ID: 665

Effect of Roasting Methods and Microwave-Assisted Extraction on the Bioactive Compounds of Date Seeds

Hande Baltacıoğlu[1]*, Gözde Doğanay[1]

[1]Niğde Omer Halisdemir University, Faculty of Engineering, Department of Food Engineering, 51240, Niğde, Turkey.

Abstract

In this study, phenolic compounds were extracted from unroasted and roasted date seeds by microwave-assisted (300, 450, 600, and 800W) method, and their changes were investigated. TPC values ranged from 6628.89 to 9763.29 mg GAE/100 g dry weight. DPPH inhibition values ranged from 44.78% to 58.17%, with the highest antioxidant activity observed in the traditional roasting samples extracted at 800 W for 2 min. Generally, unroasted samples gave higher TPC results than traditional and microwave roasting. On the other hand, traditional roasting samples gave higher antioxidant activity results. HPLC analysis showed that rutin was the predominant phenolic compound in unroasted date seeds. Gallic acid and 3,4-dihydroxybenzoic acid became detectable after roasting and reached their highest concentrations in traditional roasting samples. PCA, as a multivariate analysis, was a successful method for the classification of the unroasted, traditional roasting, and microwave-roasting samples based on TPC and antioxidant activity.

Key Words: *Date seed, Microwave extraction, Roasting methods, TPC, Antioxidant activity*

Introduction

Date palm (*Phoenix dactylifera* L.) is an important crop widely cultivated in arid and semi-arid regions. Date palm cultivation has traditionally extended across a broad geographical region, from Morocco to Egypt in North Africa, throughout the Arabian Peninsula, into Iraq and Iran in the Middle East, and as far as Pakistan in South Asia (Flowers et al., 2019). Date seeds account for approximately 13–15% of the total fruit weight. During date processing, the seeds are commonly removed and discarded, resulting in considerable waste. While date seeds are commonly used in animal feeding, recent research has indicated that their bioactive compounds may also provide health-promoting properties (Al Harthi et al., 2015). Date seeds contain important nutritional components, including dietary fiber, minerals, vitamins, fatty acids, and amino acids. They are also considered a valuable source of bioactive compounds, such as carotenoids, polyphenols, flavonoids, and tocopherols (Zarie et al., 2023). Roasting is an important thermal treatment in the production of coffee-like beverages from date seeds. During roasting, Maillard reactions, caramelization, and pyrolysis contribute to the development of characteristic color, aroma, and flavor while also altering the physicochemical and bioactive properties of the seeds (Shi et al., 2025). Fikry et al. (2019) reported that roasting temperature and time significantly affected the total phenolic content, antioxidant activity, color, and sensory properties of date seed beverages. In their study, date seeds were roasted at 160, 180, and 200 °C for 10–30 min, and the optimum conditions were determined as approximately 200 °C for 21.5 min. Similarly, Halabi et al. (2024) found that roasting conditions ranging from 160 to 200 °C and 15 to 45 min influenced phenolic compounds and antioxidant activity. Microwave-assisted extraction (MAE) has emerged as a sustainable alternative to conventional extraction methods. It enables rapid processing, reduces solvent consumption, and improves extraction efficiency and extract quality (Melikoğlu, 2025). Microwave-assisted extraction (MAE) uses electromagnetic waves in the frequency range of 300 MHz to 300 GHz to heat the sample matrix. The applied electric field induces the movement of ionic species, promoting solvent penetration into the matrix and facilitating the release and dissolution of target compounds (Baltacıoğlu et al., 2019). In this study, unroasted and roasted date seeds were subjected to microwave-assisted extraction at 300, 450, 600, and 800 W for 2 min. The effects of roasting method and microwave extraction power on total phenolic content and DPPH radical-scavenging activity were evaluated. Individual phenolic compounds were identified and quantified by HPLC. Principal component analysis was also performed to examine the relationships between total phenolic content and antioxidant activity and to distinguish the different roasting treatments.

Materials and Methods

The date seeds were obtained from a local market and stored at 4 °C before processing.

