Grasas y Aceites 76 (3)
July-September 2025, 2319
ISSN-L: 0017-3495, eISSN: 1988-4214
https://doi.org/10.3989/gya.0434251.2319

Characterization of oleogels containing olive oil, beeswax, and orange essential oil

N. Yaman[a,b,✉], and Ö. Özdestan Ocak[b]

[a]General Directorate of Agricultural Research and Policies, Aegean Agricultural Research Institute, Quality and Technology Laboratory, 35660 Menemen, Izmir, Türkiye
[b]Faculty of Engineering, Department of Food Engineering, Ege University, 35040 Bornova, Izmir, Türkiye
✉Corresponding author: nihal.yaman@tarimorman.gov.tr

Submitted: 22 April 2025; Accepted: 18 December 2025; Published: 23 March 2026

SUMMARY: Oleogels are structured oil systems proposed as alternatives to trans and saturated fats. This study aimed to optimize an olive-oil-based beeswax oleogel enriched with orange essential oil (OEO) in order to improve oil binding capacity, textural characteristics, and oxidative stability. Using a Box–Behnken design, the optimal formulation (11.40% BW, 80 °C heating temperature, 20 °C cooling temperature) exhibited high oil binding (99.58%), improved firmness and cohesiveness, and a low peroxide value (12.81 meq O_2/kg). Rheological and DSC analyses confirmed a stable, thermo-reversible crystalline network. Notably, D-limonene exhibited high volatile stability during storage, indicating that the BW crystalline matrix effectively retained functional components and supported oxidative resistance. These findings demonstrate the potential of the optimized oleogel as a healthier lipid phase and a carrier for volatile bioactive compounds. Future studies should evaluate its performance in real food applications.

KEY-WORDS: *Box-Behnken design; Essential oil; Oil binding capacity; Oxidative stability; Texture analysis; Thermal analysis.*

RESUMEN: ***Caracterización de oleogeles conteniendo aceite de oliva, cera de abejas y aceite esencial de naranja.*** Los oleogeles son sistemas oleosos estructurados que se proponen como alternativas a las grasas *trans* y saturadas. Este estudio tuvo como objetivo optimizar un oleogel de cera de abeja a base de aceite de oliva, enriquecido con aceite esencial de naranja (AEO), para mejorar la capacidad de unión al aceite, las características texturales y la estabilidad oxidativa. Mediante un diseño de Box-Behnken, la formulación óptima (11,40 % BW, 80 °C de temperatura de calentamiento y 20 °C de temperatura de enfriamiento) mostró una alta capacidad de unión al aceite (99,58 %), mejor firmeza y cohesión, y un bajo índice de peróxidos (12,81 meq O_2/kg). Los análisis reológicos y de DSC confirmaron una red cristalina estable y termorreversible. Cabe destacar que el D-limoneno mostró una alta estabilidad volátil durante el almacenamiento, lo que indica que la matriz cristalina de BW retuvo eficazmente los componentes funcionales y favoreció la resistencia oxidativa. Estos hallazgos demuestran el potencial del oleogel optimizado como una fase lipídica más saludable y un vehículo para bioactivos volátiles. Estudios futuros deberían evaluar su rendimiento en aplicaciones alimentarias reales.

PALABRAS CLAVE: *Aceite esencial; Análisis térmico; Análisis de textura; Capacidad de unión de aceites; Diseño de Box-Behnken; Estabilidad oxidativa.*

Citation/Cómo citar este artículo: Yaman N, Özdestan Ocak Ö. 2025. Characterization of Oleogels Containing Olive Oil, Beeswax, and Orange Essential Oil. *Grasas Aceites* **76** (3), 2319. https://doi.org/10.3989/gya.0434251.2319

1. INTRODUCTION

Fats are not only energy sources but also key structural components that affect the texture, stability, and flavor release of foods. To obtain solid fat functionality, liquid oils are often converted into semi-solid systems for applications such as margarines, spreads, bakery products, and confectionery. However, conventional structuring methods frequently increase trans and saturated fat contents, which are associated with adverse health effects. Oleogelation has therefore attracted growing interest as a strategy to structure liquid oils without altering their chemical composition, thereby providing healthier lipid phases (Manzoor *et al.,* 2022).

