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Impact of Boiling, Ultrasonic, and Microwave–Ultrasonic Assisted Extraction on Phenolic Content, Antioxidant Activity, and Sun Protection Factor of Black Tea (*Camellia sinensis*) Extracts

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ABSTRACT

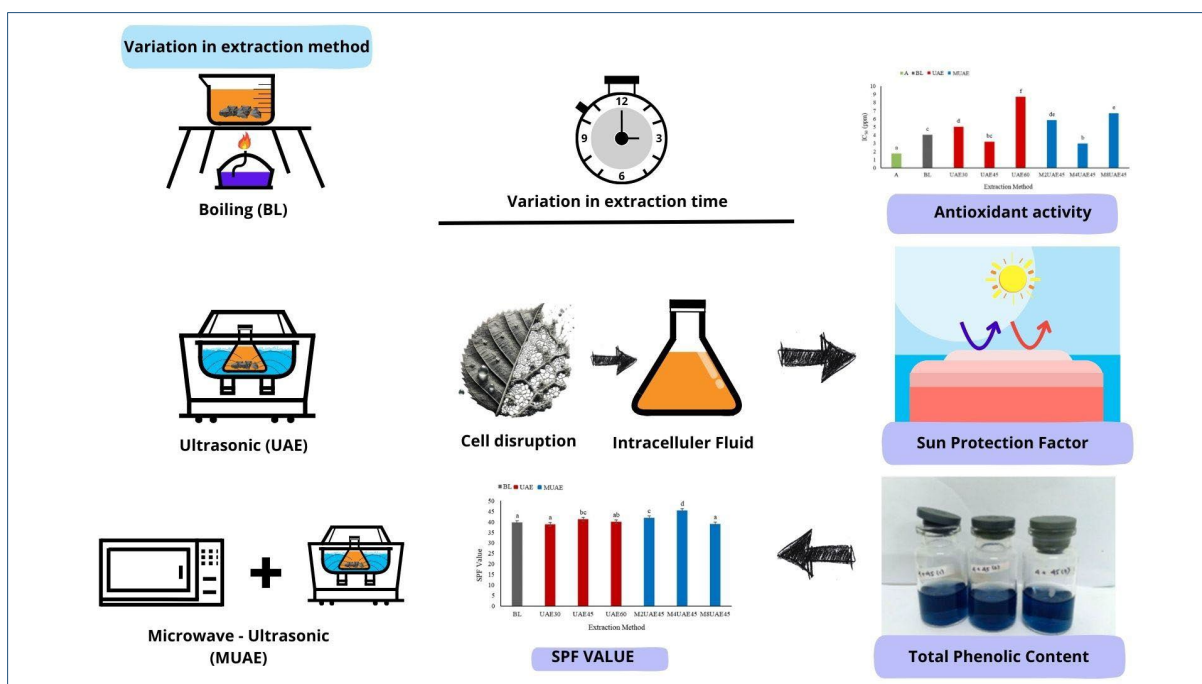
The extraction efficiency of bioactive compounds from *Camellia sinensis* (black tea) strongly influences its antioxidant and photoprotective performance. This study compared three water-based extraction methods—boiling (BL), ultrasonic-assisted extraction (UAE), and microwave–ultrasonic-assisted extraction (MUAE)—in terms of extraction yield, total phenolic content (TPC), antioxidant activity, and *in vitro* sun protection factor (SPF). The combined MUAE treatment, consisting of 4 min of microwave pre-heating followed by 45 min of ultrasound (M4UAE45), produced the most effective extract, exhibiting the highest TPC (457.64 ± 2.31 mg GAE/g), the lowest IC₅₀ value (2.97 ± 0.41 ppm), and the highest SPF (45.4 ± 0.59). This study provides a novel and systematic comparison of fully water-based MUAE with conventional boiling and UAE methods for black tea and to the best of our knowledge, this study establishes a direct correlation between optimized water-based MUAE conditions and enhanced phenolic recovery and SPF performance. These improvements arise from a synergistic cell disruption mechanism in which microwave-induced structural damage and ultrasonic cavitation promote rapid and efficient release of phenolic constituents. FTIR spectra supported the presence of predominant hydroxyl and aromatic functional groups characteristic of polyphenols, supporting the higher phenolic abundance observed in MUAE extracts. Overall, the M4UAE45 protocol emerges as a green, efficient, and scalable strategy for producing phenolic-rich black tea extracts with strong antioxidant and UV-protective properties, highlighting their potential application as natural active ingredients in cosmetic and pharmaceutical sunscreen formulations.

Keywords: Antioxidant activity, *Camellia sinensis*, Microwave–ultrasonic assisted extraction, Sun protection factor, Total phenolic content

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Graphical Abstract:



INTRODUCTION

Ultraviolet (UV) radiation from sunlight poses significant risks to skin health, leading to premature aging, oxidative stress, and increased skin cancer risk due to DNA damage (Sharma, 2024). The use of sunscreens is an effective strategy for protecting the skin by absorbing, reflecting, or scattering harmful UV rays (Chen, 2024). In recent years, there has been growing interest in incorporating natural ingredients into sunscreen formulations due to their minimal side effects and added benefits for skin health, including moisturizing, firming, and anti-inflammatory properties (Sieniawska et al., 2024).

Among these natural ingredients, tea (*Camellia sinensis*) has gained attention for its rich polyphenol and catechin content, which exhibit strong antioxidant and photoprotective effects. Studies have demonstrated that tea polyphenols, particularly catechins, can mitigate UVB-induced skin damage and reduce the risk of skin cancer (Bhattacharya and Sherje, 2020; Zheng et al., 2024). Indonesia contributes approximately 2% to the global tea trade, primarily exporting black tea (82.16% of total exports), positioning it as a sustainable source for cosmeceutical applications (Muflihah et al., 2023).

Black tea, produced through oxidation during fermentation, contains elevated levels of polyphenols that neutralize UV-induced reactive oxygen species (ROS) and exhibit anti-inflammatory properties (Sopyan et al., 2019; Fakhri et al., 2024).

Although numerous studies have compared extraction techniques for tea polyphenols, most focus on yield and antioxidant properties using organic solvents or single-step MAE/UAE. Vaquero et al. (2020) and Biswas et al. (2023) found MUAE reduces extraction time compared to UAE alone, while Frecentese et al. (2023) and Prasetyaningrum et al. (2022) showed accelerated rates with minimized energy consumption. However, water-based extraction yields remain low (Bakht et al., 2019; Mungwari et al., 2025), and no comprehensive study has systematically linked optimized sequential water-based MUAE (4 min microwave + 45 min ultrasound) to both enhanced phenolic recovery and SPF performance with FTIR structural validation (Shen et al., 2023).

Therefore, this study evaluates three water-based extraction techniques—boiling (BL), ultrasonic-assisted extraction (UAE), and microwave–ultrasonic-assisted extraction (MUAE)—on yield, total phenolic content (TPC), antioxidant activity, and SPF of black tea extracts. FTIR spectroscopy characterizes functional group variations to elucidate extraction efficiency mechanisms. By establishing explicit correlations between green extraction optimization, phenolic composition, and photoprotective performance, this work fills critical gaps and supports sustainable natural sunscreen ingredient development.