Methods

Roasting

100 g of date seeds were weighed for each roasting process. The date seeds were heated in an oven at 180 °C for 20 minutes in the traditional roasting method. The microwave roasting process was carried out in a household microwave oven (Samsung MS23K3515AW) at a power of 300 W for 12 minutes. Unroasted seeds were used as control. Date seeds were ground with a grinder (HC100, Lavion, Türkiye) and passed from a 250 µm sieve.

Microwave-assisted extraction

For each treatment, 5 g of unroasted or roasted date seed sample was weighed. The final volume was adjusted to 100 mL with 80% methanol containing 1% HCl. Microwave-assisted extraction was performed at four different power levels (300, 450, 600, and 800 W) for 2 min. After extraction, any volume loss due to evaporation was compensated by readjusting the final volume to 100 mL. The extracts were centrifuged at 5000 rpm for 20 min and then filtered.

Total phenolic content (TPC)

The TPC of the extracts was analyzed according to the Folin–Ciocalteu method. The results were expressed as gallic acid equivalents mg GAE/100 g dry weight (Baltacıoğlu et al., 2024). Briefly, 0.1 mL of extract was mixed with 0.75 mL of 10% Folin–Ciocalteu reagent. After 5 min of incubation, 0.75 mL of sodium carbonate solution (75 g/L) was added to the mixture. After vortexing, the mixture was kept in the dark at room temperature for 60 min, and the absorbance was recorded at 725 nm.

Antioxidant activity (AA)

Antioxidant activity was determined using the DPPH radical scavenging assay. Briefly, 0.1 mL of extract was mixed with 3.9 mL of 0.1 mM DPPH solution prepared in 80% methanol. After vortexing, the mixture was incubated in the dark at room temperature for 30 min. The absorbance was then measured at 517 nm. For the control, 0.1 mL of 80% methanol was used instead of the extract. Antioxidant activity was calculated by Equation (1) as inhibition (%) Okur et al. (2019) .

$$\%\ inhibition\ = \frac{Ac - As}{Ac} \times 100 \quad (1)$$

Ac = Control of the absorbnas

As = Samples of the absorbans

Determination of phenolic compounds by HPLC

Phenolic compounds were determined by HPLC according to Okur et al. (2019) with slight modifications. The extracts of unroasted, traditionally roasted, and microwave-roasted samples were filtered through a 0.45 µm PTFE membrane filter before injection. Then, 20 µl of the filtered extracts were injected into the HPLC (Shimadzu Corporation, Kyoto Japan) with the SIL 10AD vp automatic sampling system. The HPLC system had a pump (LC10ADvp), degasser (DGU-14A), control oven (CTO10Avp), and a UV-VIS detector. Polyphenols were separated using a C18 column (GL Sciences, 250x4.60 mm, 5 microns, Japan). The column temperature was 30 °C. The separation was done by applying a gradient program with a binary solvent system. The mobile phases were 5% formic acid (A) and 20% A + 80% ACN (B). The flow rate was 1.0 ml/min. Phenolic compounds were carried out at 320 nm. The gradient program for phenolic compounds was applied as follows: 0–0.01 min: A 100%; 15 min: 90% A + 10% B; 90.00 min: 60% A + 40% B; 93.00 min: 100% B; 98 min: 100% A.

Multivariate analysis

Principal component analysis (PCA) was used to differentiate unroasted and roasted samples, and the results were presented as a score plot. PCA was performed using Minitab 19 (State College, PA, USA). The TPC and antioxidant activity of the samples were chosen as variables (Baltacıoğlu et al., 2024).

Statistical analysis

The Minitab (19.1.0.0, State College, PA, USA) was applied for the statistical analysis of the results. One-way ANOVA (analysis of variance) and Tukey's multiple range test were employed to determine the statistical differences.

