



ORIGINAL ARTICLE

Investigating the Impact of Roasted Date Seed (*Phoenix dactylifera* L.) Oil on the Quality and Oxidative Stability of Bakery Margarine

Rahma Mayouf¹ | Fatiha Hamitri-Guerfi¹ | Aicha Benbouriche¹ | Mohamed Ali Ayadi² | Samir Hadjal³ | Brahim Zeroual³ | Toufik Ouatmani³ | Morad Hamitri³ | Hamadi Attia⁴ | Lila Boulekbache-Makhlouf¹ | Imène Felfoul⁴ [5]

¹Université de Bejaia, Faculté Des Sciences de la Nature et de la Vie, Laboratoire de Biochimie Biophysique Biomathématique et Scientométrie, Bejaia, Algeria | ²Laboratory of Quality and Safety of Agri-Food Products, Gembloux Agro-Bio Tech, University of Liège, Gembloux, Belgium | ³Laboratoire Central Des Corps Gras, Complexe Agro-Alimentaire de CEVITAL Agro-Industrie, Bejaia, Algeria | ⁴Laboratoire Analyses, Valorisation et Sécurité Des Aliments (LAVASA), Ecole Nationale D'ingénieurs de Sfax, Université de Sfax, Sfax, Tunisie

Correspondence: Imène Felfoul (imenef@gmail.com; imen.falfoul@isbs.usf.tn)

Received: 10 February 2025 | Revised: 15 June 2025 | Accepted: 28 June 2025

Funding: The authors received no specific funding for this work.

Keywords: date seed oil | enrichment | margarine | oxidative stability | roasting

ABSTRACT

Bakery margarine is one of the most commonly used fat products in the food industry. Its oxidative stability is a critical quality parameter, typically maintained using synthetic antioxidants such as tertiary butylhydroquinone (TBHQ) due to its strong antioxidant activity. However, excessive daily consumption of TBHQ has been linked to potential health risks, prompting increased interest in natural alternatives. In this work, oil extracted from roasted and unroasted date seeds were analyzed and compared for there phenolic composition and free radical scavenging capacity. In addition, bakery margarines prepared by formulation with the roasted and unroasted date seed oils (10 g/kg) were compared to margarine supplemented with the synthetic antioxidant TBHQ (200 m/kg) as a control. They were evaluated in terms of quality parameters, oxidative stability measured by induction time, peroxide value during accelerated storage for 10 weeks at 60°C, and fatty acid profile. The results showed that pre-treatment of the seeds by roasting significantly increased phytochemical content and antioxidant capacity. Margarine containing roasted date seed extracts showed oxidation resistance similar to the control without altering its quality. However, it showed superior resistance properties and a longer induction time (increased from 24.6 to 31 h) compared to margarine containing unroasted date seed oil. This study highlights, for the first time, the role of roasting in improving the quality of date seed oil. These results suggest that roasted date seed oil can effectively improve the shelf life and stability of margarine, offering a promising natural alternative to synthetic antioxidants in food applications.

1 | Introduction

Margarine, an emulsified fatty food (water-in-fat), is widely used as a substitute for spreadable butter, which is mainly composed of an average of 80 to 90% fat (Fruehwirth et al. 2021). Margarines can be classified based on the firmness and melting

point of their fats into bakery margarine, which includes hard and medium plastic margarine used as bakery fat in short pastry, cakes, cookies, breads, and pastries, and table margarine, which is generally medium and soft plastic margarine commonly used as spreads for table consumption (Miskandar et al. 2005; Pădureţ 2022).

© 2025 AOCS.

Bakery margarine, one of the essential fatty products in the food industry, imparts a variety of advantageous qualities to both dough and finished cookies (Das and Das 2024). These include facilitating dough mixing, providing essential aeration, improving flavor release, and creating the desired texture and mouthfeel in cookies (Cheong et al. 2009). Therefore, industries rely on the addition of some vegetable oils such as soybean, corn, sunflower, and palm oils in order to stabilize solid fat crystals (Bolini et al. 2023). In addition, many synthetic antioxidants (such as tertiary-butylhydroquinone, butylated hydroxyanisole, butylated hydroxytoluene, and propyl gallate) have been used to prevent lipid oxidation due to the high oxidative deterioration of margarine during storage and heat treatment, caused by the large contact surface between oxidizable lipids in the emulsion droplets and water-soluble prooxidants (Ghelichi et al. 2023), which ultimately reduces the sensory and nutritional quality of the food.

Several studies have investigated the effectiveness of these antioxidants and have suggested that tertiary-butylhydroquinone (TBHQ) has superior antioxidant properties compared to others such as butylated hydroxyanisole (BHA) and butylated hydroxytoluene (BHT), which explains its extensive use in the food industry (López et al. 2022; Palanna et al. 2025). However, the antioxidant efficiency of TBHQ is better at low temperatures than at high temperatures due to its high thermal decomposition (Liu et al. 2016). Indeed, its high consumption can induce toxic and carcinogenic effects (Azizkhani and Zandi 2009). As a result, consumers have become more interested in replacing synthetic products with healthier and natural ones. The food industry focuses on using natural products from many sources (such as plants, micro-organisms, fungi, algae, animals and agri-food waste) as substitutes for synthetic antioxidants (Moure et al. 2001). Among agri-food waste, date seeds, which represent about 15% of the total weight of dates, are considered an important by-product resulting from fruit processing. This byproduct is a valuable source of bioactive compounds that can be valorized by the food industry (Golshan Tafti et al. 2017). In addition, date seeds contain between 5% to 13% of oil with a healthy fatty acid profile composed mainly of oleic and linoleic acids (monounsaturated fatty acids) and lauric, myristic, and palmitic acids (saturated fatty acids) (Subhash et al. 2024; Ali and El-Anany 2025). The richness of oleic acid in oils plays important roles in increasing health benefits, including lowering blood cholesterol levels and preventing cholesterol build-up in blood vessels (Chen et al. 2023). Furthermore, date seed oil is rich in lipophilic antioxidants and exhibits thermal stability, making it an ideal candidate for use as high-quality edible oil in food formulations (Harkat et al. 2022). Notably, the stability of vegetable oil during heating, as well as the functional properties and sensory characteristics of mayonnaise, were improved by replacing synthetic antioxidants and corn oil with date seed oil, respectively (Basuny and Al-Marzooq 2011; Taha et al. 2019). Beyond its functional properties, date seed oil is a valuable economic resource that makes a significant contribution to reducing industrial waste, with an annual extraction potential of around 3900t from date seeds (El Harkaoui et al. 2024; Subhash et al. 2024).

In order to improve the oxidative stability of the extracted oil by improving its phenolic content and antioxidant activity, the food industry has increasingly adopted various techniques, including refining, blending, and enrichment with natural antioxidants (Benbouriche et al. 2022; Benbouriche et al. 2023). Among these methods, roasting has emerged as a particularly effective approach, as it promotes the release of bound phenolic compounds and significantly boosts the antioxidant potential of the resulting oil. This has led to the widespread application of roasting in the treatment of seeds prior to oil extraction (Belcadi-Haloui et al. 2018; Akinoso et al. 2023). Moreover, roasting of date seeds is commonly optimized and applied due to the enhanced antioxidant activity and total polyphenol content (TPC) of the resulting powder, which last is often used in some preparations, such as coffee, regarding its favorable sensory attributes (Fikry et al. 2019). To the best of the authors' knowledge, no data are currently available in the literature regarding the impact of roasting date seeds on the quality of their oil or its potential applications in food products. In this context, the present study focuses on the use of roasted date seed oil to enhance the quality of bakery margarine through a novel formulation, emphasizing its potential as a natural source of antioxidants. This new formulation is evaluated and compared to a control margarine fortified with 200 mg/kg of the synthetic antioxidant TBHQ.

2 | Material and Methods

2.1 | Reagents

All the chemical reagents and analytical standards used in the present study were purchased from Sigma-Aldrich (St. Louis, MO, USA).

2.2 | Date Seeds Preparation and Oil Extraction

Date palm (*Phoenix dactylifera* L.) fruits of Degla Beida variety were acquired from the Biskra region (Algeria). The seeds were manually removed and soaked to remove adhering residuals, then divided into two portions, each weighing 100 g. One portion was roasted in a forced-ventilation oven (MEMMERT, Germany) at 200°C for 22 min, following the procedure described by Fikry et al. (2019), while the other portion was kept without roasting treatment. The roasting process was performed in triplicate.

Oil extraction from roasted and unroasted date seeds followed the method described by Rakhshanda et al. (2020). About 20 g of ground date seeds were placed in a cellulose thimble and stirred with 250 mL of n-hexane for 4h at room temperature (25°C). After that, the solvent was removed using a rotary evaporator at 40°C until weight stabilization. The extraction process was carried out in triplicate. The extraction yield values were 9.52 \pm 0.41 (w%) and 10.49 \pm 0.38 (w%) for unroasted and roasted date seeds, respectively.

2.3 | Phenolic Compounds Measurement of Date Seed Oil

Polyphenol compounds from oil samples were extracted by liquid-liquid separation using methanol-water (80:20 v/v)

as extraction solvent, following the procedure of Nenadis et al. (2018). A mass of 2.5g of oil sample was mixed with $5\,\mathrm{mL}$ of hexane and $5\,\mathrm{mL}$ of methanol–water (80:20 v/v) solvent in a separating funnel. After mixing, centrifugation was carried out at $5000\,\mathrm{rpm}$ for $10\,\mathrm{min}$ at room temperature ($20\,^\circ\mathrm{C}$) in order to separate the two phases obtained. The lower phase, containing the phenolic compound, was collected, and the solvent was then evaporated under vacuum ($60\,^\circ\mathrm{C}$). The methanolic extract was filtered through a $0.45\,\mu\mathrm{m}$ membrane filter before injection into the high-performance liquid chromatography (Agilent 1260 Infinity quaternary LC, Germany) system.