MATERIALS AND METHODS

Materials

The black tea used in this study was obtained from PTPN XII Wonosari Tea Plantation, Malang City, Indonesia. It consisted of crushed black tea leaves, typically used for tea bags, and was marketed under the Rolas Tea brand. All extractions were performed in triplicate to ensure reproducibility.

The chemicals used included distilled water, ethanol, ascorbic acid, gallic acid, Folin–Ciocalteu reagent, Na_2CO_3 , FeCl_3 , and anhydrous acetic acid, all purchased from MERCK, Germany. Additional reagents such as DPPH, magnesium powder, HCl, Mayer’s reagent, Dragendorff’s reagent, chloroform, and sulfuric acid were obtained from Sigma Aldrich (Germany).

Extraction technologies and procedures

Three extraction methods were applied: boiling (BL), ultrasonic-assisted extraction (UAE), and microwave–ultrasonic-assisted extraction (MUAE). Each method used water as a solvent to support environmentally friendly and safe extraction conditions.

Ultrasonic extraction was first optimized by varying extraction durations to determine the condition that produced the highest antioxidant activity and total phenolic content (TPC). The optimized duration was then used for subsequent microwave–ultrasonic-assisted extraction (MUAE). Both advanced methods were compared with conventional boiling extraction to evaluate extraction efficiency and compound preservation.

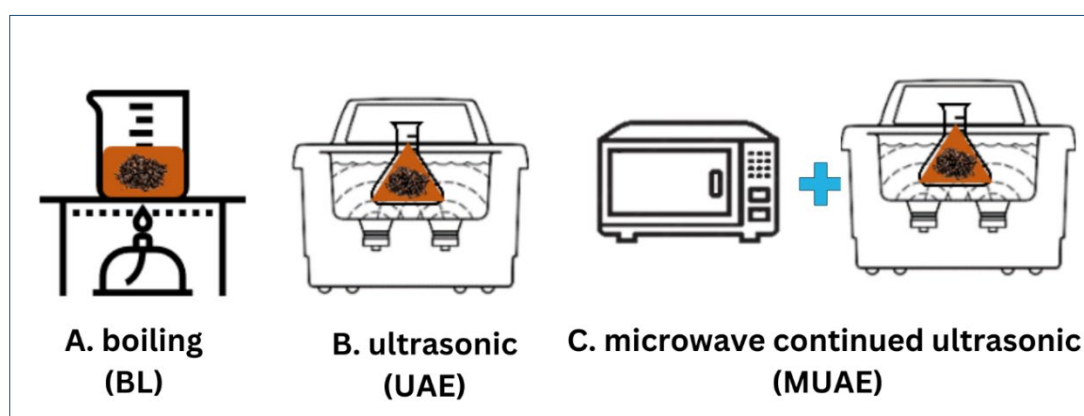


Figure 1. (A) Boiling extraction (BL); (B) Ultrasonic-assisted extraction (UAE); (C) Microwave–ultrasonic-assisted extraction (MUAE).

Boiling extraction (BL)

In the boiling method, 5 g of black tea were added to 100 mL of distilled water in a 250 mL glass beaker. The mixture was boiled at 100°C on a hot plate for 30 minutes, ensuring constant heating and occasional stirring to maintain uniform extraction. After heating, the extract was filtered through Whatman No. 1 filter paper to remove solid residues. The filtrate was freeze-dried to obtain a brownish powdered crude extract.

Ultrasonic-assisted extraction (UAE)

UAE was conducted using a water bath ultrasonic cleaner (Branson 3510, 42 kHz, 95 W). Ten grams of black tea were added to 80 mL of distilled water, maintaining a sample-to-solvent ratio of 1:8 (w/v). The mixture was sonicated at 40°C for 30, 45, and 60 minutes to evaluate the effect of time variation (designated as UAE30, UAE45, and UAE60). After sonication, the mixture was filtered and freeze-dried to obtain the crude extracts. The temperature during extraction was monitored continuously to prevent overheating, which can degrade polyphenols.

Microwave-ultrasonic-assisted extraction (MUAE)

This method evaluated the combined action effect of microwave pre-treatment followed by ultrasonic extraction. Ten grams of black tea were mixed with 80 mL of distilled water and subjected to microwave irradiation (Samsung ME711K) at 450 W for 2, 4, or 8 minutes (M2UAE45, M4UAE45, and M8UAE45), following the procedure described by Zhang et al. (2023). Immediately after microwave treatment, the mixtures were transferred to an ultrasonic water bath (42 kHz, 40°C) and sonicated for the optimized duration of 45 minutes, as determined in the UAE step. The resulting extracts were then filtered and freeze-dried. All samples were stored in airtight containers at 4°C until further analysis.

Characterization of extracts

All extracts were characterized for yield, antioxidant capacity, total phenolic content (TPC), qualitative phytochemical composition, and sunscreen efficacy (SPF).

Yield calculation

The extraction yield was calculated based on the weight of the freeze-dried crude extract using Equation 1 (Hu et al., 2019):

$$\text{Yield (\%)} = \frac{A}{W} \times 100 \% \quad (1)$$

Where A is the mass of extract obtained, and W is the initial mass of black tea sample

Antioxidant activity analysis

The antioxidant activity of the extracts was determined using the DPPH radical scavenging assay, as modified from El-Fadl et al. (2020). One milligram of extract was dissolved in 10 mL of water, and serial dilutions (2, 4, 6, 8, and 10 ppm) were prepared. Each 3 mL of sample solution was mixed with 1 mL of 0.2 mM DPPH solution, vortexed, and incubated at 37°C for 30 minutes. Absorbance was read at 520 nm using a UV-Vis spectrophotometer (Varian Cary 50). Ascorbic acid served as the positive control. The percentage of inhibition was calculated using Equation 2 (Jittasai et al., 2024):

$$\text{Inhibition (\%)} = \frac{(A-B)}{A} \times 100 \% \quad (2)$$

Where A is the absorbance of the control (DPPH only) and B is the absorbance with the sample. IC₅₀ values (extract concentration required to inhibit 50% of DPPH radicals) were determined to compare antioxidant potency among extraction methods

Total phenolic content (TPC) analysis

TPC was determined using the Folin–Ciocalteu colorimetric method, slightly modified from Opris et al. (2021). One milligram of extract was dissolved in 10 mL of water; then, 1 mL of the solution was mixed with 2.5 mL of 10% Folin–Ciocalteu reagent and 2 mL of 10% Na₂CO₃ solution. After 30 minutes of incubation, absorbance was measured at 765 nm using a UV–Vis spectrophotometer. A gallic acid calibration curve was used to quantify total phenolics, expressed as mg GAE g⁻¹ extract, according to Equation 3:

$$\text{Total Phenol Content (mg GAE/g)} = \frac{(C \times V)}{m} \times 100 \% \quad (3)$$

Where C = concentration from calibration curve (mg mL⁻¹), V = volume of sample (mL), and m = mass of extract (g). All determinations were performed in triplicate.