Results

Roasted and unroasted samples were subjected to microwave-assisted extraction under different parameters. Total phenolic compounds (TPC) and antioxidant activity were determined in the extracts. The total phenolic content of the unroasted and roasted samples was shown in Table 1. The TPC values ranged from 6628.89 to 9763.29 mg GAE/100 g dry weight depending on the roasting method and microwave extraction treatment. The

highest TPC was determined in the unroasted samples extracted at 300 W for 2 min ($p < 0.05$), followed by the unroasted sample extracted at 800 W. In microwave-roasting samples, the TPC values showed no statistically significant differences between the 300 and 450 W, or 600 and 800 W, extraction treatments ($p > 0.05$). However, extraction at 600 and 800 W resulted in higher TPC values than extraction at 300 and 450 W in MW-roasting samples. Generally, high TPC values were observed at 800 W across the roasting treatments. Accordingly, higher microwave power may improve solvent penetration and promote the release of extractable phenolic compounds from the roasted seed matrix. Generally, MW-roasting samples showed lower TPC values than unroasted and traditional roasting samples.

Table 1. The TPC of unroasted and roasted samples extracted with different MW treatment

Treatment	*TPC (mg GAE / 100g dry weight)*		
	Unroasted	Traditional Roasting	MW Roasting
300W-2 minutes	$9763{,}29 \pm 435{,}98^{a}$	$8117{,}84 \pm 83{,}70^{e,f}$	$6903{,}84 \pm 234{,}98^{g}$
450 W-2 minutes	$9065{,}99 \pm 296{,}93^{b,c,d}$	$8566{,}68 \pm 51{,}97^{d,e}$	$6628{,}89 \pm 28{,}46^{g}$
600W-2 minutes	$7127{,}75 \pm 81{,}72^{g}$	$8915{,}66 \pm 19{,}52^{c,d}$	$7878{,}52 \pm 45{,}36^{f}$
800 W-2 minutes	$9506{,}98 \pm 39{,}29^{a,b}$	$9372{,}35 \pm 212{,}24^{a,b,c}$	$7993{,}97 \pm 107{,}30^{f}$

Values are expressed as mean ± standard deviation. Means that do not share a common superscript letter are significantly different according to Tukey's test ($p < 0.05$).

Table 2. The antioxidant activity of unroasted and roasted samples extracted with MW treatment

Treatment	*% inhibition*		
	Unroasted	Traditional Roasting	MW Roasting
300W-2 minutes	$56{,}70 \pm 0{,}90^{a,b}$	$54{,}90 \pm 2{,}00^{b,c}$	$44{,}78 \pm 2{,}16^{g}$
450 W-2 minutes	$50{,}48 \pm 0{,}42^{d,e}$	$54{,}59 \pm 1{,}47^{b,c}$	$51{,}26 \pm 0{,}90^{d,e}$
600W-2 minutes	$50{,}10 \pm 0{,}07^{d,e}$	$55{,}26 \pm 0{,}05^{a,b,c}$	$46{,}41 \pm 1{,}00^{f}$
800 W-2 minutes	$48{,}89 \pm 0{,}42^{e,f}$	$58{,}17 \pm 0{,}42^{a}$	$52{,}68 \pm 0{,}42^{c,d}$

Values are expressed as mean ± standard deviation. Means that do not share a common superscript letter are significantly different according to Tukey's test ($p < 0.05$).

Table3. Phenolic compounds and their amounts in roasted and unroasted samples

	Gallic acid (mg/kg)	*3,4-Dihydroxybenzoic acid (mg/kg)*	*Caffeic acid (mg/kg)*	*p-coumaric acid (mg/kg)*	*Rutin (mg/kg)*
Unroasted	ND	ND	$72{,}44 \pm 4{,}33$	$20{,}99 \pm 0{,}55^{a}$	$270{,}38 \pm 8{,}19^{a}$
Traditional Roasting	$9394{,}42 \pm 92{,}70^{a}$	$1821{,}06 \pm 58{,}53^{a}$	ND	$18{,}55 \pm 1{,}81^{b}$	ND
MW Roasting	$5135{,}45 \pm 113{,}86^{b}$	$779{,}0567 \pm 34{,}49^{b}$	ND	$15{,}43 \pm 0{,}33^{c}$	$53{,}91 \pm 11{,}72^{b}$

ND: Not detected; Different letters (a, b, c) in the same column indicated a significant difference between the treatments

