also required a vortex mixer, dark incubation chamber, Erlenmeyer flasks, rotary vacuum evaporator, amber vials, micropipettes, measuring cylinders, stationery, and a laptop.

The materials used in this study were cocoa beans (Forastero type, BR 25 variety) sourced from Bolang Village, Enrekang Regency, and liquefied petroleum gas (LPG). The chemicals and reagents used were 95% ethanol, 50 µM DPPH solution, gallic acid (10 mg), distilled water, Folin-Ciocalteu reagent, and 20% anhydrous sodium carbonate (Na_2CO_3). Additional materials comprised zipper-lock plastic packaging, Whatman filter paper, an aluminum foil roll, tissue paper, labels, and latex gloves.

Research Method

A laboratory-based experimental method was used. The study utilized a single-factor experimental design focused on the roasting duration. Four treatments were implemented, each replicated four times, as described below.

T1 = 10 min

T2 = 14 min

T3 = 18 min

T4 = 22 min

a. Sample Preparation

Cocoa beans were fermented for five days and then sun-dried under direct sunlight for five days. The dried beans were cleaned to remove foreign materials and sorted to eliminate damaged specimens. The beans were standardized in size to ensure uniformity

b. Cocoa Bean Roasting

The roasting process was adapted from Pallawa et al. (2023) with minor modifications. For each treatment, 250 g of cocoa beans was roasted in a drum roaster using a charge temperature of 135˚C. Roasting commenced upon bean insertion. After the designated roasting period (10, 14, 18, or 22 min), the cocoa shells were separated from the nibs and collected for subsequent analysis.

c. Analytical Procedures

The following analyses were conducted to evaluate the total phenolic content and antioxidant activity of cocoa bean shells:

1. Preparation of Concentrated Extracts

Cocoa shell extracts were prepared using a maceration technique adapted from Datu et al. (2023). Roasted cocoa bean shells were ground and stored in airtight, dark containers. Five grams of the powdered sample was macerated with 95% ethanol in a ratio of 1:5 for 24 h, repeated thrice. The combined filtrates were filtered and concentrated using a rotary vacuum evaporator until a viscous extract was obtained for analysis.

2. Determination of Total Phenolic Content

The total phenolic content of the cocoa shell extracts was determined using the Folin–Ciocalteu method, as described by Datu et al. (2023) and Hidayah et al. (2020). A gallic acid

calibration curve was prepared by dissolving 10 mg of gallic acid in 10 mL of a 1:1 ethanol-water mixture. The solution was diluted to obtain concentrations of 10, 20, 30, 40, 50 mg/L. For calibration, 1 mL of each standard was mixed with 10 mL of distilled water and 1 mL of Folin–Ciocalteu reagent. After 8 min, 3 mL of 20% Na_2CO_3 solution was added. The mixture stood for 2 hours. Absorbance was then measured at 765 nm to construct the calibration curve.

For sample analysis, 10 mg of cocoa shell extract was dissolved in 10 mL of an ethanol-water mixture. One milliliter of this solution was treated identically to the gallic acid standards. The total phenolic content was calculated using the regression equation of the calibration curve. The results were expressed as mg gallic acid equivalents per gram of extract (mg GAE/g extract).

3. Determination of Antioxidant Activity Using the DPPH Method (IC50)

Antioxidant activity was assessed using the DPPH radical scavenging method, as described by Datu et al. (2023) and Hidayah et al. (2020). A 10 mg sample of the extract was dissolved in 10 mL of ethanol to prepare a 1000 ppm stock solution. Serial dilutions were prepared to reach concentrations of 20, 40, 60, 80, and 100 ppm. Each 1 mL aliquot was mixed with 3 mL of 50 µM DPPH solution, homogenized, and incubated in the dark for 30 min. Absorbance was measured at 517 nm. The percentage of inhibition was calculated, and the IC50 value was determined from the regression equation of the inhibition curve.

RESULTS AND DISCUSSION

Total Phenolic Content

The results confirmed that cocoa bean shell extract, obtained using ethanol extraction, contained phenolic compounds with antioxidant activity, as determined by spectrophotometric analysis.