Phytochemical qualitative screening

The crude extracts were screened for flavonoids, alkaloids, saponins, steroids, terpenoids, and tannins using standard qualitative methods based on color changes or foam formation upon reaction with specific reagents (Alexandra et al., 2020).

Sun protection factor (SPF) analysis

SPF was evaluated *in vitro* using a modified Mansur method (Opris et al., 2021). A 0.05% extract solution was prepared in water, and absorbance was recorded from 290–320 nm at 5 nm intervals using a UV–Vis spectrophotometer. SPF was calculated using Equation 4:

$$SPF = CF \times \Sigma (EE(\lambda) \times I(\lambda) \times Abs(\lambda)) \quad (4)$$

Where *EE* = erythemal efficiency, *I* = solar intensity, *Abs* = absorbance, and *CF* = correction factor (10).

FTIR spectroscopy

Each extract was finely ground with KBr using an agate mortar and analyzed using a Fourier-transform infrared spectrophotometer (Varian Scimitar 1000) over the wavenumber range of 4,000–500 cm⁻¹ (Aboulwafa et al., 2018).

Statistical analysis

All experimental data were obtained from triplicate measurements and expressed as mean ± standard deviation (SD). Statistical analyses were performed using IBM SPSS Statistics software (version 26.0; IBM Corp., Armonk, NY, USA). One-way analysis of variance (ANOVA) was employed to evaluate significant differences among extraction methods for yield, total phenolic content (TPC), antioxidant activity (IC₅₀), and sun protection factor (SPF) values. When ANOVA indicated significance, Tukey's post hoc test was applied for multiple comparisons. Differences were considered statistically significant at *P* < 0.05 (Bakht et al., 2019).

RESULTS

Extraction optimization

Black tea (*Camellia sinensis*) extracts were obtained using boiling (BL), ultrasonic-assisted extraction (UAE), and microwave-ultrasonic-assisted extraction (MUAE). All extractions employed water as the solvent to ensure environmentally friendly and safe processing conditions. The freeze-dried extracts appeared as fine brownish powders, with darker coloration observed in samples subjected to combined microwave and ultrasonic treatment, indicating enhanced extraction efficiency.

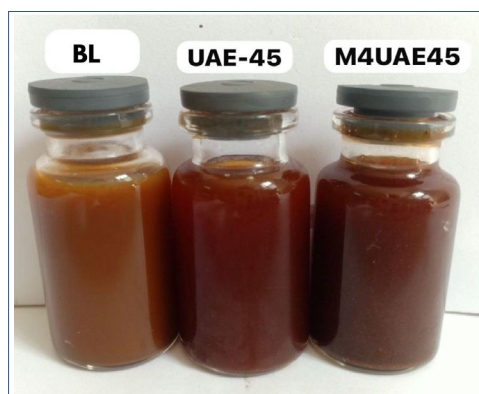


Figure 2. Filtrates obtained from different extraction methods: (A) boiling for 30 minutes (BL), (B) ultrasonic-assisted extraction for 45 minutes (UAE45), and (C) microwave-assisted extraction for 4 minutes followed by ultrasonic-assisted extraction for 45 minutes (M4UAE45).

Comparison of different extraction methods

The extraction methods were compared based on yield, total phenolic content, antioxidant capacity, qualitative phytochemical composition, and SPF performance.

Yields

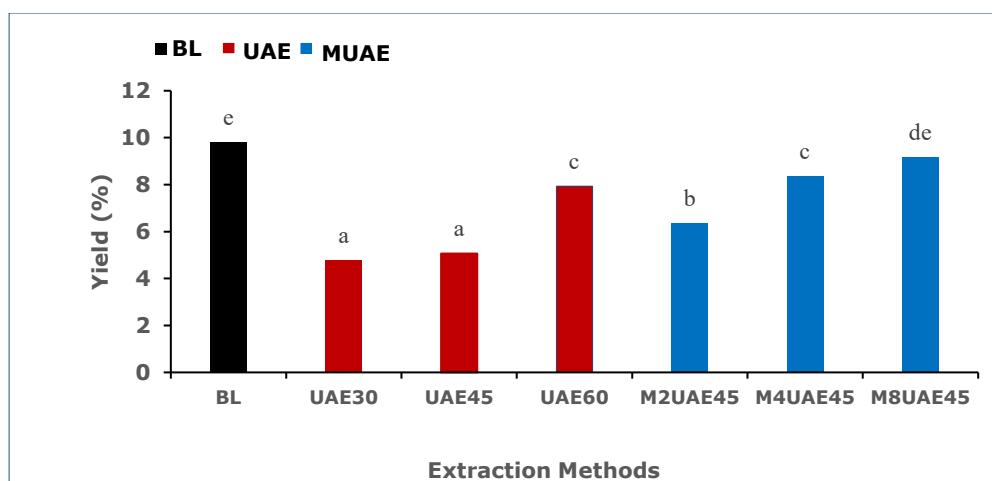


Figure 3. Average yield values of black tea leaf extracts obtained through different extraction methods: BL, UAE30, UAE45, UAE60, M2UAE45, M4UAE45, and M8UAE45. Different letters (a–e) indicate significant differences ($P < 0.05$, one-way ANOVA followed by Tukey's test).

The extraction yields obtained from the different methods are presented in Figure 3. Conventional boiling produced the highest crude extract yield ($9.82 \pm 0.44\%$), followed by MUAE treatments with prolonged microwave exposure, particularly M8UAE45 ($9.18 \pm 0.31\%$) and M4UAE45 ($8.36 \pm 0.29\%$). In contrast, single-step UAE treatments resulted in lower yields, with UAE30 and UAE45 yielding $4.77 \pm 0.55\%$ and $5.06 \pm 0.77\%$, respectively.

Although boiling generated a higher overall yield, this did not correspond to improved recovery of phenolic compounds or antioxidant performance. In comparison, MUAE treatments demonstrated enhanced extraction efficiency relative to UAE, suggesting that microwave pre-treatment facilitated solvent penetration and mass transfer during subsequent ultrasonic extraction.

Total phenolic content (TPC)

The total phenolic content of the extracts is shown in Figure 5. Among the UAE conditions, UAE45 exhibited greater phenolic recovery (316.60 ± 2.92 mg GAE/g) than UAE30 and UAE60, indicating that an intermediate sonication time favored phenolic extraction.

A substantial increase in phenolic recovery was observed under MUAE conditions. The M4UAE45 treatment yielded the highest phenolic concentration (457.64 ± 2.31 mg GAE/g), followed by M2UAE45 (442.67 ± 1.39 mg GAE/g). However, extending microwave exposure to 8 minutes resulted in a marked decrease in TPC, suggesting partial degradation or oxidation of heat-sensitive phenolic compounds. The TPC obtained from boiling extraction (262.17 ± 4.16 mg GAE/g) was comparable to that of M8UAE45 but considerably lower than that achieved under optimized MUAE conditions.