Phenolic compounds were separated using a C18 column (Thermo Electron, Dreieich, Germany) (4.6 mm \times 250 mm, $3\,\mu m)$ and then analyzed by a Shimadzu SPD6AUV detector measuring the optical density at 254 nm during 50 min. The mobile phase was a mixture of A and B solutions: (A) 70% acetonitrile in water and (B) 0.1% formic acid in water, with the percentage by volume of (A) solution varying linearly during the time as follows: from 10% to 25% for the first 25 min, then from 25% to 80% up to 35 min, and finally from 80% to 100% up to 50 min. The column temperature was maintained at 40°C, and the mobile flow rate was fixed at 0.5 mL/min (Abid et al. 2017).

2.4 | Antioxidant Activity Measurement of Date Seed Oil

2.4.1 | Ferric Ion Reducing Power (FRAP) Test

The ferric ion reducing power (FRAP) test was conducted as previously described by Mohd Jaih et al. (2019). 1 mL of each oil sample, diluted in DMSO (dimethyl sulfoxide), was mixed with 2 mL of FRAP reagent and adjusted to 10 mL with redistilled water. FRAP reagent contained a mixture of TPTZ (2,4, 6-tri-(2-pyridyl)-1,3,5-triazine) solution (10 mM in 40 mM HCl), FeCl₃ (20 Mm) and acetate buffer (0.1 M, pH 3.6) in a volume ratio of 1:1:10. The absorbance was observed at 593 nm after 10 min of incubation using a blank control, which consisted only of FRAP reagent and redistilled water, without any oil sample. The results were calculated using a Trolox standard curve prepared at different concentrations of 0.1, 0.05, 0.025, 0.0125, and 0.00625 mg/mL and expressed as mg of Trolox equivalent per g of sample (mg TE/g).

2.4.2 | β-Carotene Bleaching Test

According to the method of Yao et al. (2020), the β -carotene bleaching assay was measured by dissolving 10 mg of β -carotene in chloroform (10 mL). Then, the solution was combined with 20 mg of linoleic acid and 200 mg of Tween 40. After removing the chloroform using the rotary evaporator at 40°C, 50 mL of distilled water was added to formulate an emulsion. 200 μ L of seed oil at different concentrations previously diluted in DMSO (0.4 to 0.00625 g/mL) were mixed with 5 mL of the emulsion. The absorbance was immediately measured at 470 nm and after incubation in a water bath at 50°C for 60 min at the same wavelength. The control consisted of 200 μ L of DMSO instead of

diluted seed oil. Antioxidant activity was expressed as percent inhibition as follows (Equation 1):

$$AA\% = 100 \times (DC - DS) / DC \tag{1}$$

where, AA (%): antioxidant activity; DC: degradation rate of the control; DS: degradation rate of the sample. Both DC and DS=[$\ln(a/b)/60$], where a: absorbance at 0min and b: absorbance at 60min. The results were described by IC50 (mg/mL), which is the concentration required to induce a 50% β -carotene bleaching inhibition.

2.4.3 | Metal Chelating Activity by Ferrozine Test

Metal chelating activity was performed as described by Wannes and Marzouk (2016). 100 μL of seed oil at various concentrations, prepared in DMSO, were combined with $50\,\mu L$ of FeCl $_2$ (2 mM) solution and incubated at room temperature (25°C) for 5 min. Then, 0.1 mL of ferrozine (5 mM) was added, and the total volume was adjusted to 3 mL with 1 mL distilled water. The resulting solution was maintained at room temperature (25°C) for 10 min. In the control sample, DMSO (100 μL) was used instead of seed oil. The absorbance was then measured at 562 nm. The percentage of metal-chelating activity was calculated using the following formula (Equation 2):

Metal chelating activity (%) =
$$[(Ac - As)/Ac] \times 100$$
 (2)

Ac: absorbance of the control, As absorbance of sample. The results were reported as IC50 (mg/mL), representing the concentration required to induce a 50% metal chelation activity.

2.5 | Study of Margarine

2.5.1 | Margarine Formulation

Margarines preparation for bakery applications was manually carried out at laboratory scale at CEVITAL Spa, an agri-food company located in Béjaïa, northern Algeria. The lipid phase contains 80% of the total amount, including an emulsifier (lecithin) dissolved in the heated oil phase, composed of palm oil, sunflower oil, and an equivalent of hydrogenated soybean oil. The remainder represents the liquid phase containing salt, lactic acid, and potassium sorbate, all dissolved in deionized water. During the formulation process, 5g (10g/kg) of both unroasted and roasted date seed oil was introduced into the lipid phase to formulate margarine with unroasted oil (UOM) and margarine with roasted oil (ROM), respectively. The quantity of date seed oil added was adjusted to achieve a final margarine weight of 500 g for each formulation. This amount was selected based on economic criteria defined by the company's laboratory experts, while ensuring that the fatty acid profile of the bakery margarine was not altered. Commercial margarine produced by the industrial agri-food CEVITAL was used as the control (MC). It was prepared by fortification with 200 ppm of TBHQ instead of date seed oil, which corresponds to the maximum standard limit for the use of synthetic antioxidants in fat and oil (United States Food and Drug Administration 2024). The final margarine

products were stored in 250g plastic trays and kept at 4°C in the dark for 1 day prior to analysis.

2.5.2 | Measurement of Physicochemical Properties

Physicochemical properties of the produced margarines, including pH, moisture content, water activity, acidity (oleic), peroxide value, melting point, salt content, and solid fat content (SFC), were determined.

pH value: The pH of the margarines was measured directly by immersing the pH-meter electrode (Testo 206, Testo-AG Germany) into the aqueous phase.

Moisture content: The moisture content was determined by drying 3g of margarine at 105°C until a constant weight was achieved (ISO-662 1998).

Water activity: Water activity (aw) was measured at 25°C using a water activity meter (Novasina Aw Sprint TH-500, Axair Ltd., Pfäffikon, Switzerland).

Peroxide value (PV): PV was determined according to ISO-3960 (2007). Briefly, 5g of margarine oil was dissolved in a solution containing acetic acid (18 mL), chloroform (12 mL) and saturated potassium iodide solution (1 mL). The resulting mixture was kept in the dark for 5 min. A total volume of 75 mL distilled water and a few drops of starch solution (1%) were added, and then the solution was titrated with sodium thiosulfate solution (0.01 N). The Peroxide Value (meqO $_2$ /kg) was calculated using the following Equation (3):

$$PV (meqO2 / kg) = (V \times N \times 1000) / M$$
 (3)

where, N, normality of the sodium thiosulfate, V, volume (mL) of sodium thiosulfate, M, mass of the sample (g).

Acidity value (A%): The acidity was determined by neutralizing free fatty acids with a solution of NaOH in the presence of phenolphthalein as a color indicator (ISO-660 2009). The result is expressed as follows (Equation 4):

$$A(\%) = (282 \times N \times V) / (10 \times M)$$
 (4)

Where, 282: molar mass of oleic acid (g/mol), N: normality of NaOH solution (0.1 N), M: weight of the sample (g), V: volume of the NaOH solution.

Salt content: The salt level was determined by dissolving 5 g of margarine in boiling distilled water (100 mL). The resulting solution was then titrated with a solution of silver nitrate $AgNO_3$ (0.1 N) in the presence of a color indicator (potassium chromate 5%) (ISO-15648 2004). The salt content (NaCl) is determined according to the following formula (Equation 5):

Salt content (%) =
$$[(V \times N \times 58.5) / (10 \times M)] \times 100$$
 (5)

Where, V: volume (mL) of the AgNO₃ solution, N: normality of AgNO₃ (0.1 N), 58.5: equivalent weight of NaCl (g/mol), M: weight of the sample (g).

Melting point: The melting point refers to the minimum temperature, expressed in degrees Celsius, at which a lipid changes from a solid to a liquid state. The method consists of introducing margarine fat into two glass capillary tubes at a height of 1 cm. After cooling for 20 min, the capillary tubes were connected to a thermometer. The whole was immersed in water heated to 0.5°C/min. The temperature at which the margarine fat melted was then recorded (ISO-6321 2002).

Solid fat content (SFC): SFC was carried out using a nuclear magnetic resonance (NMR) spectrometer (minispecmq 20, Germany). The process consists of separating and filtering of fat from margarine and incubating it in a water bath for 30 min at temperatures ranging from 0°C to 40°C, in accordance with ISO-8292-1 (2008).