Gallic Acid Standard Curve

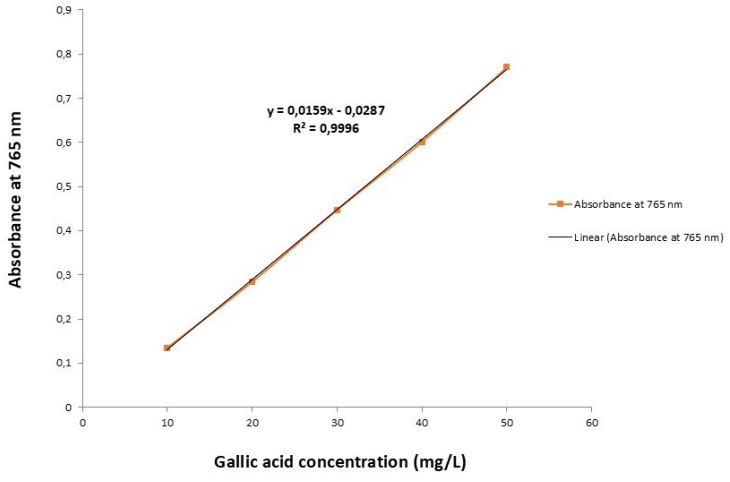

Figure 1. Gallic acid standard curve for the determination of total phenolic content.

The gallic acid standard curve showed a linear relationship between concentration and absorbance at 765 nm, with the regression equation $y = 0.01587x - 0.02870$ and a coefficient of determination (R^2) of 0.9996. This result indicates excellent linearity of the calibration curve and confirms that it is suitable for determining the total phenolic content of cocoa bean shell extract.

The total phenolic content of the cocoa bean shell extract varied with roasting time. Figure 2 shows how prolonged roasting alters the concentration of phenolic compounds.

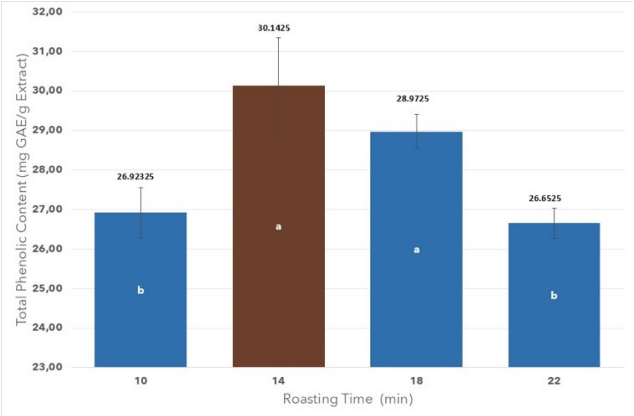

Figure 2. Total phenolic content of cocoa bean shell extract at different roasting times. Bars with different letters indicate significant differences ($p < 0.05$) according to Tukey's honestly significant difference test.

Roasting for 14 min resulted in the highest total phenolic content, as measured by spectrophotometric analysis at 765 nm using the Folin-Ciocalteu method. One-way analysis of variance revealed that roasting time significantly affected total phenolic content ($F = 18.819$, $p < 0.001$). Tukey's honest significant difference test showed that the 14 min treatment was not significantly different from the 18 min treatment but differed significantly from the 10 min and 22 min treatments. Likewise, the 10 and 22 min treatments were not significantly different from each other.

These findings demonstrate that roasting time significantly affects the total phenolic content of cocoa bean shell extract. Previous studies have shown that thermal processing may temporarily increase phenolic content by releasing bound compounds from the plant matrix; however, prolonged heating may reduce it due to oxidative and thermal degradation. This pattern is consistent with the present results, in which the total phenolic content increased at 14 min, remained statistically similar at 18 min, and then declined at 22 min.

The increase in total phenolic content under moderate roasting may be associated with the improved extractability of phenolic compounds during heating. Similar trends have been reported by Kataria et al. (2021) in teff grains and by Calinoiu and Vodnar (2020) in wheat and oat bran, where thermal processing enhanced the release of phenolic compounds. In cocoa bean shell, Lembong et al. (2021) also reported that roasting influenced polyphenol content and antioxidant activity, indicating that thermal treatment plays an important role in determining its functional properties.

In contrast, the decrease in total phenolic content at longer roasting times may be attributed to the degradation of heat-sensitive phenolic compounds during extended heat exposure. Prolonged heating can promote oxidation and structural degradation of phenolic compounds, thereby reducing their concentration in the extract. This explanation is in line with Calinoiu and Vodnar (2020), who reported that excessive thermal treatment increases the risk of phenolic degradation.