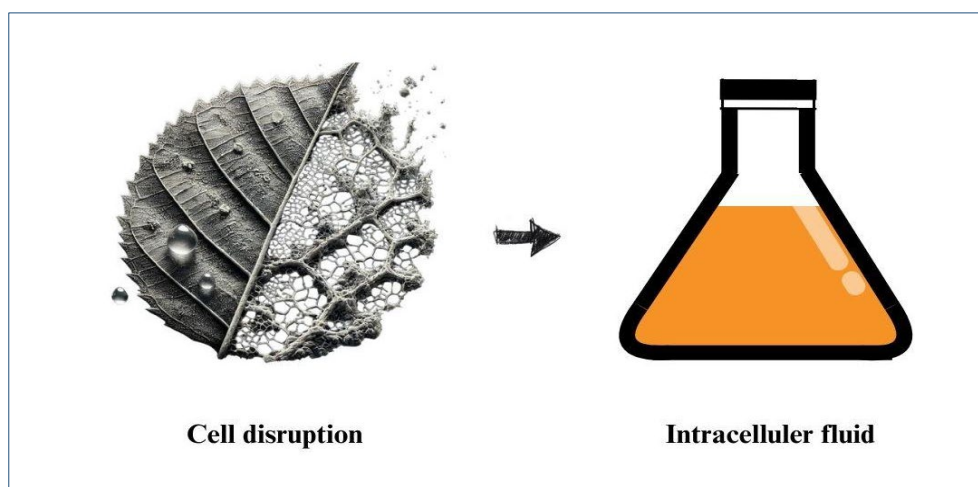


Figure 4. Illustrates the mechanism of MUAE, where microwave pre-treatment facilitates the disruption of cell structures, enhancing subsequent ultrasonic cavitation and solvent diffusion.

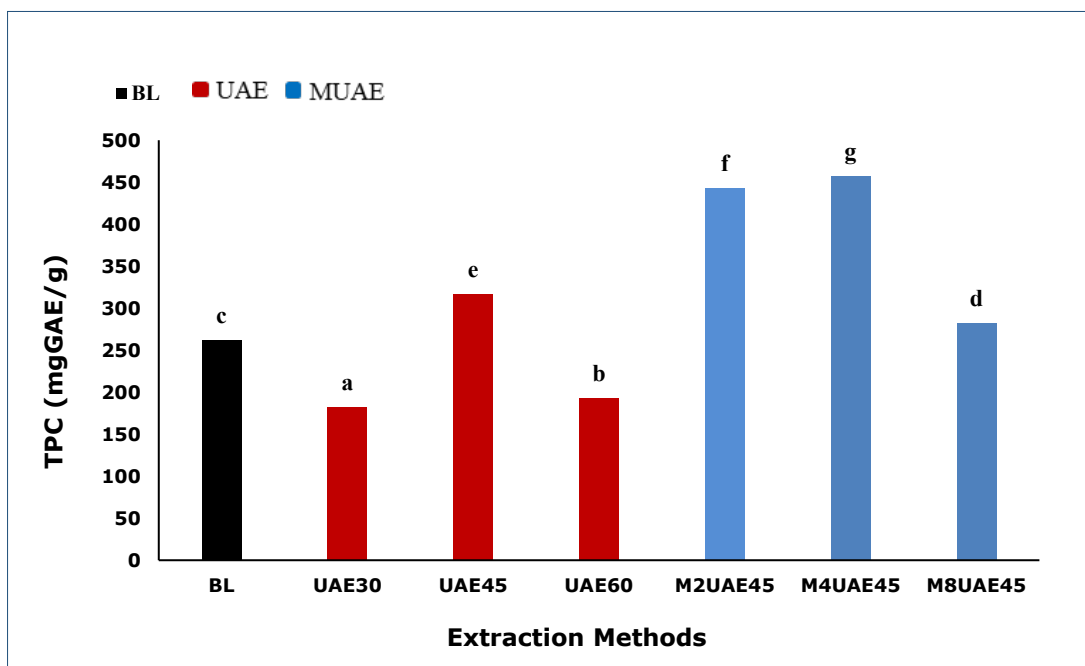


Figure 5. Total phenolic content (mg GAE/g extract) of black tea leaf extracts obtained through different extraction methods: BL, UAE30, UAE45, UAE60, M2UAE45, M4UAE45, and M8UAE45. Different letters (a–g) indicate significant differences ($P < 0.05$, one-way ANOVA followed by Tukey's test).

Antioxidant activity (DPPH Assay)

The DPPH radical scavenging activity of all extracts increased in a concentration-dependent manner (Figure 6). At concentrations below 2 ppm, only the boiling extract showed measurable inhibition, whereas UAE45 and MUAE-derived extracts exhibited stronger scavenging effects at higher concentrations.

The IC_{50} values presented in Figure 7 further clarify differences in antioxidant performance among the extracts. The M4UAE45 extract exhibited the lowest IC_{50} value (2.97 ± 0.41 ppm), indicating superior radical scavenging efficiency, followed by UAE45 (3.20 ± 0.19 ppm) and boiling extraction (4.03 ± 0.32 ppm). In contrast, prolonged microwave treatment resulted in reduced antioxidant activity, consistent with the observed decline in phenolic content. These results indicate that optimized MUAE conditions promote efficient antioxidant extraction while minimizing thermal degradation.

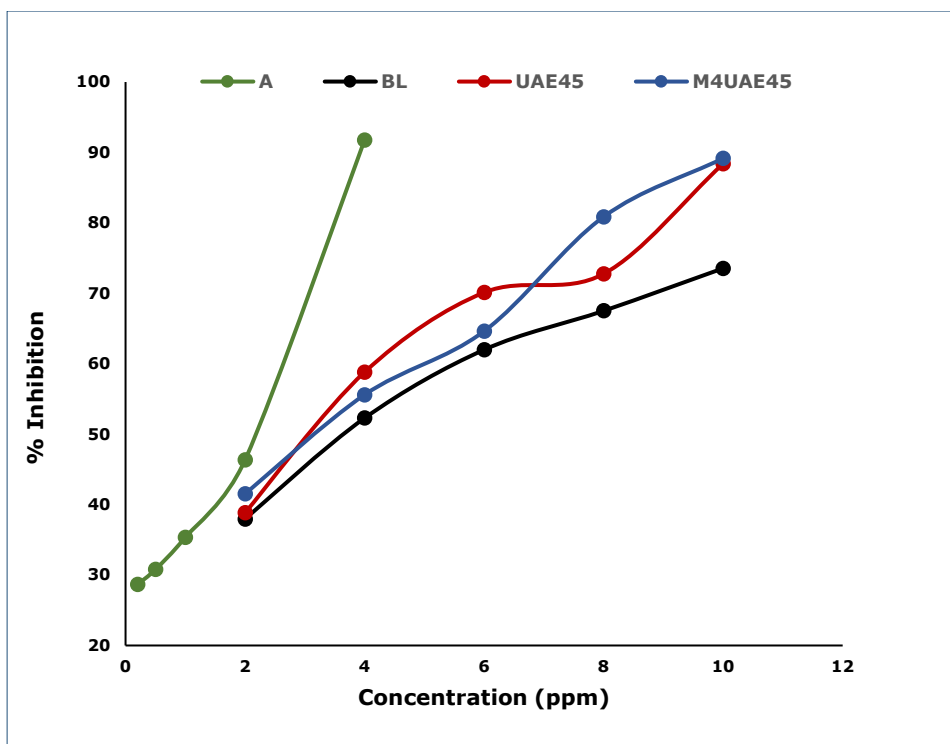


Figure 6. DPPH radical scavenging inhibition (%) of black tea leaf extracts from A = ascorbic acid, BL, UAE45, and M4UAE45 at different concentrations (2–10 ppm).