The AA of the unroasted and roasted samples was shown in Table 2. The antioxidant activity ranged from 44.78% to 58.17% depending on the roasting method and microwave extraction treatment. The highest antioxidant activity was observed in the traditional roasting sample extracted at 800 W for 2 min, while the lowest value was determined in the MW-roasting sample extracted at 300 W for 2 min. In unroasted samples, antioxidant activity tended to decrease as the microwave extraction power increased. In unroasted samples, antioxidant activity tended to decrease as the microwave extraction power increased. In contrast, traditional roasting samples generally showed higher antioxidant activity, with the maximum value obtained at 800 W. Additionally, MW-roasting samples exhibited lower antioxidant activity than traditional roasting samples.

The phenolic compounds and their amounts detected in date seed samples, extracted at 800 W for 2 min, were shown in Table 3. HPLC chromatograms of roasted and unroasted samples of date seed was shown in Figure 1. As shown in Table 3, caffeic acid, p-coumaric acid, and rutin were identified in unroasted date seeds, whereas gallic acid and 3,4-dihydroxybenzoic acid were additionally detected in roasted samples. These results indicate that roasting significantly influenced the phenolic composition of date seeds ($p < 0.05$). The roasting process enables the release of phenolic compounds in the structure. Their highest concentrations were observed in the traditional roasting samples, reaching 9394.42 and 1821.06 mg/kg, respectively. On the other hand, roasting caused a decrease in the amount of p-coumaric acid and rutin compounds. MW-roasting was less effective than traditional roasting in terms of the release of bound phenolic compounds in the structure. Rutin was the predominant phenolic compound in unroasted date seed samples.

As shown in the PCA score plot, the first two principal components explained 100% of the total variance, with PC1 accounting for 51.1% and PC2 for 48.9%. Traditional roasting samples were located on the positive side of PC1, indicating relatively high combined total phenolic content and antioxidant activity. According to PC2, the unroasted samples were separated from both traditional roasting and MW-roasting samples. The close clustering of the replicates within each treatment indicated good repeatability of the measurements.