Thus, the present study suggests that moderate roasting durations, particularly 14–18 min, are more favorable for maintaining total phenolic content than shorter or longer roasting times. Although roasting for 14 min produced the highest mean total phenolic content, it was statistically similar to roasting for 18 min.

Antioxidant Activity

The antioxidant activity of cocoa bean shell extract, expressed as IC50 values from the DPPH radical scavenging assay, varied significantly with roasting duration (Figure 3). The IC50 values at 10, 14, 18, and 22 min were 133.61 ± 16.42, 83.69 ± 10.36, 105.63 ± 9.75, and 140.79 ± 10.79 ppm, respectively. The lowest IC50 value was observed at 14 min, indicating the strongest antioxidant activity. Tukey's honestly significant difference (HSD) test revealed that the 14 min treatment did not differ significantly from the 18 min treatment, but both showed significantly lower IC50 values than the 10 and 22 min treatments. In contrast, the 10 and 22 min treatments were not significantly different from each other.

These results indicate that roasting time significantly affected the antioxidant activity of cocoa bean shell extract, as reflected by the IC50 values. Roasting for 14 min produced the lowest IC50 value, indicating the strongest antioxidant activity, although it was not significantly different from roasting for 18 min. Previous studies have shown that moderate thermal treatment can enhance antioxidant activity by improving the release or extractability of phenolic and other bioactive compounds, whereas excessive heating may reduce antioxidant capacity because of thermal degradation and oxidative changes. Similar trends were reported by Rodríguez-Solana et al. (2019), and Hatamian et al. (2020), who found that appropriate roasting conditions could enhance antioxidant-related properties in plant materials.

In the present study, the increase in IC50 after moderate roasting suggests a decline in antioxidant activity with prolonged heat exposure. This may be attributed to the degradation of heat-sensitive antioxidant compounds during roasting. Therefore, moderate roasting durations, particularly 14–18 min, appear to be more favorable for maintaining antioxidant activity than shorter or longer roasting times.

These insights suggest that optimizing thermal conditions can be an effective strategy for enhancing the antioxidant capacity of food products. The current study strongly supports this finding, highlighting the importance of selecting ideal roasting parameters to maximize the health-promoting properties of cocoa-based ingredients.

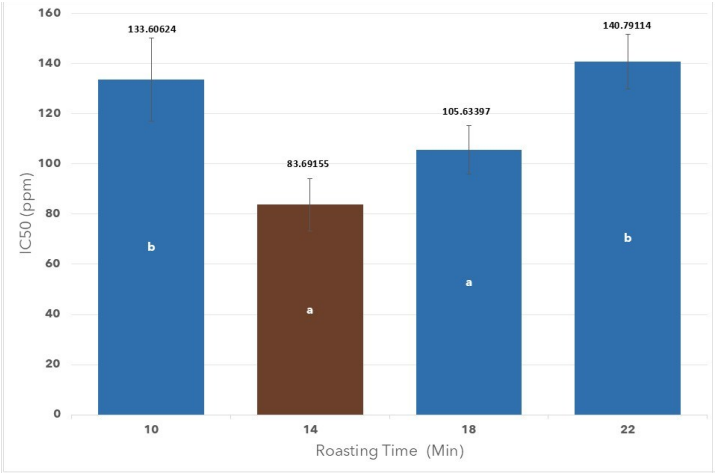

Figure 3. Antioxidant activity of cocoa bean shell extract at different roasting times based on the DPPH assay. Bars with different letters indicate significant differences at $p < 0.05$ according to Tukey's HSD test.

Correlation Between Total Phenolic Content and Antioxidant Activity

Figure 4 shows a strong negative correlation between total phenolic content and IC50 values, with the regression equation $y = -15.245x + 545.42$ and $R^2 = 0.9272$. As the phenolic content increased, the IC50 values decreased, indicating stronger antioxidant activity. This result suggests that phenolic compounds play an important role in the antioxidant capacity of cocoa bean shell extract.