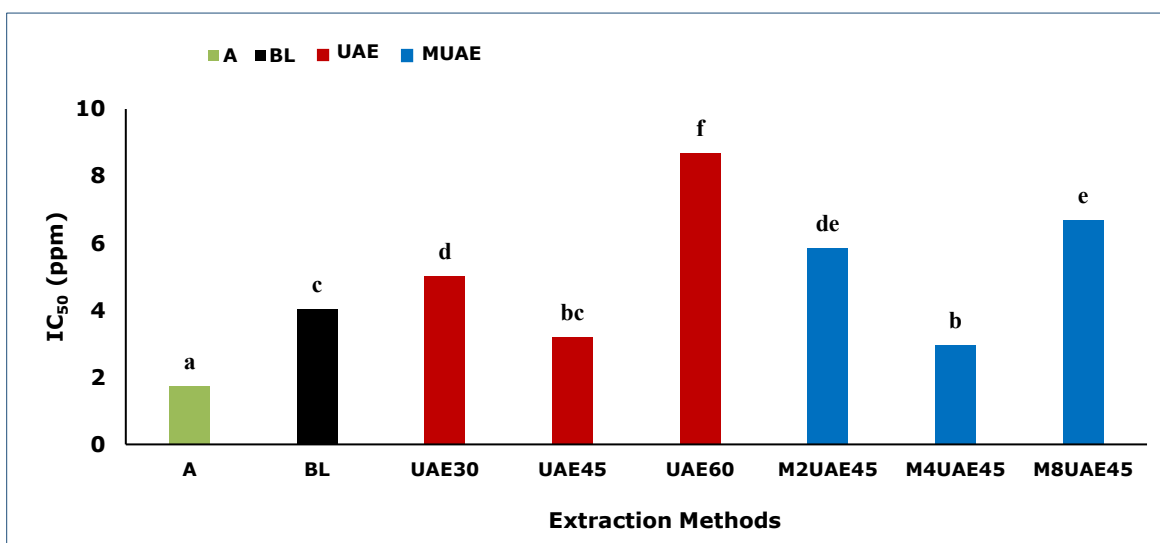


Figure 7. IC₅₀ values of black tea leaf extracts (A = ascorbic acid, BL, UAE30, UAE60, M2UAE45, M4UAE45, M8UAE45). Different letters (a–f) indicate significant differences ($P < 0.05$, one-way ANOVA followed by Tukey's test).

Qualitative phytochemical analysis

Table 1. Phytochemical screening results of black tea extracts obtained using BL, UAE45, and M4UAE45.

Phytochemical Test	BL	UAE45	M4UAE45
Flavonoids	+	+	+
Alkaloids (Mayer)	–	–	–
Alkaloids (Dragendorff)	+	–	+
Steroids	–	+	+
Triterpenoids	+	–	+
Tannins	+	+	+
Saponins	+	+	+

Note: (+ indicates presence, – indicates absence)

The qualitative phytochemical profiles of selected extracts are summarized in Table 1. All extracts tested positive for flavonoids, tannins, and saponins. Notably, the MUAE-derived extract (M4UAE45) exhibited a wider range of phytochemical responses in qualitative assays, including steroids, triterpenoids, and Dragendorff-positive alkaloids, which were absent or less pronounced in UAE45. The expanded phytochemical profile observed under MUAE conditions suggests that the combined microwave and ultrasonic treatment enhanced cell matrix disruption and facilitated the solubilization of less-polar bioactive compounds.

Sun protection factor (SPF) activity

The *in vitro* SPF values of the extracts are presented in Figure 8. MUAE-derived extracts demonstrated greater UV-protective capacity compared with boiling and single-step UAE. The M4UAE45 treatment achieved an SPF value of 45.40 ± 0.57 , whereas UAE45 and M2UAE45 yielded SPF values of 41.33 ± 0.05 and 41.96 ± 0.50 , respectively.

Lower SPF values were observed in extracts subjected to prolonged thermal exposure, particularly M8UAE45 and boiling extraction. These findings indicate that moderate microwave pre-treatment combined with ultrasonic extraction enhances the recovery of UV-absorbing compounds, while excessive heating may compromise photoprotective efficacy.

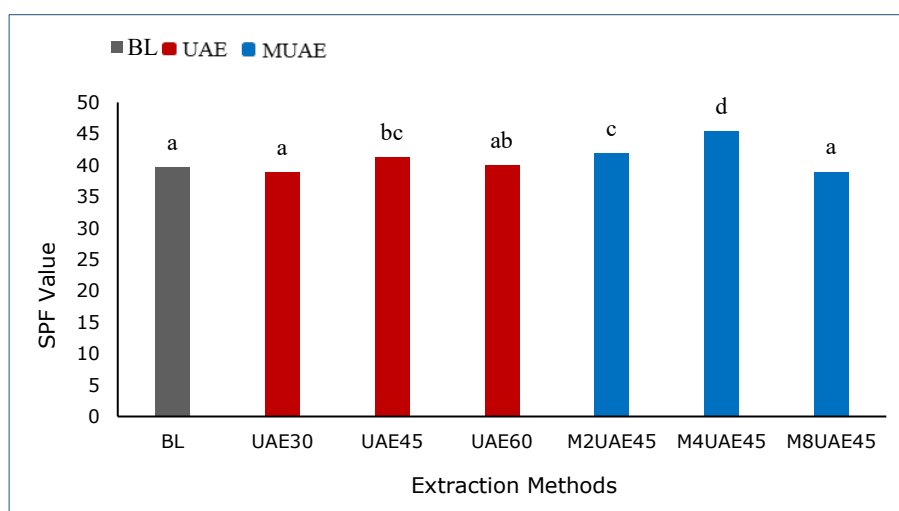


Figure 8. SPF values of black tea leaf extracts obtained using BL, UAE, and MUAE methods. Different letters (a–f) indicate significant differences ($P < 0.05$, one-way ANOVA followed by Tukey's test).

FTIR spectral analysis

The FTIR spectra of the extracts are shown in Figure 9. All samples exhibited broad absorption bands around $3,400\text{ cm}^{-1}$ corresponding to O–H stretching vibrations characteristic of phenolic compounds. The intensity of these bands was higher in UAE and MUAE extracts compared with boiling extraction, supporting enhanced phenolic recovery.

Distinct absorption peaks observed at $1,632\text{--}1,636\text{ cm}^{-1}$ were attributed to C=C stretching vibrations in aromatic rings, supporting the presence of flavonoid-related functional groups. Additional bands at $1,451\text{--}1,455\text{ cm}^{-1}$ and $1,381\text{--}1,384\text{ cm}^{-1}$ corresponded to C–H bending and C–O stretching vibrations, respectively. MUAE spectra displayed sharper and more intense absorption bands, suggesting a stronger representation of phenolic functional groups relative to other extracts, which is consistent with the higher total phenolic content and bioactivity determined by complementary quantitative analyses.