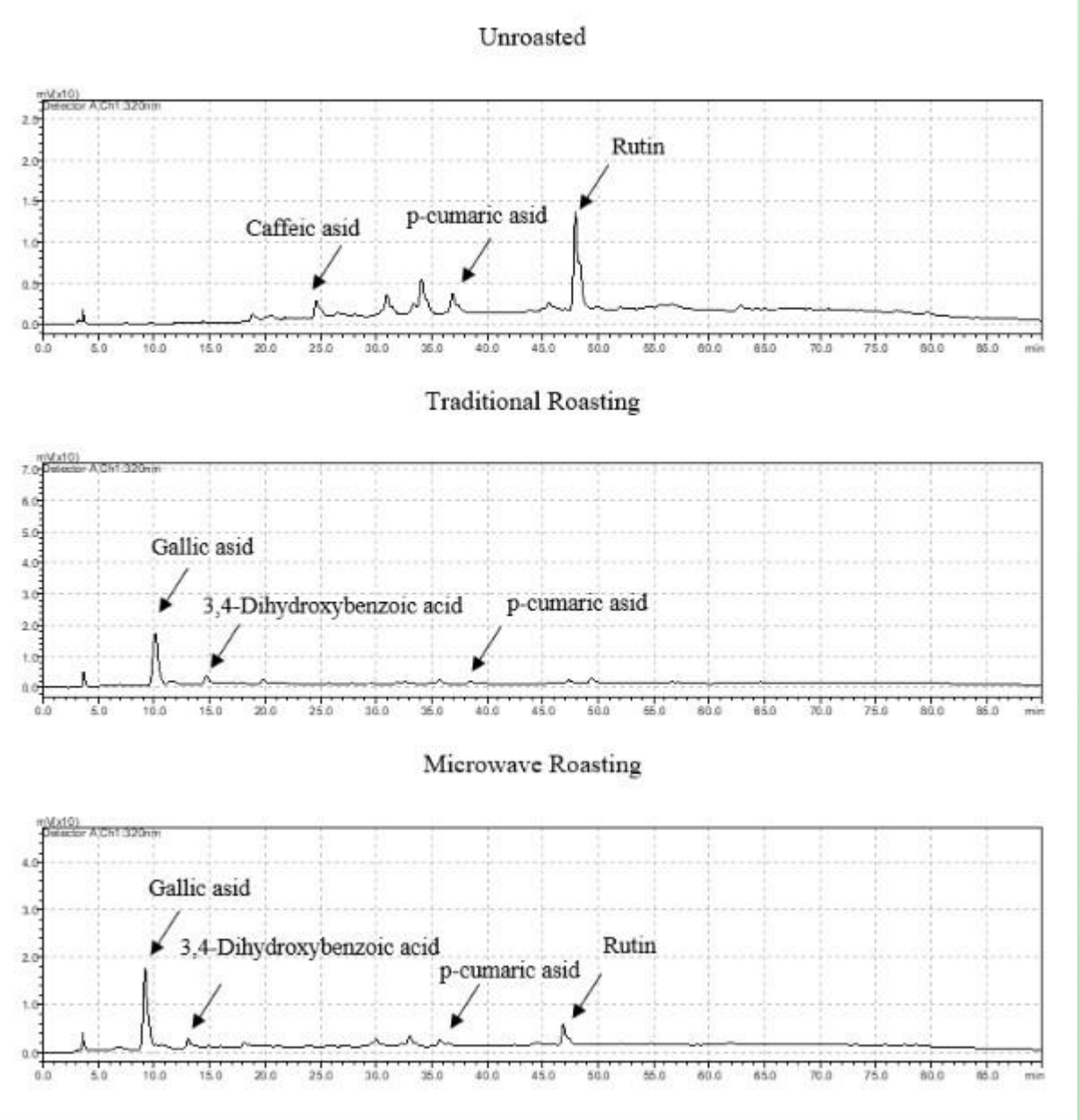

Figure 1. HPLC chromatogram of roasted and unroasted samples of date seed at 320 nm

Discussion

The total phenolic content was influenced by both the roasting method and microwave extraction power. In the present study, TPC values ranged from 6629 to 9763 mg GAE/100g dry weight, which were higher than the range of 33.4–58.9 mg GAE/g. Khalfi et al. (2024) determined that microwave-assisted extraction performed at 1200 W under varying process conditions resulted in total phenolic contents ranging from 33.4 to 58.9 mg GAE/g dry powder.

Ranasinghe et al. (2024) reported that the total phenolic content of defatted date seed powder reached 15.88 mg GAE/g DSP under optimized microwave-assisted extraction conditions. In this study, traditional roasting samples generally exhibited higher TPC values than MW-roasting samples. Likewise, Fikry et al. (2019) observed an increase in TPC during conventional oven roasting and associated this increase with structural changes in the seed matrix and the formation of Maillard reaction products. In contrast, Doğanay and Baltacıoğlu (2025) reported that microwave roasting at 300 W for 12 min resulted in a higher total phenolic content than traditional roasting. The lower TPC values in microwave-roasted samples may be related to the degradation of heat-sensitive phenolic compounds during microwave heating (Vicente-Zurdo et al., 2025).