2.5.3 | Color Parameters Measurement

The color of margarine and date seed oil samples used in their formulation was measured using a colorimeter (Konica Minolta Inc. Japan) in the CIE $L^*a^*b^*$ system. The colorimetric parameters L^* (lightness), a^* (redness/greenness) and b^* (yellowness/blueness) were determined. L^* ranges from 0 (black) to 100 (white), a^* ranges from -100 (greenness) to +100 (redness) and b^* ranges from -100 (blueness) to +100 (yellowness) (Samet-Bali et al. 2009). As the values of L^* , a^* , and b^* rise, the color becomes more saturated or chromatic; while for neutral colors (white, gray or black), these values approach zero. The chroma (C^*) and hue angle (h°), representing the saturation level and shade of the color, respectively, as well as the color index (ΔE), were calculated as follows (Equations 6-8):

$$h^{\circ} = \tan^{-1} \left(b^* / a^* \right) \tag{6}$$

$$C^* = \left(a^{*2} + b^{*2}\right)^{1/2} \tag{7}$$

$$\triangle E = \left[\left(\triangle L^{*^{\circ}2} \right) + \left(\triangle a^{*^{\circ}2} \right) + \left(\triangle b^{*^{\circ}2} \right) \right]^{-1/2} \tag{8}$$

2.5.4 | Texture Parameters Measurement

Texture of margarine samples was determined using Textural Profile Analysis (TPA) test. A Texture Analyzer (LLOYD instruments, Fareham, England) was used to measure the force–time curve for two cycles of compression (Ammar et al. 2021). All measurements were performed in a controlled room at 25°C. The measurements were carried out on 50 mm-width \times 50 mm-length \times 40 mm-height margarine samples. An aluminum cylinder probe was used to compress the margarine to 50% (20 mm) of its original height with a displacement speed of 2 mm/s. Texture profile parameters, hardness (N), cohesiveness, adhesiveness (N) and springiness (mm) were measured. Texture analysis was assisted on three independent refrigerated (4°C) margarine samples from each type, 24h after preparation.

2.5.5 | Measurement of Margarines Oxidative Stability

The oxidative stability of the different margarine samples was carried out by using two methods:

Rancimat test: Rancimat test is based on measuring the increase in conductivity of demineralized water induced by volatile acids transported by dry air passing through the heated sample (Farhoosh 2007). A total of 3g of margarine was introduced into the Rancimat apparatus (Metrohm AG, Herisau, Switzerland) under a temperature of 98° C and an airflow rate of $10 \, \text{L/h}$. The oxidative stability of the margarine was expressed in induction time given in hours (ISO-6886 2006).

Schaal oven test: Oxidative stability was assessed by measuring the peroxide value during a 10-week storage period in a hot air oven maintained at $60^{\circ}\text{C} \pm 1^{\circ}\text{C}$, following the method described by Azizkhani and Zandi (2009). Each type of margarine was divided into three independent replicates of approximately 100g, each stored in a sealed glass jar. During the storage period, 10g was withdrawn weekly from each replicate for peroxide value determination.

2.5.6 | Fatty Acids Composition Measurement

Fatty acid methyl esters were produced in the first step in accordance with ISO-5509 (2000). Briefly, 0.1 g of margarine fat was combined with 2 mL of heptane in a 5 mL screw-cap tube and shaken for 30 s at room temperature (20°C). Subsequently, 0.2 mL of methanolic potassium hydroxide solution (2 N) was added. The tube was tightly closed and shaken vigorously for 30 s. A volume of 0.8 μ L of FA methyl esters was injected into Chromatogram (Chrompack CP 9002) equipped with a Flame Ionization Detector and a column (30 m×0.2 mm×0.25 μ m, RTX 2330) with a split ratio of 1/100 at 250°C. The carrier gas

was nitrogen at a flow rate of 1 mL/min, and the injector and detector temperatures were set at 250°C and 260°C, respectively. Fatty acid peaks were identified and quantified by comparing their retention time, equivalent chain length, and peak surface area with respect to standard fatty acid methyl ester standards (Sigma-Aldrich, Steinheim, Germany) were used.

2.6 | Statistical Analysis

All experiments and analyses were carried out in triplicates, and the obtained data were expressed as means \pm standard deviation. The data were statistically analyzed using ANOVA following the LSD test (Least Significant Difference) by Statistica Software version 10.0 (Stat Soft Inc.). Statistical significance was considered at p < 0.05.

3 | Results and Discussion

3.1 | Determination of Phenolic Compounds of Date Seed Oil

Natural oils contain only a small proportion of phenolic compounds; however, these compounds can exhibit significant biological effects, including anticancer and antiatherogenic properties, alongside their antioxidant capabilities (Vujasinovic et al. 2012). The phenolic compounds identified in the methanol extract of the oil samples, along with their retention times, are summarized in Table 1. In the case of unroasted date seed oil, only four compounds were detected. The predominant

TABLE 1 | Phytochemical profile of date seed oil in methanolic extracts and antioxidant activities of date seed oils.

	Rt (min)	Unroasted	Roasted
Concentration (mg/kg oil)			
Gallic acid	7.482	1.35 ± 0.01^{b}	2.52 ± 0.01^{a}
Protocatchuic acid	12.355	ND	0.73 ± 0.00^{a}
Cafeic acid	17.259	ND	0.09 ± 0.01^{a}
Tyrosol	17.464	0.03 ± 0.00^{b}	0.26 ± 0.00^{a}
Vanillic acid	21.858	0.16 ± 0.00^{b}	0.22 ± 0.01^{a}
Benzoic acid	24.229	ND	0.03 ± 0.00^{a}
Vanillin	29.406	ND	0.08 ± 0.00^{a}
Rutin	32.189	0.01 ± 0.00^{b}	0.05 ± 0.01^{a}
Quercetin	36.967	ND	0.11 ± 0.02^{a}
Apegenin	38.353	ND	0.60 ± 0.02^{a}
Trans-cinnamic acid	38.588	ND	0.11 ± 0.01^{a}
Antioxidant test			
FRAP (mg TE/g)	_	0.57 ± 0.027^{b}	1.80 ± 0.025^{a}
β -Carotene bleaching IC50 (mg/mL)	_	0.271 ± 0.003^{b}	0.0673 ± 0.0027^{a}
Metal chelating activity IC50 (mg/mL)	_	0.305 ± 0.004^{b}	0.0437 ± 0.0022^a

Note: Means with different superscript letters in the same row within a sample type are significantly different according to LSD test (p < 0.05). Abbreviations: ND, not detected; Rt, retention time.

compound was gallic acid $(1.35\pm0.01\,\mathrm{mg/kg})$, followed by vanillic acid $(0.16\pm0.00\,\mathrm{mg/kg})$, tyrosol $(0.03\pm0.00\,\mathrm{mg/kg})$, and rutin $(0.01\pm0.00\,\mathrm{mg/kg})$. Harkat et al. (2022) reported higher concentrations of vanillic acid and tyrosol $(3.49\pm0.0\,\mathrm{mg/kg})$ and $1.24\pm0.01\,\mathrm{mg/kg}$, respectively) in the same Algerian variety. Additionally, 10 other compounds were identified, with concentrations ranging from $15.41\pm0.01\,\mathrm{mg/kg}$ to $0.04\pm0.01\,\mathrm{mg/kg}$. These variations may be attributed to differences in the maturity stage of the samples and the protocols employed for extraction and analysis.

The effect of roasting as a pre-treatment on the phenolic content of the extracted oil is presented in Table 1. Roasting significantly increased the phenolic content (p<0.05), with concentrations reaching 2.52±0.01 mg/kg for gallic acid, 0.22±0.01 mg/kg for vanillic acid, 0.26±0.01 mg/kg for tyrosol, and 0.05±0.01 mg/kg for rutin. Additionally, other phenolic compounds were detected in roasted date seed oil, including caffeic acid (0.09±0.01 mg/kg), benzoic acid (0.03±0.00 mg/kg), vanillin (0.08±0.00 mg/kg), apigenin (0.60±0.02 mg/kg), and trans-cinnamic acid (0.11±0.01 mg/kg).

Currently, there is any available data on the polyphenolic levels in date seed oil after roasting the seeds. However, the levels of phenolic content were found to increase significantly in roasted seeds compared to raw ones (Paranthaman et al. 2012; Abd-Elkarim et al. 2024). A similar increase in the concentrations of individual phenolic compounds due to roasting has been reported for the Gleisdorf variety of pumpkin seed oil, which aligns with the findings of this study. The authors investigated the impact of roasting at temperatures ranging from 90°C to 200°C for 45 min, observing an initial increase in polyphenol concentrations, followed by a decrease at higher temperatures. Specifically, the concentrations of vanillic acid, caffeic acid, trans-cinnamic acid, and p-coumaric acid increased from 0.37 to 0.77 mg/kg, 0.09 to 0.23 mg/kg, 0.96 to 1.35 mg/kg, and 0.96 to 1.91 mg/kg, respectively (Potočnik et al. 2018). Polyphenols naturally occur in both free and bound forms. The observed increase in phenolic compound concentrations during roasting can be attributed to heat-induced hydrolytic reactions, which release native esterified or bound forms into their free forms (Chbani et al. 2020).

3.2 | Determination of Antioxidant Activity of Date Seed Oil

The effectiveness of an antioxidant depends on several factors such as its reactivity as a hydrogen or electron donor, the stability and fate of the resulting antioxidant-derived radical (influenced by its ability to stabilize or delocalize the unpaired electron), its interactions with other antioxidants, and its capacity to chelate transition metals (Rice-Evans et al. 1997).

In the present study, the antioxidant activities of date seed oils were evaluated using three methods: the FRAP test, which measures the antioxidant's reducing power by converting ferric ions to ferrous ions; the β -carotene bleaching test, which assesses the antioxidant's ability to prevent β -carotene color loss caused by free peroxides formed during linoleic acid

oxidation; and the metal chelation activity test, which evaluates the antioxidant's capacity to inhibit the formation of the ferrozine-Fe²⁺ complex.