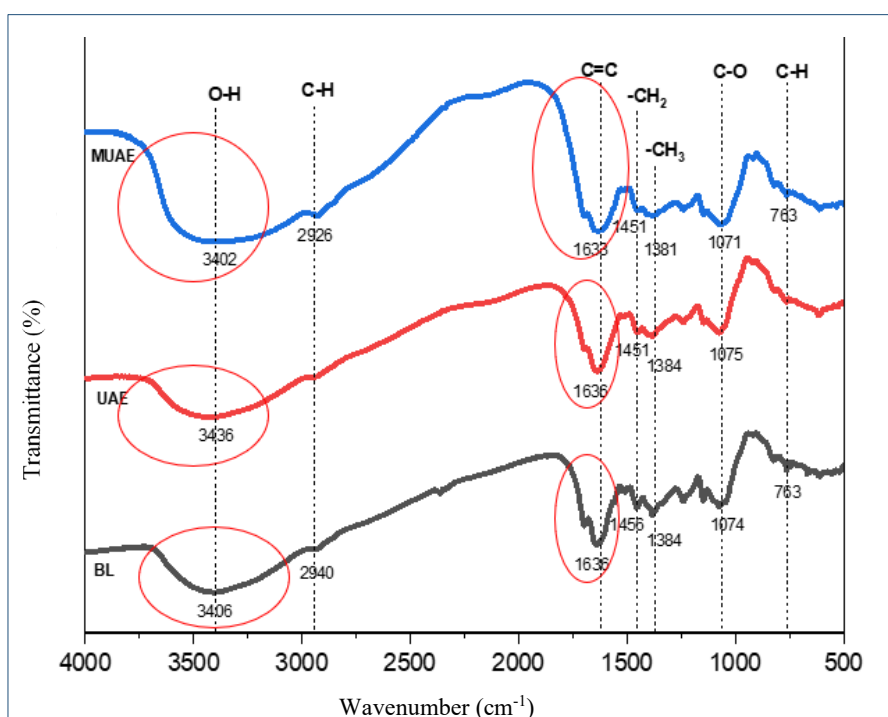


Figure 9. FTIR spectra of extracts obtained from BL, UAE, and MUAE methods.

Summary of extraction efficiency

Table 2. Summary comparison of extraction methods based on total phenolic content (TPC), antioxidant activity (IC_{50}), and sun protection factor (SPF).

Extraction method	Extraction conditions	TPC (mg GAE/g)	IC_{50} (ppm)
BL	30 min at 100°C	262.17 ± 4.16	4.03 ± 0.32
UAE45	45 min, 40°C , 42 kHz	316.60 ± 2.92	3.20 ± 0.19
M4UAE45	Microwave 4 min (450 W) + Ultrasound 45 min (40°C)	457.64 ± 2.31	2.97 ± 0.41

Among the evaluated techniques, M4UAE45 exhibited superior overall performance, as reflected by enhanced phenolic recovery, improved antioxidant efficacy, and elevated SPF values. These findings confirm that the sequential combination of microwave pre-treatment and ultrasonic cavitation improves extraction efficiency relative to single-step or conventional methods.

Overall, the results indicate that M4UAE45 represents the most effective extraction protocol under the investigated conditions. The observed improvements can be attributed to the synergistic interaction between microwave-induced cell disruption and ultrasonic-enhanced mass transfer, which facilitates efficient release of bioactive compounds while limiting thermal degradation. Collectively, these results support the potential of M4UAE as a green, efficient, and scalable approach for producing bioactive-rich black tea extracts with photoprotective properties.

DISCUSSION

This study demonstrated that extraction techniques profoundly affect phenolic recovery, antioxidant capacity, and photoprotective efficacy of *Camellia sinensis* black tea extracts. The microwave-ultrasonic-assisted extraction (M4UAE45) outperformed others across all metrics, delivering the highest total phenolic content (457.64 ± 2.31 mg GAE/g), strongest antioxidant activity (IC_{50} 2.97 ± 0.41 ppm), and greatest SPF value (45.4 ± 0.59).

These results align with literature showing extended extraction times improve yields in UAE and M4UAE processes (Lasunon and Sengkhampan, 2022). Prolonged solvent contact enhances mass transfer, releasing bioactives from plant matrices. However, excess heat degrades thermolabile phenolics, underscoring the need for optimized temperature and duration (Gil-Martín et al., 2022).

M4UAE45 achieved comparable yields to boiling but superior bioactivity due to controlled thermal exposure preserving sensitive compounds. Microwave irradiation induces rapid volumetric heating within the plant matrix, generating internal pressure gradients that weaken cell wall integrity and enhance solvent accessibility. Subsequent ultrasonic treatment promotes cavitation-driven microstreaming and shear forces, facilitating solvent penetration and accelerating the release of intracellular phenolic constituents (Khadhraoui et al., 2018; Wang et al., 2018). The pronounced increase in phenolic recovery observed under M4UAE conditions is therefore attributed to the synergistic interaction between microwave-induced matrix loosening and ultrasound-enhanced mass transfer, rather than prolonged thermal exposure. Similar effects of microwave power in promoting membrane permeability and improving extraction efficiency have been widely reported in combined extraction systems (Kusuma et al., 2019; Marsiglia et al., 2023).

TPC from M4UAE45 substantially exceeded Indonesian black tea benchmarks. Conventional 80°C extraction yielded only 1 mg GAE/g (Nugraheni et al., 2021), while concentrated extracts reached 224.69-254.62 mg GAE/g (Rahman et al., 2021). Microwaves enable rapid matrix breakdown while ultrasound cavitation improves solvent diffusion, yielding higher-quality extracts faster than single methods. Comparable hybrid benefits appear in Vaquero et al. (2020), Frecentese et al. (2023), and Laina et al. (2024).

Boiling produced lower TPC due to thermal oxidation of polyphenols and catechins. UAE at 40°C preserved stability but limited mass transfer. M4UAE45 overcame both via rapid energy transfer and mechanical disruption without overheating (Bakht et al., 2019; Ahmad et al., 2023). Antioxidant potency correlated directly with phenolic levels, confirming polyphenols as primary radical scavengers (Bakht et al., 2019; Oprüş et al., 2021).

SPF followed the same pattern, with M4UAE45 exhibiting peak UV protection. Phenolics serve dual roles as antioxidants and natural UV absorbers, reducing erythema via ROS scavenging. This multifunctional profile supports black tea extracts

in sunscreen formulations (Sharma et al., 2024; Sieniawska et al., 2024). Qualitative screening revealed broader compound recovery in M4UAE45, including steroids and triterpenoids absent in UAE45.