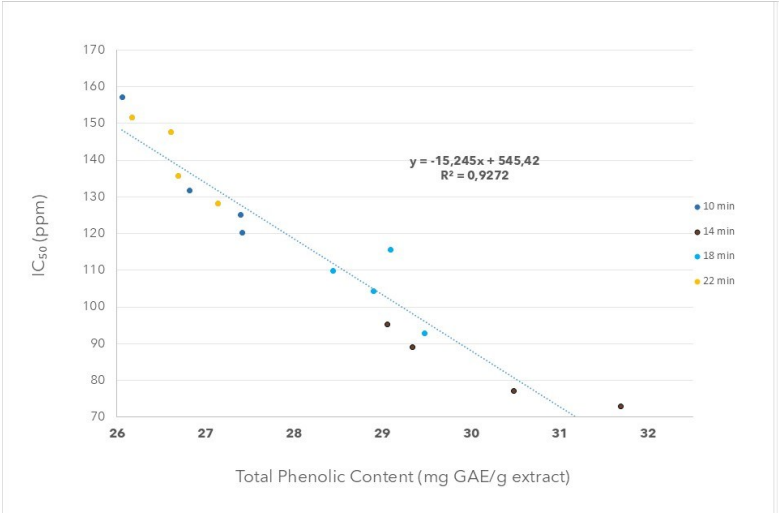

Figure 4. Correlation between total phenolic content and IC50 values of cocoa bean shell extract at different roasting times.

These findings are in agreement with previous studies showing that roasting under appropriate conditions can enhance phenolic content and antioxidant activity, as reported for rapeseed oil by Siger et al. (2017), carob by Rodríguez-Solana et al. (2019), and chia seeds by

Hatamian et al. (2020). Such evidence underscores the importance of optimizing roasting conditions to maximize the antioxidant potential associated with phenolic compounds.

In the present study, antioxidant activity was strongly associated with total phenolic content. Roasting for 14 min produced the strongest antioxidant activity, although it was statistically similar to roasting for 18 min. This finding suggests that phenolic compounds play an important role in the antioxidant capacity of cocoa bean shell extract. However, because the IC50 values remained above 50 ppm, a higher extract concentration may still be required to achieve greater antioxidant effectiveness.

A major strength of this study is the use of a roasting method that closely resembles small-scale industrial practice, enhancing its practical relevance and potential applicability. Nevertheless, the relatively high IC50 values may limit the extract's effectiveness in certain applications. Therefore, further studies involving alternative processing methods, extraction conditions, and larger-scale systems are warranted to confirm and extend these findings.

CONCLUSIONS

Roasting duration significantly affected the total phenolic content and antioxidant activity of fermented cocoa bean shells. Moderate roasting durations (14–18 min) were more favorable than shorter or longer roasting times. Roasting for 14 min produced the highest mean total phenolic content and the lowest IC50 value, although it was statistically similar to roasting for 18 min. These results suggest that controlled roasting may improve the functional potential of cocoa bean shells; however, further optimization is required to enhance antioxidant effectiveness.

ACKNOWLEDGMENT

The authors gratefully acknowledge the Directorate of Research and Community Service (DRTPM) for providing research grant funding under the 2024 fiscal year scheme. This support significantly aided the implementation of this study. We thank the laboratory staff at Politeknik Palu for their technical assistance during the roasting process and for utilizing the provided equipment. We also appreciate Universitas Tadulako for providing access to laboratory facilities and support during spectrophotometric analysis. Special thanks are due to the local cocoa farmers in Bolang Village, Enrekang Regency, who supplied the fermented cocoa beans used in this research. The authors also thank their colleagues for contributing academic input and feedback on the manuscript draft.

REFERENCES

Agustriana, E., Valentino, H. A., Rahmani, N., Nuryati, N., Firmanto, H., Rachmayati, R., Yulianti, S. E., Nuryana, I., Yopi, Y., & Lisdiyanti, P. (2023). Fermentation effect of cacao beans originate from Jember on polyphenol-flavonoid content and radical scavenging activity. *Alchemy: Jurnal Penelitian Kimia*, 19(1), 23–31. https://doi.org/10.20961/alchemy.19.1.60831.23-31

Barbosa-Pereira, L., Belviso, S., Ferrocino, I., Rojo-Poveda, O., & Zeppa, G. (2021). Characterization and classification of cocoa bean shells from different regions of Venezuela using HPLC-PDA-MS/MS and spectrophotometric techniques coupled to chemometric analysis. *Foods*, 10(8), 1791. https://doi.org/10.3390/foods10081791

Calinoiu, L. F., & Vodnar, D. C. (2020). Thermal processing for the release of phenolic compounds from wheat and oat bran. Biomolecules, 10(1), 21. https://doi.org/10.3390/biom10010021

Cortez, D., Flores, M., Calampa, L., Oliva-Cruz, M., Gonas, M., Meléndez-Mori, J. B., & Chavez, S. G. (2024). From the seed to the cocoa liquor: Traceability of bioactive compounds during the postharvest process of cocoa in Amazonas-Peru. *Microchemical Journal*, 201, 110607. https://doi.org/10.1016/j.microc.2024.110607

Datu, O. S., Lebang, J. S., & Lestari, U. S. (2023). Uji aktivitas antioksidan, analisis total fenol, flavonoid, dan tanin dari ekstrak buah salak. Pharmacy Medical Journal, 6(2), 117–122.