The results of the FRAP test are expressed as Trolox equivalents per gram of sample, while IC50 (mg/mL) values are used to represent the outcomes for β -carotene bleaching and metal chelating activity. Notably, a lower IC50 value indicates higher antioxidant activity. The summarized results are presented in Table 1.

Roasting date seeds resulted in a significant increase (p < 0.05) in antioxidant activity across all measurement methods. In the FRAP assay, antioxidant activity increased notably from 0.57 ± 0.027 to 1.80 ± 0.025 mg TE/g. For the β -carotene bleaching test, the IC50 value decreased significantly from 0.271 ± 0.003 to 0.067 ± 0.0027 mg/mL. Similarly, in the metal chelating activity assay, the IC50 value decreased from 0.305 ± 0.004 to 0.044 ± 0.0022 mg/mL. The enhanced antioxidant effect observed after heat treatment aligns with the increased phenolic acid levels and may be attributed to the formation of Maillard reaction products during roasting, which are well-known for their antioxidant properties (Mohamed Ahmed et al. 2020; Karrar et al. 2021).

Consistent with our findings, several studies have reported that roasting seeds enhances the antioxidant activity of the resulting oils, including peanut oil, walnut oil, and nigella oil (Gao et al. 2018; Ciou et al. 2021; Suri et al. 2022).

3.3 | Study of Margarine

3.3.1 | Physicochemical Properties Measurement

The physicochemical properties of the margarines UOM, ROM, and MC are summarized in Table 2. A significant difference (p < 0.05) was observed in the pH values of the three margarines, which ranged from 4.62 ± 0.01 to 5.10 ± 0.07 . Overall, these values fell within the standard range, typically set between 4 and 5.5. In contrast, no significant differences (p < 0.05) were observed in the acidity levels among the margarine samples, all of which remained within the recommended limit of 0.2% (Bentayeb Ait Lounis et al. 2018).

The moisture content of the two samples, UOM and ROM $(18.33\% \pm 0.07\% \text{ and } 18.25\% \pm 0.10\%, \text{ respectively}), \text{ was higher}$ than that of the margarine control (16.53%) and exceeded the ISO 662 (1998) standard of 16%. These differences may be attributed to the higher water retention capacity due to the emulsifying properties of the added date seed oil. Ouahrani, Casal, et al. (2022) demonstrated that the incorporation of Moringa oleifera leaves extract into margarine reduced water droplet size and improved emulsion stability. Moreover, Besbes et al. (2004) reported that date seed oil contains approximately 0.25% phospholipids, which may contribute to its emulsifying potential. A similar observation was reported by Kaanin-Boudraa et al. (2023) who found that the moisture content of the control margarine was 13.85%, while the margarines prepared with 100 and 50 ppm of date seed oil exhibited higher moisture contents of 15% and 15.06%, respectively.

TABLE 2 Physicochemical properties of margarines formulated with the unroasted and roasted date seed oil.

Parameter	UOM	ROM	MC
рН	4.62 ± 0.01°	4.75 ± 0.02^{b}	5.1 ± 0.07^{a}
Acidity (%)	0.19 ± 0.01^{a}	0.158 ± 0.003^{a}	0.149 ± 0.039^{a}
Moisture (%)	18.33 ± 0.07^{a}	18.25 ± 0.10^{a}	16.53 ± 0.11^{b}
Water activity (aw (–))	0.931 ± 0.01^{a}	0.949 ± 0.01^{b}	0.928 ± 0.02^a
Salt content (%)	0.41 ± 0.007^{a}	0.40 ± 0.010^{a}	0.38 ± 0.009^{b}
Melting point (°C)	$37.80 \pm 0.03^{\circ}$	38.70 ± 0.06^{a}	38.40 ± 0.05^{b}
Temperature (°C)		Solid fat content (%)	
5	51.70 ± 0.29^{ab}	51.20 ± 0.51^{a}	51 ± 0.14^{b}
10	43.90 ± 0.22^{a}	43.60 ± 0.43^{a}	44.10 ± 0.14^{a}
15	34.70 ± 0.14^{a}	34.60 ± 0.29^a	35 ± 0.22^a
20	24.50 ± 0.14^{b}	24.50 ± 0.11^{b}	24.9 ± 0.29^{a}
25	16.10 ± 0.17^{b}	16.40 ± 0.15^{a}	16.4 ± 0.04^{a}
30	10.30 ± 0.06^{b}	10.20 ± 0.11^{b}	10.60 ± 0.03^{a}
35	5.10 ± 0.06^{b}	5.50 ± 0.07^{a}	5.20 ± 0.04^{b}
40	2.80 ± 0.02^{b}	2.90 ± 0.04^{a}	2.80 ± 0.01^{b}

Note: Means with different superscript letters in the same row within a sample type are significantly different according to LSD test (p < 0.05). Abbreviations: MC, Margarine control; ROM, Margarine with roasted date seed oil; UOM, Margarine with unroasted date seed oil.

The water activity of UOM (0.931 ± 0.01) was not significantly different from that of the margarine control (0.928 ± 0.02) , whereas the water activity of ROM was slightly higher at 0.949 ± 0.01 . In most types of food, when water activity exceeds 0.950, bacteria become the predominant microbial flora. Below this threshold, yeast and molds are primarily responsible for biological alterations (Fernandez-Salguero et al. 1993).

The salt content of the developed margarines ranged from 0.38% to 0.41%. Regulations limit the salt content of margarine and butter to 2% (Pietinen et al. 2008). The results of this study are consistent with industry standards, which specify an acceptable salt content between 0.3% and 0.4%.

The melting point of the margarines varied from $37.38^{\circ}C \pm 0.03^{\circ}C$ to $38.7^{\circ}C \pm 0.06^{\circ}C$, which is consistent with the standard values set by the company for bakery margarine, typically ranging from $36^{\circ}C$ to $42^{\circ}C$.

Table 2 presents the solid fat content (SFC) profile of the developed margarines formulated with unroasted seed oil (UOM) and roasted seed oil (ROM), as well as the control margarine (MC). SFC at different temperatures affects the mouthfeel and spreadability of margarine. At 10°C, it determines the firmness of the margarine when refrigerated, while at 20°C, it significantly influences oil separation; maintaining a solid fat content of 10% at this temperature effectively prevents oil exudation. The cold sensation experienced when fat crystals dissolve in the mouth depends on the difference between the SFC values at 15°C and 25°C. At 35°C, SFC influences the release of aroma and flavor in the mouth (Dollah et al. 2020).

The values obtained for ROM and UOM, compared to commercial margarine, were similar and decreased with increasing

temperature, ranging from approximately 51% at 5°C to less than 3% at 40°C. These values fall within the range recommended by the standard for bakery margarine: 45%–51% at 10°C, 27%–31% at 20°C, 11%–18% at 30°C, and 2%–6% at 40°C. Similar results were reported by Sahri and Idris (2010) for bakery margarine, who suggested an SFC range of 47%–60% at 5°C, 38%–50% at 10°C, and 19%–26% at 20°C.

The measured and calculated color parameters of the date seed oil and margarine samples are shown in Table 3. The lightness (L*) and yellowness (b*) of the oil color decreased significantly after roasting, likely due to the formation of Maillard reaction products, which caused the oil to darken. No significant change was observed in the a* value. Similar color changes have been reported for microwave-roasted flaxseed oil (Suri et al. 2020). Regarding the margarine samples, the L* value showed no significant differences among the various margarines. The a* values were consistently negative, indicating a greenish hue, and ranged from -0.96 ± 0.01 to -2.05 ± 0.01 , with the highest value recorded for the ROM sample. The positive b* value (yellow color) of the margarine made with roasted oil was similar to that of the control margarine. In contrast, the margarine made with unroasted seed oil showed a significantly higher b* value (14.23 ± 0.02) due to the higher b* value of the unroasted oil (p < 0.05).

Roasted date seed oil exhibited the highest color saturation (C*) and hue (h*) values. The total color difference (ΔE) between the oil samples was easily noticeable to the human eye, with a value of 7.20. The hue angle values for the margarines showed a dominant yellow spectral component (angle of 90°), with no significant differences between ROM and MC. However, UOM exhibited a higher hue angle (h°). A similar

TABLE 3 | Color parameters of date seed oil and the formulated margarines.

	L*	a*	b*	C*	h°	ΔΕ
Unroasted	2.36 ± 0.23^{a}	2.36 ± 0.1^{a}	5.77 ± 0.16^{a}	3.00 ± 0.057^{b}	23.37 ± 0.38 ^b	
Roasted	-3.69 ± 0.07^{b}	2.38 ± 0.06^{a}	1.85 ± 0.03^{b}	6.23 ± 0.185^{a}	73.73 ± 0.99^a	
Unroasted/Roasted						7.20
Margarine						
UOM	91.33 ± 0.01^{a}	-0.96 ± 0.01^{c}	14.23 ± 0.02^{a}	14.27 ± 0.02^a	93.89 ± 0.03^{b}	
ROM	89.81 ± 0.03^{a}	-2.05 ± 0.01^{a}	12.44 ± 0.01^{b}	12.60 ± 0.01^{b}	99.37 ± 0.04^{a}	
MC	91.06 ± 0.01^{a}	-1.78 ± 0.00^{b}	12.30 ± 0.02^{b}	12.43 ± 0.02^{b}	98.24 ± 0.02^{a}	
UOM/MC						2.11
ROM/MC						1.28
UOM/ROM						2.58

Note: Means with different superscript letters in the same row within a sample type are significantly different according to LSD test (p < 0.05).