FTIR analysis revealed more pronounced O–H ($3,400\text{ cm}^{-1}$), aromatic C=C ($1,632\text{--}1,636\text{ cm}^{-1}$), and C–O stretching bands in MUAE extracts, supporting the presence of phenolic-associated functional groups. These spectral features are consistent with the enhanced phenolic recovery measured by TPC analysis. Water-only extraction with low-energy technologies embodies green chemistry principles, minimizing waste and hazards versus organic solvents. Future work should refine microwave power, ultrasound intensity, and solid-liquid ratios, incorporating kinetic modeling and life-cycle assessments to validate industrial viability.

CONCLUSION

Extraction techniques critically determine phenolic recovery, antioxidant capacity, and photoprotective potential of *Camellia sinensis* black tea extracts. Among tested methods, M4UAE45 yielded the optimal bioactive profile, achieving peak performance across all parameters. Notably, the implementation of a water-only microwave–ultrasonic assisted extraction protocol with reduced processing time represents a novel, green, and sustainable approach, demonstrating significant practical implications for the development of high-quality bioactive extracts suitable for cosmetic and pharmaceutical applications. These findings highlight the potential of the proposed extraction strategy to support environmentally friendly production and to facilitate future translation into functional sunscreen and cosmeceutical formulations. Future research should focus on further optimization of process parameters, as well as comprehensive stability and safety evaluations of black tea-based sunscreen formulations to enable commercial-scale application. Extraction techniques critically determine phenolic recovery, antioxidant capacity, and photoprotective potential of *Camellia sinensis* black tea extracts. Among the tested methods, M4UAE45 yielded the optimal bioactive profile, achieving the highest performance across all evaluated parameters. This outcome results from the synergistic effects of microwave-induced heating and ultrasonic cavitation, which promote efficient matrix disruption and rapid diffusion of bioactive compounds while minimizing thermal degradation.

Future research should focus on further optimization of process parameters, as well as comprehensive stability and safety evaluations of black tea-based sunscreen formulations to enable commercial-scale application.

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AUTHOR CONTRIBUTIONS

Eny Yulianti: Conceptualization (Lead), Methodology (Equal), Investigation (Equal), Data Curation (Equal), Formal Analysis (Equal), Visualization (Equal), Writing – Original Draft (Lead), Writing – Review and Editing (Equal); **Ivvani Aulia Putri and Siti Amanatus Sholikah:** Investigation (Equal), Data Curation (Equal), Formal Analysis (Supporting), Visualization (Supporting), Writing – Original Draft (Supporting); **Warsito Warsito, Akhmad Sabarudin, and Bayyinatul**

Muchtaromah: Conceptualization (Supporting), Methodology (Equal), Supervision (Lead/Equal), Validation (Equal), Writing – Review and Editing (Equal). All authors have read and approved the final version of the manuscript.

CONFLICT OF INTEREST

The authors declare that they hold no competing interests.

REFERENCES

- Aboulwafa, M.M., Youssef, F.S., Gad, H.A., Sarker, S.D., Nahar, L., Al-Azizi, M.M., and Ashour, M.L. 2018. Authentication and discrimination of green tea samples using UV-Vis, FTIR and HPLC techniques coupled with chemometrics analysis. *Journal of Pharmaceutical and Biomedical Analysis*. 164: 653-658.
<https://doi.org/10.1016/j.jpba.2018.11.036>
- Ahmad, R., Aldholmi, M., Alqathama, A., Althomali, E., Aljishi, F., Mostafa, A., Alqarni, A.M., and Shaaban, H. 2023. The effect of natural antioxidants, pH, and green solvents upon catechins stability during ultrasonic extraction from green tea leaves (*Camellia sinensis*). *Ultrasonics Sonochemistry*. 94: 106337.
<https://doi.org/10.1016/j.ultsonch.2023.106337>
- Alexandra, S., Jamora, N., Smale, M., and Ghanem, M.E. 2020. The tale of taro leaf blight: A global effort to safeguard the genetic diversity of taro in the Pacific. *Food Security*. 12(5): 1005-1016.
<https://doi.org/10.1007/s12571-020-01039-6>
- Bakht, M.A., Geesi, M.H., Riadi, Y., Imran, M., Ali, I.M.D., Ahsan, M.J., and Ajmal, N. 2019. Ultrasound-assisted extraction of some branded tea: Optimization based on polyphenol content, antioxidant potential, and thermodynamic study. *Saudi Journal of Biological Sciences*. 26(5): 1043-1052.
<https://doi.org/10.1016/j.sjbs.2018.07.013>
- Bhattacharya, S. and Sherje, A.P. 2020. Development of resveratrol and green tea sunscreen formulation for combined photoprotective and antioxidant properties. *Journal of Drug Delivery Science and Technology*. 60: 102000.
<https://doi.org/10.1016/j.jddst.2020.102000>
- Biswas, R., Sarkar, A., Alam, M., Roy, M., and Hasan, M.M. 2023. Microwave and ultrasound-assisted extraction of bioactive compounds from papaya: A sustainable green process. *Ultrasonics Sonochemistry*. 101: 106677.
<https://doi.org/10.1016/j.ultsonch.2023.106677>
- Chen, Y. 2024. Formula preparation of sunscreen (experimental phenomenon and property description) and evaluation of its efficacy. *Academic Journal of Materials and Chemistry*. 5(3): 43-50.
<https://doi.org/10.25236/AJMC.2024.050307>
- El-Fadl, S.R.A., Osman, A., Al-Zohairy, A., Dahab, A., and El Kheir, A.Z. 2020. Assessment of total phenolic, flavonoid content, antioxidant potential and HPLC profile of three *Moringa* species leaf extracts. *Scientific Journal of Flowers and Ornamental Plants*. 7(1): 53-70.
<https://doi.org/10.21608/sjfop.2020.91397>
- Fakhri, M., Jamshidbeigi, T., Dehkordi, A.H., Abdan, M., Mirfendereski, S., and Sarokhani, D. 2024. Relationship between green or black tea consumption and cerebral stroke: A systematic review and meta-analysis. *Journal of Herbmед Pharmacology*. 13(1): 1-9.
<https://doi.org/10.34172/jhp.2024.44838>