Hatamian, M., Noshad, M., Abdanan-Mehdizadeh, S., & Barzegar, H. (2020). Effect of roasting treatment on functional and antioxidant properties of chia seed flours. *NFS Journal*, 21, 1–8. https://doi.org/10.1016/j.nfs.2020.07.004

Hidayah, N., Fitriansyah, S. N., Aulifa, D. L., Dewi, S., & Barkah, W. (2020). Determination of total phenolic, flavonoid content and antioxidant activity of Campolay (*Pouteria campechiana* (Kunth) Baehni) extract. *Advances in Health Sciences Research*, 26, 107–110.

Kataria, A., Sharma, S., & Dar, B. N. (2021). Changes in phenolic compounds, antioxidant potential and antinutritional factors of teff (Eragrostis tef) during different thermal processing methods. *International Journal of Food Science & Technology*. https://doi.org/10.1111/ijfs.15210

Lembong, E., Djali, M., Zaida, Z., & Utama, G. L. (2021). The potential of dry fermented cocoa (*Theobroma cacao* L.) variety Lindak bean shell treated at different degrees of roasting as a functional food. *IOP Conference Series: Earth and Environmental Science*, 733(1), 012068. https://doi.org/10.1088/1755-1315/733/1/012068

Liu, Y., Chin, F. W. L., Huang, D., Liu, S.-Q., & Lu, Y. (2024). Thermal degradation of glucomoringin and changes of phenolic compounds in moringa seed kernels during different degrees of roasting. Food Chemistry, 454, 139782. https://doi.org/10.1016/j.foodchem.2024.139782

Pallawa, N. B. A., Salengke, & Pirman. (2023). Effects of roasting process on the precursor components for Maillard reaction in fermented cocoa beans. AIP Conference Proceedings, 2596(1), 020002. https://doi.org/10.1063/5.0119776

Pallawa, N. B. A., Salam, M. B. H. B., & Mantoki, S. (2025). Pengaruh waktu penyangraian terhadap aktivitas antioksidan biji kakao fermentasi. *Media Pertanian*, 10(2), 121–131. https://doi.org/10.37058/mp.v10i2.15176

Rodríguez-Solana, R., Salgado, J. M., Pérez-Santín, E., & Romano, A. (2019). Effect of carob variety and roasting on the antioxidant capacity and the phenolic and furanic contents of carob liquors. *Journal of the Science of Food and Agriculture*, 99(6), 2697–2707. https://doi.org/10.1002/jsfa.9437

Sánchez, A., Allueva, S., Vazquez, L., & Nerín, C. (2023). Cocoa shell: A by-product with potential applications in the food industry. *Antioxidants*, 12, 1028. https://doi.org/10.3390/antiox12051028

Shawky, E., Nada, A. A., Abdelhameed, R. F. A., El-Maadawy, W. H., Ibrahim, R. S., & Elgamal, A. M. (2024). Dynamic changes in the bioactive metabolome of Coffea arabica beans during postharvest roasting processes in relation to time and temperature. *Food Chemistry*, 454, 139900. https://doi.org/10.1016/j.foodchem.2024.139900

Spychaj, R., Riahi, A., Han, J., Aljohani, M. M., Alelwani, W., Fershtat, L. L., & Kumar, A. (2025). Effect of light, medium and dark roasting on the concentration of bioactive compounds and antioxidant properties in the kernels of selected amaranth genotypes. *Food Chemistry*, 466, 141894. https://doi.org/10.1016/j.foodchem.2024.141894

Siger, A., Gawrysiak-Witulska, M., & Bartkowiak-Broda, I. (2017). Antioxidant (tocopherol and canolol) content in rapeseed oil obtained from roasted yellow-seeded *Brassica napus*. *Journal of the American Oil Chemists' Society*, 94, 37–46. https://doi.org/10.1007/s11746-016-2921-7