Abbreviations: IT: induction time measured by Rancimat test at 98°C; MC, Margarine control; ROM, Margarine with roasted date seed oil; UOM, Margarine with unroasted date seed oil.

TABLE 4 | Textural parameters and induction time of margarines formulated with the unroasted and roasted date seed oil.

Margarines	IT (h)	Hardness (N)	Cohesiveness (-)	Adhesiveness (N)	Springiness (mm)
UOM	24.60 ± 0.22^{c}	4.93 ± 0.15^{b}	0.01 ± 0.01^{a}	0.05 ± 0.04^{b}	0.25 ± 0.16^{b}
ROM	31 ± 0.43^{a}	5.41 ± 0.98^{a}	0.04 ± 0.04^{a}	0.22 ± 0.25^{a}	0.12 ± 0.10^{c}
MC	28.29 ± 0.33^{b}	5.51 ± 1.55^{a}	0.02 ± 0.01^a	0.21 ± 0.11^{a}	0.34 ± 0.11^{a}

Note: Means with different superscript letters in the same row within a sample type are significantly different according to LSD test (p < 0.05).

Abbreviations: IT: induction time measured by Rancimat test at 98°C; MC, Margarine control; ROM, Margarine with roasted date seed oil; UOM, Margarine with unroasted date seed oil

trend was observed in the chroma (C^*) results. In terms of total color difference (ΔE), the margarine formulated with roasted seed oil (ROM) was closer to the control margarine, with a lower value of 1.28, compared to the margarine made with unroasted seed oil (UOM) (2.11). The color difference between ROM and UOM was 2.58.

The results of the texture parameter measurements are presented in Table 4. The findings revealed that the hardness and adhesiveness values of margarine made with unroasted seed oil were significantly lower (p<0.05) than those of the control margarine, while the values for margarine made with roasted seed oil showed no significant difference from the control. This may be attributed to the similar solid fat content (SFC) of ROM and MC observed at 25°C, as SFC is related to fat hardness at this temperature (Devi and Khatkar 2017). In contrast, the lower SFC of UMO may explain its reduced hardness.

A previous study indicated that hardness values ranged from 12.08 to 45.63 N, while adhesiveness values ranged from 2.01 to 3.16 N in solid margarine, with a strong positive correlation observed between the two parameters (Ergönül 2013). Jeyarani et al. (2015) reported a lower hardness value of 1.74 N for bakery margarine made with mango kernel fat, compared to our study. They also found that the hardness increased to 14.57 N when the margarine was prepared without palm oil, suggesting that palm oil content affects hardness.

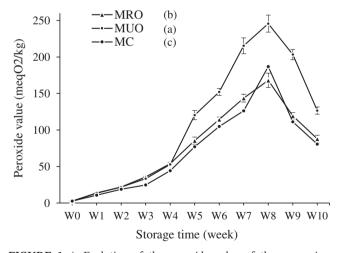


FIGURE 1 | Evolution of the peroxide value of the margarines during storage. MC, Margarine control; ROM, Margarine with roasted date seed oil; UOM, Margarine with unroasted date seed oil.

No differences were observed in the cohesiveness parameter across all samples. Regarding the springiness parameter, MC exhibited the highest value (0.34mm), followed by UOM (0.25mm), and ROM (0.12mm). This variation is likely due to differences in moisture content and water activity levels in these samples. A study on the texture properties of gluten-free biscuit formulations using TPA observed that higher moisture content

and water activity led to softer textures and lower springiness values (Gerzhova et al. 2016).

3.3.2 | Oxidative Stability of Margarines

The results showing the effect of roasted and unroasted date seed oils, as well as the synthetic antioxidant TBHQ, on oxidative stability, as determined by the Rancimat test, are presented in Table 4. The induction time (IT) for margarine with added roasted date seed oil was significantly higher (p < 0.05) than that for margarine with added unroasted date seed oil (31 h vs. 24.6 h, respectively). The unroasted date seed oil exhibited the highest sensitivity to oxidation among the tested samples. This

may be attributed to its insufficient antioxidant content, which does not match the high antioxidant potential of TBHQ. A similar observation was reported by Taha et al. (2019), who found that when unroasted date seed oil was added to soybean oil at a concentration of 50 mg/kg, it did not enhance oxidative stability to the same extent as BHT. Comparable or superior antioxidant effects were only observed at higher concentrations (100 and 150 mg/kg). Although unroasted date seed oil contains polyphenols, their antioxidant activity may be less effective in stabilizing lipids against oxidation than the higher polyphenol content and stronger antioxidant capacity of roasted date seed oil. The IT value of ROM was slightly higher than that of MC, suggesting that roasted date seed oil is as effective as synthetic antioxidants in stabilizing margarine against oxidative deterioration.

TABLE 5 | Fatty acids composition of margarines formulated with the unroasted and roasted date seed oil before and after storage at 60°C.

Fatty acids (%)	Storage (weeks)	UOM	ROM	MC
Caprylic acid (C8:0)	W_0	0.5 ± 0.02^{bB}	0.45 ± 0.025^{cB}	0.57 ± 0.001^{aA}
	W_1	0.535 ± 0.023^{aB}	0.47 ± 0.003^{bB}	0.51 ± 0.013^{aB}
	W_{10}	0.633 ± 0.003^{aA}	$0.551 \pm 0.009^{\text{bA}}$	$0.453 \pm 0.002^{\text{cC}}$
Capric acid	W_0	0.44 ± 0.01^{aA}	0.37 ± 0.02^{bB}	0.38 ± 0.02^{bA}
(C10: 0)	\mathbf{W}_1	0.429 ± 0.002^{aA}	0.398 ± 0.045^{aA}	0.376 ± 0.025^{aA}
	W_{10}	0.438 ± 0.002^{aA}	$0.407 \pm 0.005^{\text{bA}}$	0.361 ± 0.001^{cA}
Lauric acid	W_0	5.12 ± 0.02^{bB}	5.97 ± 0.035^{aA}	4.88 ± 0.125^{cB}
(C12:0)	W_1	5.17 ± 0.031^{bB}	$5.07 \pm 0.033^{\text{cC}}$	5.45 ± 0.01^{aA}
	W_{10}	6.403 ± 0.061^{aA}	5.246 ± 0.093^{bB}	4.395 ± 0.068^{cC}
Myristic acid	W_0	2.64 ± 0.165^{aB}	2.72 ± 0.015^{aA}	2.33 ± 0.005^{bB}
(C14:0)	W_1	2.48 ± 0.044^{aB}	2.48 ± 0.011^{aB}	$2.41\pm0.03^{\mathrm{bA}}$
	W_{10}	3.257 ± 0.057^{aA}	$2.721 \pm 0.015^{\text{bA}}$	$2.067 \pm 0.013^{\text{cC}}$
Palmitic acid	W_0	$35.92 \pm 0.01^{\mathrm{bB}}$	36.21 ± 0.095^{abC}	36.34 ± 0.28^{aB}
(C16:0)	W_1	36.69 ± 0.409^{aB}	36.61 ± 0.097^{aB}	36.44 ± 1.15^{aB}
	W_{10}	49.137 ± 1.309^{aA}	$41.705 \pm 0.178^{\mathrm{bA}}$	$40.223 \pm 2.249^{\text{bA}}$
Stearic acid (C18:0)	W_0	4.7 ± 0.3^{aB}	4.26 ± 0.005^{bC}	$4.41\pm0.01^{\rm abB}$
	W_1	4.73 ± 0.110^{aB}	4.42 ± 0.026^{bB}	4.33 ± 0.03^{bB}
	W_{10}	6.575 ± 0.21^{aA}	5.939 ± 0.037^{aA}	6.201 ± 0.552^{aA}
Oleic acid	W_0	$32.225 \pm 0.055^{\text{bA}}$	31.48 ± 0.315^{cA}	32.64 ± 0.04^{aA}
(C18:1)	W_1	$31.39 \pm 0.100^{\text{bA}}$	$30.74 \pm 0.414^{\text{cB}}$	31.99 ± 0.04^{aB}
	W_{10}	$28.563 \pm 1.01^{\text{bB}}$	30.595 ± 0.214^{aB}	31.767 ± 0.453^{aB}
Linoleic acid (C18:2)	W_0	17.375 ± 0.125^{cA}	$17.86 \pm 0.065^{\text{bA}}$	18.57 ± 0.18^{aA}
	W_1	$16.90 \pm 0.014^{\rm cB}$	17.46 ± 0.066 ^{bB}	18.17 ± 0.32^{aB}
	W_{10}	1.069 ± 0.034^{cC}	10.29 ± 0.178^{bC}	12.578 ± 1.501^{aC}
Linoleic/palmitic	W_0	0.484 ± 0.004^{cA}	$0.493 \pm 0.00^{\mathrm{bA}}$	0.511 ± 0.001^{aA}
(18:2/16:0)	W_1	0.460 ± 0.006^{cB}	$0.476 \pm 0.003^{\text{bB}}$	0.498 ± 0.007^{aA}
	W_{10}	$0.022 \pm 0.0001^{\text{cC}}$	0.247 ± 0.003^{bC}	0.313 ± 0.055^{aB}

Note: Means with different superscript lowercase letters in the same row within a sample type are significantly different according to the LSD test (p < 0.05, a > b > c). and Storage period results (0, 1, and 10 weeks) in the same column for each parameter with different uppercase letters are statistically different according to the LSD test (p < 0.05, A > B > C).

Abbreviations: MC, Margarine control; ROM, Margarine with roasted date seed oil; UOM, Margarine with unroasted date seed oil.