- Frecentese, F., Sodano, F., Corvino, A., Schiano, M.E., Magli, E., Albrizio, S., Sparaco, R., Andreatti, G., Nieddu, M., and Rimoli, M.G. 2023. The application of microwaves, ultrasounds and their combination in the synthesis of nitrogen-containing bicyclic heterocycles. *International Journal of Molecular Sciences*. 24: 10722.
<https://doi.org/10.3390/ijms241310722>
- Gil-Martín, E., Hernández, F.T., Romero, A., Cianciosi, D., Giampieri, F., and Battino, M. 2022. Influence of the extraction method on the recovery of bioactive phenolic compounds from food industry by-products. *Food Chemistry*. 378: 131918.
<https://doi.org/10.1016/j.foodchem.2021.131918>
- Hu, B., Li, C., Qin, W., Zhang, Z., Liu, Y., Zhang, Q., Liu, A., Jia, R., Yin, Z., Han, X., et al. 2019. A method for extracting oil from tea (*Camellia sinensis*) seed by microwave in combination with ultrasonic and evaluation of its quality. *Industrial Crops and Products*. 131: 234-242.
<https://doi.org/10.1016/j.indcrop.2019.01.068>
- Jittasai, P., Kanjanakawinkul, W., Yawootti, A., and Chaiyana, W. 2024. Phytochemical compositions and cosmeceutical activities of *Rosa damascena* Mill. leaf extracts from environmentally friendly extraction technique. *Natural and Life Sciences Communications*. 23(1): e2024008.
<https://doi.org/10.12982/NLSC.2024.008>
- Khadhraoui, B., Turk, M., Tixier, F.A.S., Petitcolas, E., Robinet, P., Imbert, R., Maataoui, M.E., and Chemat, F. 2018. Histochemistry and scanning electron microscopy for studying spatial and temporal extraction of metabolites induced by ultrasound towards chain detexturation mechanism. *Ultrasonics Sonochemistry*. 42: 482-492.
<https://doi.org/10.1016/j.ultsonch.2017.11.029>
- Kusuma, H.S., Putri, D.K.Y., Triesty, I., and Mahfud, M. 2019. Comparison of microwave hydrodistillation and solvent-free microwave extraction for extraction of agarwood oil. *Chiang Mai Journal of Science*. 46(4): 741-755.
- Laina, K.T., Drosou, C., Stergiopoulos, C., Eleni, P.M., and Krokida, M. 2024. Optimization of combined ultrasound and microwave assisted extraction for enhanced bioactive compounds recovery from four medicinal plants. *Molecules*. 29(23): 5773.
<https://doi.org/10.3390/molecules29235773>
- Lasunon, P. and Sengkhampan, N. 2022. Effect of ultrasound-assisted, microwave-assisted, and ultrasound-microwave-assisted extraction on pectin extraction from industrial tomato waste. *Molecules*. 27: 1157.
<https://doi.org/10.3390/molecules27041157>
- Marsiglia, L.W.I.M., Oliveira, L.S.C., Almeida, L.J.R., Santos, N.C., Neto, S.J.M., Santiago, Â.M., de Melo, B.C.A., and Silva, H.F.L. 2023. Thermal stability of total phenolic compounds and antioxidant activities of jaboticaba peel: Effect of solvents and extraction methods. *Journal of the Indian Chemical Society*. 100(5): 100995.
<https://doi.org/10.1016/j.jics.2023.100995>
- Muflihah, U., Mutolib, A., and Nuraini, C. 2023. Analysis of Indonesian tea export competitiveness on the international market. *East African Journal of Multidisciplinary Research*. 2(10): 4209-4216.
<https://doi.org/10.55927/eajmr.v2i10.6354>
- Mungwari, C.P., King'onde, C.K., Sigauke, P., and Obadele, B.A. 2025. Conventional and modern techniques for bioactive compounds recovery from plants: Review. *Scientific African*. 27: e02509.
<https://doi.org/10.1016/j.sciaf.2024.e02509>
- Nugraheni, Z.V., Laili, H.N., Gunawan, W.D., Putro, H.S., Wahyudi, A., Zetra, Y., and Burhan, R.Y.P. 2021. Effect of black tea extract (*Camellia sinensis*) on antioxidant activity of pukis. *AIP Conference Proceedings*. 2349: 020078.
<https://doi.org/10.1063/5.0051536>

- Oprış, O., Soran, M.-L., Lung, I., Stegarescu, A., Guțoiu, S., Podea, R., and Podea, P. 2021. Optimization of extraction conditions of polyphenols, antioxidant capacity, and sun protection factor from *Prunus spinosa* fruits. *Journal of the Iranian Chemical Society*. 18: 2625-2636.
<https://doi.org/10.1007/s13738-021-02217-9>
- Prasetyaningrum, A., Jos, B., Ratnawati, R., Rokhati, N., Riyanto, T., and Prinanda, G.R. 2022. Sequential microwave-ultrasound assisted extraction of flavonoid from *Moringa oleifera*. *Indonesian Journal of Chemistry*. 22: 303-316.
<https://doi.org/10.22146/ijc.65252>
- Rahman, M., Jahan, I.A., Ahmed, S., Ahmed, K.S., Roy, M., Zzaman, W., and Ahmad, I. 2021. Bioactive compounds and antioxidant activity of black and green tea available in Bangladesh. *Food Research*. 5(3): 107-111.
[https://doi.org/10.26656/fr.2017.5\(3\).491](https://doi.org/10.26656/fr.2017.5(3).491)
- Sharma, M. 2024. Understanding the impact of UV radiation on skin health: Mechanisms, risks, and photoprotection strategies. *Indian Journal of Health Care Medical and Pharmacy Practice*. 5(1): 88-95.
<https://doi.org/10.59551/IJHMP/25832069/2024.5.1.120>
- Shen, L., Pang, S., Zhong, M., Sun, Y., Qayum, A., Liu, Y., Rashid, A., Xu, B., Liang, Q., Ma, H., et al. 2023. A comprehensive review of ultrasonic assisted extraction for bioactive components. *Ultrasonics Sonochemistry*. 101: 106646.
<https://doi.org/10.1016/j.ultsonch.2023.106646>
- Sieniawska, D., Proszowska, P., Madoń, M., Kotowicz, Z., Orzeł, A., Pich-Czekierda, A., and Sieniawska, J. 2024. Ultraviolet protective clothing and sunscreen: Sun protection for healthy skin. *Journal of Education, Health and Sport*. 71: 51237.
<https://doi.org/10.12775/JEHS.2024.71.51237>
- Sopyan, I., Permata, R.D., Gozali, D., and Syah, I.S.K. 2019. Formulation of lotion from black tea extract (*Camellia sinensis* L.) as sunscreen. *International Journal of Applied Pharmaceutics*. 11(1): 205-209.
<https://doi.org/10.22159/ijap.2019v11i1.29564>
- Vaquero, G.M., Ummat, V., Tiwari, B., and Rajauria, G. 2020. Exploring ultrasound, microwave, and ultrasound-microwave assisted extraction technologies to increase the extraction of bioactive compounds from brown macroalgae. *Marine Drugs*. 18: 172.
<https://doi.org/10.3390/md18030172>
- Wang, Y., Li, R., Jiang, Z.T., Tan, J., Tang, S.H., Li, T.T., Liang, L.L., He, H.J., Liu, Y.M., Li, J.T., et al. 2018. Green and solvent-free simultaneous ultrasonic-microwave assisted extraction of essential oil from white and black peppers. *Industrial Crops and Products*. 114: 164-172.
<https://doi.org/10.1016/j.indcrop.2018.02.002>
- Zhang, Y., Lei, Y., Qi, S., Fan, M., Zheng, S., Huang, Q., and Lu, X. 2023. Ultrasonic-microwave assisted extraction for enhancing antioxidant activity of *Dictyophora indusiata* polysaccharides. *Ultrasonics Sonochemistry*. 95: 106356.
<https://doi.org/10.1016/j.ultsonch.2023.106356>
- Zheng, X.Q., Zhang, X.H., Gao, H.Q., Huang, L.Y., Ye, J.J., Ye, J.H., Lu, J.L., Ma, S.C., and Liang, Y.R. 2024. Green tea catechins and skin health. *Antioxidants*. 13: 1506.
<https://doi.org/10.3390/antiox13121506>

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