Incorporating lipophilic bioactive components, particularly essential oils (EOs), into oleogels is a promising approach to improving oxidative stability and adding functionality. EOs can delay lipid oxidation during processing and storage and are considered natural alternatives to synthetic antioxidants such as BHA and BHT. Orange essential oil (OEO), which is rich in limonene, exhibits antioxidant, antimicrobial, and antifungal activities and has been proposed as a natural preservative, especially in bakery applications (Razola-Díaz *et al.,* 2021; da Silva *et al.,* 2023).

Beeswax (BW), classified as a low-molecular-weight oleogelator and determined to be "Generally Recognized as Safe" (GRAS) in the food industry, is widely employed in food applications owing to its strong gelation capacity, wide availability, and low cost (Puşcaş *et al.*, 2020). BW readily forms a stable three-dimensional crystalline network even at low incorporation levels and exhibits desirable technological functionalities such as stabilization, plasticization, and film-forming ability. Its natural origin, biodegradability, and favorable processing properties further position BW as a sustainable and effective structurant in oleogel systems. However, potential limitations including shear sensitivity and possible sensory alterations should also be considered (Guo *et al.*, 2023).

Previous studies have described oleogels containing essential oils prepared with different oils and oleogelators, mainly focusing on textural, rheological, or oxidative properties (Pinto *et al.*, 2021; da Silva *et al.*, 2023; Oyom *et al.*, 2024). However, there is limited information on the multi-response optimization of such systems and on the storage stability of volatile bioactive compounds such as D-limonene within the oleogel matrix. Yet, for applications as functional fat phases, both structural stability and the retention of bioactive compounds during storage are crucial. Our study provides a novel contribution to the literature by simultaneously investigating oleogel properties and the storage stability of D-limonene within the oleogel matrix.

This study introduces an optimized oleogel system enriched with OEO and prepared through direct oleogelation. Unlike previous studies, it provides a comprehensive evaluation of an optimized oleogel system and examines changes in volatile compound content during storage. Accordingly, the study addresses both the development of an optimized essential-oil-enriched oleogel system and the stability of its functional components during storage. The formulation exhibited promising characteristics as a functional lipid matrix which is comparable to margarine.

2. MATERIALS AND METHODS

2.1. Materials

The olive oil (OO) and beeswax (BW) used in oleogel preparation were obtained from the Aegean Agricultural Research Institute (Turkey), while the orange essential oil (OEO) was commercially sourced. Sodium carbonate, sodium acetate trihydrate, and sodium thiosulfate were purchased from TEKKİM (Turkey); chloroform, glacial acetic acid, potassium iodide, starch, ethanol, potassium persulfate, n-hexane, Folin–Ciocalteu reagent, and gallic acid were obtained from Merck (Germany). Methanol was supplied by ISOLAB (Germany), and 2,2-azino-bis (3-ethylbenzothiazoline-6-sulfonic acid) (ABTS), 2,2-diphenyl-1-picrylhydrazyl (DPPH), and Trolox (6-hydroxy-2,5,7,8-tetramethylchroman-2-carboxylic acid) were purchased from Sigma-Aldrich (USA).

2.2. Methods

2.2.1. Experimental design and statistical analysis

Response Surface Methodology (RSM) enables the identification of optimal experimental parameters and facilitates the development of mathematical models to evaluate interaction effects among variables with a reduced number of experiments. For this reason, the Box–Behnken design (BBD) of RSM was employed to investigate and optimize the effects of beeswax (BW) concentration, heating temperature (T_H), and cooling temperature (T_C) — the key parameters governing oleogel formation.