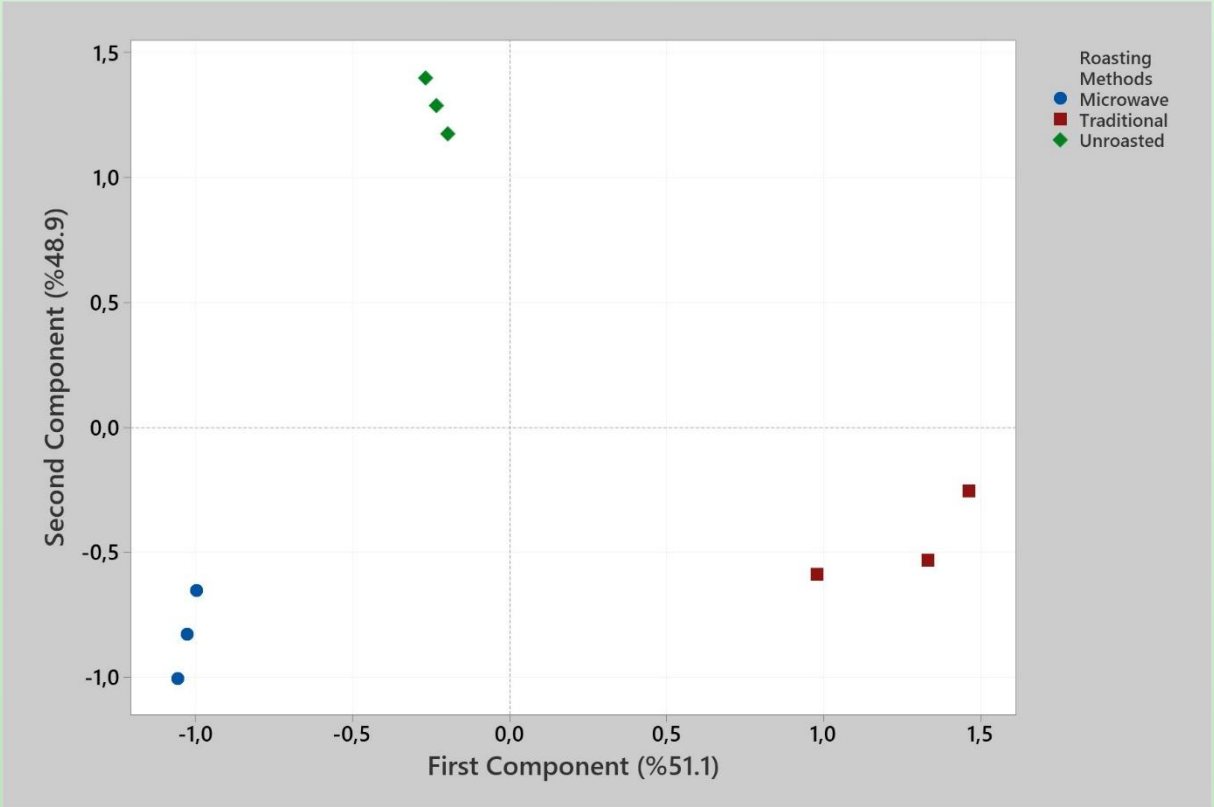

Figure 2. Score plot of two components for the effect of different roasting methods on the extraction of date seeds.

Antioxidant activity was affected by both the roasting method and microwave extraction power. In the present study, DPPH inhibition values ranged from 44.78% to 58.17%. The highest antioxidant activity was obtained from the traditional roasting samples extracted at 800 W for 2 min. Doğanay and Baltacıoğlu (2025) also found that traditional roasting date seeds exhibited the highest antioxidant activity among the roasting treatments. These results associated with structural changes in the seed matrix and the formation of antioxidant Maillard reaction products during roasting. Fikry et al. (2019) reported that the DPPH radical-scavenging activity of date seed brews increased from approximately 40% to 81% as roasting temperature and time increased. This improvement was attributed to antioxidant Maillard reaction products, particularly melanoidins, formed during roasting. Additionally, the lower values observed in some MW-roasting samples may indicate that rapid heating affected thermostable antioxidant compounds. Additionally, antioxidant capacity may depend not only on the total amount of phenolics but also on the types of phenolic compounds and on roasting-induced reaction products.

The phenolic acids identified in date seeds include p-hydroxybenzoic acid, protocatechuic acid, m-coumaric acid, gallic acid, vanillic acid, caffeic acid, p-coumaric acid, ferulic acid, and o-coumaric acid. Among these compounds, p-hydroxybenzoic acid, protocatechuic acid, and m-coumaric acid were reported as the predominant phenolic acids (Can, 2024). Radfar et al. (2019) reported that phenolic acids, particularly cinnamic, chlorogenic, and caffeic acids, were predominant in Iranian date palm seeds regardless of cultivar. In contrast, Babiker et al. (2020) identified hydroxybenzoic acids as the major phenolic acids in both unroasted and roasted date seeds. Previous studies have suggested that roasting may facilitate the release of polyphenols bound to cellular components (Abdel-Karim et al. 2024). In the present study, rutin was the predominant phenolic compound in unroasted date seeds, whereas gallic acid and 3,4-dihydroxybenzoic acid became detectable after roasting. The roasting process enables the release of phenolic compounds in the structure.