The oxidative stability of food emulsions can be assessed through accelerated studies conducted at elevated temperatures, using the peroxide value (PV) as a highly sensitive indicator of the initial stage of lipid oxidation (Jacobsen 2016). The oxidative stability of margarine, determined using the Schaal oven test (PV at 60° C \pm 1° C) over a 10-week storage period, is illustrated in Figure 1. At the initial stage (W0), the addition of unroasted and roasted date seed oil did not significantly alter the PV of the margarine. All margarine samples remained below the 10 meqO₂/kg limit set by the company's internal standard and by Codex Alimentarius Commission (1999). During storage, a steady increase in PV was observed across all margarine samples during the first 4weeks, with minimal differences between the formulations. However, from the fifth week onward, significant differences emerged. The unroasted date seed oil margarine (UOM) exhibited the highest PV, rising sharply from 52.88 ± 2.11 to 120.38 ± 6.26 meqO₂/kg, indicating lower oxidative stability. This trend was consistent with its short induction time, as demonstrated by the Rancimat test. Conversely, the roasted date seed oil margarine (ROM) increased from 53.94±1.89 to $85.5 \pm 4.88 \text{ meqO}_2/\text{kg}$, following a pattern similar to the control margarine (MC), which increased from 44.23 ± 1.56 to 77 ± 2.22 meqO₂/kg. After 8 weeks of storage, the PVs peaked at 177.98 meg O_2/kg for ROM, 186.87 meq O_2/kg for MC, and 245.67 meq O_2/kg for UOM.

Beyond this period, the PVs of all samples began to decline, likely due to the decomposition of primary oxidation products into secondary compounds such as aldehydes, ketones, alcohols, hydrocarbons, volatile organic acids, and epoxy compounds (Shahidi and Zhong 2010). Similarly, Stefani Juncos et al. (2024) studied the impact of various essential oils and synthetic antioxidants on sunflower oil under accelerated oxidation conditions (60°C for 28 days). They observed very high PV, reaching 240 and 150 $\rm meqO_2/kg$ in the oil enriched with essential oils and that enriched with BHT, respectively, and reaching 300 $\rm meqO_2/kg$ in the control oil, whose values decreased after 21 days.

3.3.3 | Effect of Thermal Treatment on the Fatty Acids' Composition of Margarines

Assessing fatty acid composition is an effective method for determining the oxidative state of oil. The fatty acid profiles of margarines at the beginning and after storage at 60°C are presented in Table 5. Accelerated storage at 60°C is an appropriate method for estimating shelf life at 25°C, with 1 day at 60°C being equivalent to 8.79 days at 25°C (Lopez et al. 2022). The data indicate that the incorporation of date seed oil samples caused only slight changes in the fatty acid composition, likely due to the low incorporation rate (1%). Palmitic acid (C16:0) was the most abundant saturated fatty acid, comprising 35.92%–36.34% of the total fatty acids. Small amounts of caprylic acid (C8:0), capric acid (C10:0), lauric acid (C12:0), myristic acid (C14:0), and stearic acid (C18:0) were also detected. Additionally, a significant amount of the monounsaturated fatty acid oleic acid was observed, ranging from 31.48% to 32.64%, alongside the polyunsaturated fatty acid linoleic acid, which ranged from 17.37% to 18.57%. These results were comparable to the composition of hard Serbian margarine (Vucic et al. 2015). Storage of margarines at accelerated temperatures resulted in a significant decrease (p < 0.05) in linoleic acid due to its susceptibility to oxidation, leading to a corresponding increase in saturated

fatty acids, particularly palmitic acid. Consequently, the ratio of linoleic acid to palmitic acid (C18:2/C16:0) is considered a valuable indicator of oil deterioration (Yilmaz and Yorulmaz 2023). After 1 week of storage, which is approximately equivalent to 2 months at room temperature, only slight losses in linoleic acid were observed: 2.73% in UOM, 2.23% in ROM, and 2.15% in the control margarine (CM). The corresponding C18:2/C16:0 ratios were 4.95%, 3.44%, and 2.54% for UOM, ROM, and CM, respectively. This agrees with the results of Azizkhani and Zandi (2009), who found that some margarine formulations enriched with natural antioxidant combinations were as effective as TBHQ at the beginning of storage. However, differences became more noticeable with longer storage. By week 10, thermal degradation of linoleic acid became especially severe in the UOM sample, which exhibited a 93.87% loss, compared to 41.43% in ROM. In contrast, the margarine fortified with TBHQ showed only a 24.17% reduction. The C18:2/C16:0 ratio in the UOM sample declined by 95.85%, indicating extensive oil deterioration. Meanwhile, the ROM sample maintained a ratio comparable to the control, with degradation percentages of 49.29% and 35.42%, respectively. These findings align well with those reported by Ouahrani, Tzompa-Sosa, et al. (2022), who reported that the addition of Moringa oleifera leaf extract to margarine enhanced oxidative stability and preserved the C18:2/C16:0 ratio, reducing degradation compared to unenriched margarine.

4 | Conclusion

In this study, bakery margarine was selected to evaluate the quality improvement potential obtained by its fortification with roasted and unroasted date seed oil, in comparison to enrichment with TBHQ. Roasting significantly increased the phenolic content and improved the antioxidant properties of the oil. The stability was assessed by induction time (IT) and peroxide value (PV) during accelerated storage, as well as by the evolution of the fatty acid profile after heat exposure. As a result, bakery margarine enriched with roasted date seed oil exhibited greater oxidative stability than that enriched with unroasted oil. Compared with commercial margarine enriched with TBHQ, margarine formulated with roasted date seed oil demonstrated comparable oxidative stability while retaining its physicochemical properties. These findings suggest that roasted date seed oil is a promising natural alternative to TBHQ for preserving quality and extending the shelf life of margarine. Future research should explore the impact of oil extraction methods, particularly mechanical pressing versus Soxhlet extraction, on the oil's properties, along with a sensory evaluation of the formulated margarines to assess consumer acceptance and further validate the application of roasted date seed oil as a natural preservative in bakery products.

Author Contributions

Rahma Mayouf: investigation, methodology, formal analysis, writing original draft. Fatiha Hamitri-Guerfi: conceptualization, supervision. Aicha Benbouriche: formal analysis, statistical analysis. Mohamed Ali Ayadi: formal analysis. Samir Hadjal: data curation. Brahim Zeroual and Toufik Ouatmani: preparation of the samples. Morad Hamitri: fatty acid analysis. Lila Boulekbache-Makhlouf: supervision. Hamadi Attia: supervision. Imène Felfoul: supervision, validation, writing – review and editing.

Acknowledgments

The authors would like to thank the Cevital agri-food (Bejaia, Algeria) for providing ingredients for the margarine preparation.

Conflicts of Interest

The authors declare no conflicts of interest.

Data Availability Statement

Data will be available on request.

References

Abd-Elkarim, N. A., E. A. Abdrabou, and A.-K. Osman. 2024. "Influence of Roasting and Microwave Heating on Nutritional Value and Bioactive Compounds of Siwi Date Seed." *Egyptian Journal of Food Science* 52: 123–137. https://doi.org/10.21608/ejfs.2024.223982.1190.

Abid, Y., S. Azabou, M. Jridi, I. Khemakhem, M. Bouaziz, and H. Attia. 2017. "Storage Stability of Traditional Tunisian Butter Enriched With Antioxidant Extract From Tomato Processing By-Products." *Food Chemistry* 233: 476–482. https://doi.org/10.1016/j.foodchem.2017.04.125.

Akinoso, R., E. O. Adegoroye, and M. S. Sanusi. 2023. "Effects of Roasting on Physicochemical Properties and Fatty Acids Composition of Okra Seed Oil." *Measurement: Food* 9: 100076. https://doi.org/10.1016/j.meafoo.2023.100076.

Ali, R. F. M., and A. M. El-Anany. 2025. "Physico-Chemical Characteristics, Nutritional Quality, and Oxidative Stability of Cold-Pressed Chia Seed Oil, Date Palm Seed Oil, Along With Their Binary Mixtures." *Cogent Food & Agriculture* 11, no. 1: 2466061. https://doi.org/10.1080/23311932.2025.2466061.

Ammar, I., H. Gharsallah, A. Ben Brahim, et al. 2021. "Optimization of Gluten-Free Sponge Cake Fortified With Whey Protein Concentrate Using Mixture Design Methodology." *Food Chemistry* 343: 128457. https://doi.org/10.1016/j.foodchem.2020.128457.

Azizkhani, M., and P. Zandi. 2009. "Effects of Some Natural Antioxidants Mixtures on Margarine Stability." *International Journal of Food Engineering* 3: 53–56.

Basuny, A. M. M., and M. A. Al-Marzooq. 2011. "Production of Mayonnaise From Date Pit Oil." *Food and Nutrition Sciences* 2: 938–943. https://doi.org/10.4236/fns.2011.29128.

Belcadi-Haloui, R., A. Zekhnini, Y. El-Alem, and A. Hatimi. 2018. "Effects of Roasting Temperature and Time on the Chemical Composition of Argan Oil." *International Journal of Food Science* 2018: 7683041. https://doi.org/10.1155/2018/7683041.

Benbouriche, A., H. Haddadi-Guemghar, M. Bachir-Bey, et al. 2022. "Improvement of Thermo-Resistance and Quality of Soybean Oil by Blending With Cold-Pressed Oils Using Simplex Lattice Mixture Design." Oilseeds and Fats, Crops and Lipids 29: 33. https://doi.org/10.1051/ocl/2022025.