The selected beeswax concentration range (7–13 %) aligns with previously reported effective gelation intervals in the literature (Pușcaș *et al.*, 2020; Pinto *et al.*, 2021; Çokay and Öğütçü, 2023; Çokay *et al.*, 2024) and was further supported by preliminary trials, in which this range yielded the most stable gel formation. The structural integrity of oleogels depends not only on the chemical composition of the wax and oil but also on temperature and crystallization conditions. Accordingly, T_H was chosen as an independent variable due to its role in ensuring complete wax dissolution and regulating network formation during cooling; while T_C was selected because of its strong influence on crystal morphology and the resulting physicochemical and structural properties of oleogels (Thakur *et al.*, 2022; Zhang *et al.*, 2024). As cooling temperature decreases, crystallization kinetics accelerate, producing smaller and denser crystals, which in turn affect the textural attributes of the oleogel. Literature reports indicate that lower cooling temperatures promote faster nucleation of wax crystals, leading to smaller but more compact crystal aggregates that reinforce the three-dimensional network. Similarly, another study showed that low storage temperatures (5–10 °C) in carnauba wax oleogels increased crystal packing density within the gel matrix, resulting in tighter structures and enhanced oil-binding capacity (Thakur *et al.*, 2022). Cooling rate and environmental temperature during oleogel preparation have been identified as major determinants of final textural strength and gel stability, with oleogels cooled at lower temperatures exhibiting more elastic and coherent network structures (Giacomozzi *et al.*, 2018). In light of this evidence, the cooling temperatures applied in the present study (4, 12, and 20 °C) were expected to influence wax crystal size, density, and interaction patterns, and consequently affect gel stability, mechanical strength, and oil-binding capacity.

Based on preliminary trials, a total of 15 experimental runs with three replicates at the center point were established, and the levels of the independent variables are presented in Table 1. The variable ranges were selected according to prior preliminary evaluations and relevant literature (Çokay and Öğütçü, 2023; Oyom *et al.*, 2024). The objective of the optimization was to minimize peroxide value (PV) while maximizing oil-binding capacity (OBC), firmness, and cohesiveness. The design matrix was generated using Design-Expert 13.0 (Table 1).

TABLE 1. Levels of independent variables, processing parameters and response variables for oleogels formulated with beeswax, olive oil and orange essential oil

Independent variable			Unit	Levels			
				(-1)	(0)	(1)	
Oleogelator concentration (BW)			%	7	10	13	%
Heating temperature (T_H)			(°C)	80	90	100	(°C)
Cooling temperature (T_C)			(°C)	4	12	20	(°C)
Run	BW (%)	T_H (°C)	T_C (°C)	OBC (%)	Firmness (g)	Cohesiveness (g)	PV (meq O_2/kg)
1	13	90	4	99.88	2346.15	-1374.81	12.08
2	13	90	20	99.88	3534.18	-1962.66	12.19
3	10	80	20	99.17	1566.85	-960.6	13.86
4	7	90	4	97.83	358.07	-299.23	14.19
5	10	100	4	99.84	1189.73	-729.4	14.42
6	10	100	20	99.11	1901.71	-1012.79	14.78
7	7	90	20	96.87	593.76	-407.28	15.2
8	7	80	12	97.35	425.75	-347.33	14.95
9	10	90	12	99.25	1239.06	-855.16	14.13
10	13	80	12	99.84	2400.46	-1394.11	11.96
11	13	100	12	99.83	2699.29	-1499.96	13.24
12	7	100	12	98.13	413.63	-320.25	15.27
13	10	90	12	99.84	1306.82	-823.84	14.18
14	10	80	4	99.86	1065.87	-718.9	13.27
15	10	90	12	99.33	1265.01	-805.41	14.21

OBC: Oil binding capacity, PV: Peroxide value, BW: Beeswax, T_H: Heating temperature, T_C: Cooling temperature

Preparation of oleogels. Olive oil was weighed into a 250-mL beaker and heated to 80 °C, 90 °C, or 100 °C on a magnetic stirrer (Heidolph MR-Hei, Germany), ensuring that the target temperature was not exceeded. Pre-determined amounts of beeswax were then added, and the mixture was stirred at 600 rpm for 15 minutes until a clear solution was obtained. Subsequently, OEO (0.5%) was incorporated. The mixtures were poured into containers and kept at 4 °C, 12 °C, or 20 °C for 24 hours to allow gel formation under the respective experimental conditions. The oleogels were stored at 4 °C throughout the storage period (0, 30, 60, and 90 days) until analysis (Thakur *et al.*, 2022).

The optimized oleogel was prepared in the same manner, using the beeswax concentration and the heating (T_H) and cooling (T_C) temperatures identified through the optimization study.