PCA was performed using the TPC and antioxidant activity data of samples extracted at 800 W for 2 min. The PCA results confirmed that the roasting method caused distinct changes in the phenolic and antioxidant characteristics of date seed extracts obtained at 800 W for 2 min. Similarly, Doğanay and Baltacıoğlu (2025) showed that PCA differentiated unroasted, traditional roasting, and MW-roasting date seed samples based on their bioactive compound profiles and antioxidant activities. Similarly, Halabi et al. (2024) observed a positive relationship between phenolic compounds and antioxidant activity in roasted date seed extracts.

Conclusion

This study shows that the phenolic and antioxidant properties of date seeds were influenced by both the roasting method and microwave-assisted extraction power. The highest TPC value was obtained from the unroasted samples extracted at 300 W, whereas the highest antioxidant activity was observed in the traditional roasting samples extracted at 800 W. Traditional roasting samples also contained the highest concentrations of gallic acid and 3,4-dihydroxybenzoic acid. In contrast, rutin was the predominant phenolic compound in unroasted date seeds, and its concentration decreased after roasting. MW-roasting samples generally showed lower TPC and antioxidant activity than the other treatments. PCA further confirmed the differentiation of the samples according to roasting treatment.

Declarations

Author Contribution Statement

Gözde Doağnay: Data collection, investigation, formal analysis, and writing the original draft
Hande Baltacıoğlu: Project administration, supervision, conceptualization, methodology, review and editing

Fund Statement

This research has been funded by the Scientific Research Projects Coordination Unit of Niğde Ömer Halisdemir University. Project Number: TGT 2023/14- LÜTEP.

Conflict of Interest

The authors declare no conflict of interest.

References

Abd-Elkarim, N. A., Abdrabou, E. A., & Osman, A. A. (2024). Influence of roasting and microwave heating on nutritional value and bioactive compounds of Siwi date seed. *Egyptian Journal of Food Science, 52*(1), 123–137. https://doi.org/10.21608/ejfs.2024.223982.1190

Al Harthi, S. S., Mavazhe, A., Al Mahroqi, H., & Khan, S. A. (2015). Quantification of phenolic compounds, evaluation of physicochemical properties and antioxidant activity of four date (*Phoenix dactylifera* L.) varieties of Oman. *Journal of Taibah University Medical Sciences, 10*(3), 346–352. https://doi.org/10.1016/j.jtumed.2014.12.006

Babiker, E. E., Atasoy, G., Özcan, M. M., Al Juhaimi, F., Ghafoor, K., Mohamed Ahmed, I. A., & Almusallam, I. A. (2020). Bioactive compounds, minerals, fatty acids, color, and sensory profile of roasted date (*Phoenix dactylifera* L.) seed. *Journal of Food Processing and Preservation, 44*(7), e14495. https://doi.org/10.1111/jfpp.14495

Baltacıoğlu, H., Şahin, E. M., & Karadağ, E. D. (2019). Extraction of phenolic compounds from peach pomace by microwave and ultrasound assisted extraction. *Niğde Ömer Halisdemir University Journal of Engineering Sciences, 8*(2), 875–881. https://doi.org/10.28948/ngumuh.570250

Can, N. (2024). Date seed as a functional food ingredient. *Anadolu Bil Meslek Yüksekokulu Dergisi,19*(70),129–147. https://doi.org/10.17932/IAU.ABMYOD.2006.005/abmyod_v19i70002

Baltacıoğlu, C., Baltacıoğlu, H., Okur, İ., Yetişen, M., & Alpas, H. (2024). Recovery of phenolic compounds from peach pomace using conventional solvent extraction and different emerging techniques. *Journal of Food Science, 89*(3), 1672–1683. https://doi.org/10.1111/1750-3841.16972