Benbouriche, A., H. Haddadi-Guemghar, D. Mehidi-Terki, L. Boulekbache-Makhlouf, and M. Bachir-Bey. 2023. "Enrichment of Soybean Oil With Carotenoids From Chili Paste By-Product: Enhancement of Quality, Oxidative Stability, and Thermo-Resistance." *Journal of Food Measurement and Characterization* 18: 1107–1120. https://doi.org/10.1007/s11694-023-02239-2.

Bentayeb Ait Lounis, S., L. Mekimène, D. Mazi, et al. 2018. "Nutritional Quality and Safety of Algerian Margarines: Fatty Acid Composition, Oxidative Stability and Physicochemical Properties." *Mediterranean Journal of Nutrition and Metabolism* 11: 331–342. https://doi.org/10.3233/MNM-18208.

Besbes, S., C. Blecker, C. Deroanne, G. Lognay, N. E. Drira, and H. Attia. 2004. "Quality Characteristics and Oxidative Stability of Date Seed Oil

During Storage." Food Science and Technology International 10: 333–338. https://doi.org/10.1177/1082013204047777.

Bolini, H. M. A., A. C. Medeiros, C. T. M. Pereira, et al. 2023. "Consumer Acceptance Studies of Margarine to Guide Product Development in the Food Industry." *Food* 13: 116. https://doi.org/10.3390/foods13010116.

Chbani, M., B. Matthaus, Z. Charrouf, et al. 2020. "Characterization of Phenolic Compounds Extracted From Cold Pressed Cactus (*Opuntia ficus-indica* L.) Seed Oil and the Effect of Roasting on Their Composition." *Food* 9: 1098. https://doi.org/10.3390/foods9081098.

Chen, J., Y. Zhao, R. Wu, et al. 2023. "Changes in the Quality of High-Oleic Sunflower Oil During the Frying of Shrimp (*Litopenaeus Vannamei*)." Food 12: 1332. https://doi.org/10.3390/foods12061332.

Cheong, L. Z., C. P. Tan, K. Long, M. Suria Affandi Yusoff, and O. M. Lai. 2009. "Physicochemical, Textural and Viscoelastic Properties of Palm Diacylglycerol Bakery Margarine During Storage." *Journal of the American Oil Chemists' Society* 86: 723–731.

Ciou, J.-Y., H.-C. Chen, C.-W. Chen, and K.-M. Yang. 2021. "Relationship Between Antioxidant Components and Oxidative Stability of Peanut Oils as Affected by Roasting Temperatures." *Agriculture* 11: 300. https://doi.org/10.3390/agriculture11040300.

Codex Alimentarius Commission. 1999. Codex Standards for Fats and Oils From Vegetable Sources. Codex Standard for Named Vegetable Oils. Codex Alimentarius Commission (CODEX-STAN 210–1999).

Das, M., and A. Das. 2024. "A Comprehensive Review on Strategies for Replacing Saturated Fats in Bakery Products." *Discover Food* 4: 156. https://doi.org/10.1007/s44187-024-00240-2.

Devi, A., and B. Khatkar. 2017. "Thermo-Physical Properties of Fats and Oils." *International Journal of Engineering Research & Technology* 7: 45.

Dollah, S., K. F. Chai, S. M. Abdulkarim, and H. M. Ghazali. 2020. "Comparative Study of Table Margarine Prepared From *Moringa oleifera* Seed Oil-Palm Stearin Blend and Commercial Margarines: Composition, Thermal, and Textural Properties." *European Journal of Lipid Science and Technology* 122: 1900428. https://doi.org/10.1002/ejlt. 201900428.

El Harkaoui, S., K. N'diaye, S. Gharby, et al. 2024. "Insights Into Date Seed Oil Composition: Geographical Variability and Potential Applications." *European Journal of Lipid Science and Technology* 126: e2400061. https://doi.org/10.1002/ejlt.202400061.

Ergönül, P. G. 2013. "Solid Fat Contents and Instrumental Textural Attributes of Margarines Sold in Turkish Market." *Quality Assurance & Safety of Crops and Food* 5, no. 2: 157–161. https://doi.org/10.3920/QAS2012.0122.

Farhoosh, R. 2007. "The Effect of Operational Parameters of the Rancimat Method on the Determination of the Oxidative Stability Measures and Shelf-Life Prediction of Soybean Oil." *Journal of the American Oil Chemists' Society* 84: 205–209. https://doi.org/10.1007/s11746-006-1030-4.

Fernandez-Salguero, J., R. Gomez, and M. Carmona. 1993. "Analysis Water Activity in Selected High-Moisture Foods." *Journal of Food Composition and Analysis* 6: 364–369. https://doi.org/10.1006/jfca. 1993.1040.

Fikry, M., Y. Aniza Yusof, A. M. Al-Awaadh, et al. 2019. "Kinetics Modelling of the Colour, Hardness, Grinding Energy Consumption and Oil Yield Changes During the Conventional Roasting of Palm Date Seeds." *Food Science and Technology Research* 25: 351–362. https://doi.org/10.3136/fstr.25.351.

Fruehwirth, S., S. Egger, D. Kurzbach, et al. 2021. "Ingredient-Dependent Extent of Lipid Oxidation in Margarine." *Antioxidants* 10: 105. https://doi.org/10.3390/antiox10010105.

Gao, P., Y. Cao, R. Liu, Q. Jin, and X. Wang. 2018. "Phytochemical Content, Minor-Constituent Compositions, and Antioxidant Capacity of Screw-Pressed Walnut Oil Obtained From Roasted Kernels." *European*

Journal of Lipid Science and Technology 121, no. 1: 1800292. https://doi.org/10.1002/ejlt.201800292.

Gerzhova, A., M. Mondor, M. Benali, M. Aider, and Technology. 2016. "Incorporation of Canola Proteins Extracted by Electroactivated Solutions in Gluten-Free Biscuit Formulation of Rice-Buckwheat Flour Blend: Assessment of Quality Characteristics and Textural Properties of the Product." *International Journal of Food Sciences and Nutrition* 51: 814–827. https://doi.org/10.1111/jifs.13034.

Ghelichi, S., M. Hajfathalian, B. Yesiltas, A. M. Sorensen, P. J. Garcia-Moreno, and C. Jacobsen. 2023. "Oxidation and Oxidative Stability in Emulsions." *Comprehensive Reviews in Food Science and Food Safety* 22: 1864–1901. https://doi.org/10.1111/1541-4337.13134.

Golshan Tafti, A., N. Solaimani Dahdivan, and S. Yasini Ardakani. 2017. "Palm Stearin as Low Trans Hard Stock for Margarine." *International Food Research Journal* 24: 821–827.

Harkat, H., R. Bousba, C. Benincasa, et al. 2022. "Assessment of Biochemical Composition and Antioxidant Properties of Algerian Date Palm (*Phoenix Dactylifera* L.) Seed Oil." *Plants (Basel)* 11: 381. https://doi.org/10.3390/plants11030381.

ISO-15648. 2004. Fats and Butter-Determination of Salt Content. ISO.

ISO-3960. 2007. Animal and Vegetable Fats and Oils-Determination of Peroxide Value. ISO.

ISO-5509. 2000. Animal and Vegetable Fats and Oils-Preparation of Methyl Esters of Fatty Acids. ISO.

ISO-6321. 2002. Animal and Vegetable Fats and Oils-Determination of Melting Point in Open Capillary Tube (Slip Point). ISO.

ISO-660. 2009. Animal and Vegetable Fats and Oils-Determination of Acid Value and Acidity. ISO.

ISO-662. 1998. Animal and Vegetable Fats and Oils. Determination of Moisture Content and Volatile Matter. ISO.

ISO-6886. 2006. Animal and Vegetable Fats and Oils-Determination of Oxidation Stability (Accelerated Oxidation Test). ISO.

ISO-8292-1. 2008. Animal and Vegetable Fats and Oils-Determination of Solid Fat Content by Pulsed NMR Part 1-Direct Method. ISO.

Jacobsen, C. 2016. "Oxidative Stability and Shelf Life of Food Emulsions." In *Oxidative Stability and Shelf Life of Foods Containing Oils and Fats*, 287–312. Elsevier Inc.

Jeyarani, T., S. Subramanian, R. Sneha, M. Sudha, and P. Negi. 2015. "Characterisation of Mango Kernel Fat and Preparation of Tran's-Free Margarine for Use in Muffins." *Journal of Nutrition & Food Sciences* 5: 357. https://doi.org/10.4172/2155-9600.1000357.

Kaanin-Boudraa, G., F. Hamitri-Guerfi, L. Harfi, et al. 2023. "Physicochemical Characterization and Antioxidant Capacity of the Extracted Oil From Date Pits and Its Effect on Storage Stability of Margarine." North African Journal of Food and Nutritional Research 7: 54–67. https://doi.org/10.51745/najfnr.7.16.54-67.

Karrar, E., I. A. Mohamed Ahmed, M. F. Manzoor, et al. 2021. "Effect of Roasting Pretreatment on Fatty Acids, Oxidative Stability, Tocopherols, and Antioxidant Activity of Gurum Seeds Oil." *Biocatalysis and Agricultural Biotechnology* 34: 102022. https://doi.org/10.1016/j.bcab. 2021.102022.

Liu, C., J. Li, Y. Bi, X. Wang, S. Sun, and G. Yang. 2016. "Thermal Losses of Tertiary Butylhydroquinone (TBHQ) and Its Effect on the Qualities of Palm Oil." *Journal of Oleo Science* 65: 739–748. https://doi.org/10.5650/jos.ess16041.

Lopez, P. L., M. A. Marchesino, N. R. Grosso, and R. H. Olmedo. 2022. "Comparative Study of Accelerated Assays for Determination of Equivalent Days in the Shelf Life of Roasted High Oleic Peanuts: Chemical and Volatile Oxidation Indicators in Accelerated and Room

Temperature Conditions." Food Chemistry 373: 131479. https://doi.org/10.1016/j.foodchem.2021.131479.

López, P. L., N. S. Juncos, N. R. Grosso, and R. H. Olmedo. 2022. "Minthostachys Mollis Essential Oil and Its Combination With Tert-Butylhydroquinone for Control of Lipid Oxidation." *European Journal of Lipid Science and Technology* 124, no. 11: 2200081. https://doi.org/10.1002/eilt.202200081.

Miskandar, M. S., Y. C. Man, M. S. A. Yusoff, and R. A. Rahman. 2005. "Quality of Margarine: Fats Selection and Processing Parameters." *Asia Pacific Journal of Clinical Nutrition* 14: 387–395.

Mohamed Ahmed, I. A., M. Musa Özcan, N. Uslu, et al. 2020. "Effect of Microwave Roasting on Color, Total Phenol, Antioxidant Activity, Fatty Acid Composition, Tocopherol, and Chemical Composition of Sesame Seed and Oils Obtained From Different Countries." *Journal of Food Processing & Preservation* 44: e14807. https://doi.org/10.1111/jfpp.14807.

Mohd Jaih, A., R. A. Rahman, A. Razis, A. Ariffin, A. Al-Awaadh, and N. Suleiman. 2019. "Fatty Acid, Triacylglycerol Composition and Antioxidant Properties of Date Seed Oil." *International Food Research Journal* 26: 517–527. https://doi.org/10.13140/RG.2.2.28516.22406.

Moure, A., J. M. Cruz, D. Franco, et al. 2001. "Natural Antioxidants From Residual Sources." *Food Chemistry* 72: 145–171. https://doi.org/10.1016/S0308-8146(00)00223-5.

Nenadis, N., A. Mastralexi, M. Z. Tsimidou, et al. 2018. "Toward a Harmonized and Standardized Protocol for the Determination of Total Hydroxytyrosol and Tyrosol Content in Virgin Olive Oil (VOO). Extraction Solvent." *European Journal of Lipid Science and Technology* 120: 1800099. https://doi.org/10.1002/ejlt.201800099.

Ouahrani, S., S. Casal, M. Bachir-Bey, and F. Zaidi. 2022. "Impact of Moringa oleifera Leaves Extract in the Stabilization of Margarine Under Accelerated Storage." *Journal of Food Measurement and Characterization* 17: 1455–1466. https://doi.org/10.1007/s11694-022-01714-6.

Ouahrani, S., D. A. Tzompa-Sosa, K. Dewettinck, and F. Zaidi. 2022. "Oxidative Stability, Structural, and Textural Properties of Margarine Enriched With *Moringa Oleifera* Leaves Extract." *Journal of the American Oil Chemists' Society* 99: 485–499. https://doi.org/10.1002/aocs.12586.

Pădureţ, S. 2022. "The Quantification of Fatty Acids, Color, and Textural Properties of Locally Produced Bakery Margarine." *Applied Sciences* 12: 1731. https://doi.org/10.3390/app12031731.

Palanna, M., M. Itagi, L. K. Sannegowda, et al. 2025. "A Bio-Mimicking Cobalt Tetramenthol-Substituted Phthalocyanine-Based Electrochemical Sensor for Selective and Sensitive Detection of Tert-Butylhydroquinone." *Journal of Materials Chemistry B* 13: 4188–4200. https://doi.org/10.1039/d4tb02043j.

Paranthaman, R., P. P. Kumar, and S. Kumaravel. 2012. "HPLC and HPTLC Determination of Caffeine in Raw and Roasted Date Seeds (*Phoenix Dactylifera* L)." *Scientific Reports* 1: 1–4. https://doi.org/10.4172/scientificreports.249.

Pietinen, P., L. M. Valsta, T. Hirvonen, and H. Sinkko. 2008. "Labelling the Salt Content in Foods: A Useful Tool in Reducing Sodium Intake in Finland." *Public Health Nutrition* 11: 335–340. https://doi.org/10.1017/S1368980007000249.

Potočnik, T., M. Rak Cizej, and I. J. Košir. 2018. "Influence of Seed Roasting on Pumpkin Seed Oil Tocopherols, Phenolics and Antiradical Activity." *Journal of Food Composition and Analysis* 69: 7–12. https://doi.org/10.1016/j.jfca.2018.01.020.

Rakhshanda, N., M. Mehmood, K. Mahmood, A. Sajjad, S. Munir, and N. Akbar. 2020. "Physiochemical Properties and Total Phenolic Content of Date Palm (*Phoenix Sylvestris* (L.) Roxb) Seed Oil Grown in District Panjgur, Pakistan." *International Journal of Biosciences* 16, no. 2: 272–278. https://doi.org/10.12692/ijb/16.2.272-278.

Rice-Evans, C., N. Miller, and G. J. T. I. P. S. Paganga. 1997. "Antioxidant Properties of Phenolic Compounds." *Trends in Plant Science* 2: 152–159.

Sahri, M. M., and N. A. Idris. 2010. "Palm Stearin as Low Trans Hard Stock for Margarine." *Sains Malaysiana* 39: 821–827.

Samet-Bali, O., M. Ayadi, and H. Attia. 2009. "Traditional Tunisian Butter: Physicochemical and Microbial Characteristics and Storage Stability of the Oil Fraction." *LWT – Food Science and Technology* 42: 899–905. https://doi.org/10.1016/j.lwt.2008.11.007.

Shahidi, F., and Y. J. C. S. R. Zhong. 2010. "Lipid Oxidation and Improving the Oxidative Stability." *Chemical Society Reviews* 39: 4067–4079. https://doi.org/10.1039/B922183M.

Stefani Juncos, N., C. Florencia Cravero, N. Rubén Grosso, and O. R. Horacio. 2024. "Integral Oxidation Value Used as a New Oxidation Indicator for Evaluation of Advanced Stages of Oxidative Processes: Intox Value." *Microchemical Journal* 204: 111186. https://doi.org/10.1016/j.microc.2024.111186.

Subhash, A. J., G. B. Bamigbade, and M. Ayyash. 2024. "Current Insights Into Date By-Product Valorization for Sustainable Food Industries and Technology." *Sustainable Food Technology* 2, no. 2: 331–361. https://doi.org/10.1039/D3FB00224A.

Suri, K., B. Singh, and A. Kaur. 2022. "Impact of Microwave Roasting on Physicochemical Properties, Maillard Reaction Products, Antioxidant Activity and Oxidative Stability of Nigella Seed (*Nigella Sativa L.*) oil." *Food Chemistry* 368: 130777. https://doi.org/10.1016/j.foodchem.2021. 130777.

Suri, K., B. Singh, A. Kaur, M. P. Yadav, and N. Singh. 2020. "Influence of Microwave Roasting on Chemical Composition, Oxidative Stability and Fatty Acid Composition of Flaxseed (*Linum Usitatissimum L.*) oil." *Food Chemistry* 326: 126974. https://doi.org/10.1016/j.foodchem.2020. 126974.

Taha, E., N. Abd-Elkarim, and Z. Ahmed. 2019. "Date Seed Oil as a Potential Natural Additive to Improve Oxidative Stability of Edible Vegetable Oils." *Egyptian Journal of Food Science* 47: 105–113. https://doi.org/10.21608/ejfs.2019.13050.1007.

United States Food and Drug Administration. 2024. Code of Federal Regulations, Title 21, Part 172. U.S. Government Publishing Office.

Vucic, V., A. Arsic, S. Petrovic, S. Milanovic, M. Gurinovic, and M. Glibetic. 2015. "Trans Fatty Acid Content in Serbian Margarines: Urgent Need for Legislative Changes and Consumer Information." *Food Chemistry* 185: 437–440. https://doi.org/10.1016/j.foodchem.2015.04.018.

Vujasinovic, V., S. Djilas, E. Dimic, Z. Basic, and O. Radocaj. 2012. "The Effect of Roasting on the Chemical Composition and Oxidative Stability of Pumpkin Oil." *European Journal of Lipid Science and Technology* 114: 568–574. https://doi.org/10.1002/ejlt.201100158.

Wannes, W. A., and B. Marzouk. 2016. "Characterization of Myrtle Seed (*Myrtus communis* Var. Baetica) as a Source of Lipids, Phenolics, and Antioxidant Activities." *Journal of Food and Drug Analysis* 24: 316–323. https://doi.org/10.1016/j.jfda.2015.11.001.

Yao, X.-H., Y.-S. Shen, R.-Z. Hu, et al. 2020. "The Antioxidant Activity and Composition of the Seed Oil of Mulberry Cultivars." *Food Bioscience* 37: 100709. https://doi.org/10.1016/j.fbio.2020.100709.

Yilmaz, E. G., and A. Yorulmaz. 2023. "Improvement in Frying Stability of Safflower Oil by Blending With Refined Olive Pomace Oil During Deep Fat Frying." *Journal of Oleo Science* 72: 901–910. https://doi.org/10.5650/jos.ess23016.