Doğanay, G., & Baltacıoğlu, H. (2025). Effect of microwave roasting and different solvents on the extraction of bioactive compounds from date seed. *Eurasian Journal of Science Engineering and Technology, 6*(1), 41–46. https://doi.org/10.55696/ejset.1622241

Fikry, M., Yusof, Y. A., Al-Awaadh, A. M., Abdul Rahman, R., Chin, N. L., & Mohd Ghazali, H. (2019). Antioxidative and quality properties of full-fat date seeds brew as influenced by the roasting conditions. *Antioxidants, 8*(7), 226. https://doi.org/10.3390/antiox8070226

Halabi, Y., Nasri, C., El Guezzane, C., Harhar, H., Gharby, S., Zarrouk, A., Bellaouchou, A., & Tabyaoui, M. (2024). Optimized roasting parameters of full-fat date palm seed (*Phoenix dactylifera* L.) using a central composite design and chemometric approach to prepare antioxidant-rich beverage. *Letters in Applied NanoBioScience, 13*(3), 113. https://doi.org/10.33263/LIANBS133.113

Flowers, J. M., Hazzouri, K. M., Gros-Balthazard, M., Mo, Z., Koutroumpa, K., Perrakis, A., Ferrand, S., Khierallah, H. S. M., Fuller, D. Q., Aberlenc, F., Fournaraki, C., & Purugganan, M. D. (2019). Cross-species hybridization and the origin of North African date palms. *Proceedings of the National Academy of Sciences of the United States of America, 116*(5), 1651–1658. https://doi.org/10.1073/pnas.1817453116

Khalfi, A., Garrigós, M. C., Ramos, M., & Jiménez, A. (2024). Optimization of the microwave-assisted extraction conditions for phenolic compounds from date seeds. *Foods, 13*(23), 3771. https://doi.org/10.3390/foods13233771

Melikoglu, M. (2025). Microwave-assisted extraction: Recent advances in optimization, synergistic approaches, and applications for green chemistry. *Sustainable Chemistry for Climate Action, 7*, 100122. https://doi.org/10.1016/j.scca.2025.100122

Radfar, R., Farhoodi, M., Ghasemi, I., Mousavi Khaneghah, A., Shahraz, F., & Hosseini, H. (2019). Assessment of phenolic contents and antioxidant and antibacterial activities of extracts from four varieties of Iranian date palm (*Phoenix dactylifera* L.) seeds. *Applied Food Biotechnology, 6*(3), 173–184. https://doi.org/10.22037/afb.v6i3.23379

Ranasinghe, M., Sivapragasam, N., Mostafa, H., Airouyuwa, J. O., Manikas, I., Sundarakani, B., Maqsood, S., & Stathopoulos, C. (2024). Valorizing date seeds in biscuits: A novel approach to incorporate bioactive components extracted from date seeds using microwave-assisted extraction. *Resources, Environment and Sustainability, 15*, 100147. https://doi.org/10.1016/j.resenv.2023.100147

Shi, L., Wu, H., Ghafoor, K., Gonzalez Viejo, C., Fuentes, S., Ahmadi, F., & Suleria, H. A. R. (2025). Impacts of roasting intensity and cultivar on date seed beverage quality traits and volatile compounds using digital technologies. *Foods, 14*(22), 3902. https://doi.org/10.3390/foods14223902

Vicente-Zurdo, D., Gómez-Mejía, E., Morante-Zarcero, S., Rosales-Conrado, N., & Sierra, I. (2025). Analytical strategies for green extraction, characterization, and bioactive evaluation of polyphenols, tocopherols, carotenoids, and fatty acids in agri-food bio-residues. *Molecules, 30*(6), 1326. https://doi.org/10.3390/molecules30061326

Zarie, A. A., Hassan, A. B., Alshammari, G. M., Yahya, M. A., & Osman, M. A. (2023). Date industry by-product: Date seeds (*Phoenix dactylifera* L.) as potential natural sources of bioactive and antioxidant compounds. *Applied Sciences, 13*(21), 11922. https://doi.org/10.3390/app132111922