Oil binding capacity. Oleogels were melted in a 90 °C water bath for 30 minutes, and 1 mL of the melted sample was transferred into pre-weighed Eppendorf tubes (a). The tubes were stored at 4 °C for 1 hour and then reweighed (b). Samples were centrifuged (Hettich Universal 320 R) at 10 000 rpm for 15 minutes at 20 °C, inverted, and left undisturbed for another 15 minutes. The separated oil was removed, tubes were reweighed (c), and OBC% was calculated using Equation (1) (Yılmaz *et al.*, 2021).

$$OBC\% = 100 - \left[\frac{(b-a)-(c-a)}{(b-a)} x100\right] \quad (1)$$

Texture profile analysis. A texture analyzer (TA-XT Plus, Stable Microsystems Ltd., UK) with a 35 mm compression probe and 30 kg load cell was used. Oleogels were placed in cylindrical containers (55 mm × 70 mm) and subjected to a single compression cycle at 1 mm/s over a 30 mm distance. Firmness (maximum force) and cohesiveness (area under the negative force-time curve) were recorded (Yılmaz and Öğütçü, 2014).

Peroxide value. The PVs of the oleogels prepared for optimization, as well as that of the optimal oleogel, were determined using the acetic acid/chloroform solution method. The optimal oleogel was stored at 4 °C for 90 days, with evaluations conducted every 30 days.

2.2.2. Optimization and validation of models

The effects of independent variables on OBC, textural properties, and PV were analyzed using quadratic interaction models, evaluated by the F (Fisher) test at a 95% confidence level. Statistically insignificant parameters were excluded. Model fit was assessed through analysis of variance (ANOVA) using statistical parameters including the coefficient of determination (R^2), F-value, adjusted regression coefficient (Adj-R^2), predicted residual sum of squares (PRESS), lack of fit (LOF), predicted R^2 (Pre-R^2), and adequate precision (AP). Three-dimensional (3D) response surface plots were generated in Design-Expert 13 to visualize the effects of preparation conditions. Regression analysis was performed by fitting a second-degree polynomial equation. The desirability value (D), ranging from 0 (undesirable) to 1 (optimal), was used to identify optimum preparation conditions.

2.2.3. Determination of volatile compounds and quantification of D-limonene

OEO volatiles were analyzed by GC–MS (Agilent 8890 GC, 5977C MSD) on an HP Innowax column (60 m × 0.25 mm × 0.25 μm) with helium as carrier gas (0.8 mL/min). The oven program and MS conditions followed Özek *et al.* (2010).

D-limonene in OBO was quantified by GC–FID using calibration standards (84.2–1347.2 ppm). About 0.1 g of oleogel was extracted with n-hexane and treated with KOH in methanol under ultrasonication, then centrifuged and injected (Özek *et al.*, 2010). Measurements were taken at 0, 30, 60, and 90 days.

2.2.4. Characterization of oleogels

Rheological properties. The rheological properties of OBO and MRG were measured on a DHR-30 rheometer (TA Instruments, USA) using roughened parallel plates (60 mm diameter) at 25 °C. The linear viscoelastic region was identified by an amplitude sweep test conducted at 1 Hz and 25 °C. Within this region, frequency sweep tests were performed between 1–100 Hz under a constant strain of 0.1%. During the frequency sweep, the storage modulus (G′), loss modulus (G″), loss factor (tan δ), and complex viscosity (η) were recorded.

***Thermal properties*.** The thermal behaviors of the optimized oleogel were evaluated using a Differential Scanning Calorimeter (DSC; Q20, TA Instruments). Samples (5–8 mg) were sealed in aluminum pans. The program involved heating to 120 °C (10 °C/min), cooling to −30 °C (10 °C/min) with a 3-min isothermal hold, and reheating to 110 °C (5 °C/min). Onset and peak temperatures and enthalpies were calculated from thermograms using Universal V4.5A software (Yılmaz *et al.*, 2021).

X-ray diffraction (XRD). Polymorphic forms of the optimized oleogel were determined using a Rigaku Ultima IV XRD system (Rigaku, Japan). Samples were stored at 20 ± 3 °C overnight before measurement. Scans were conducted using Cu-Kα radiation (λ = 1.54056 Å, 40 kV, 40 mA) over 2θ = 2.0–50°, 0.02° step size, and 2°/min speed. Data were analyzed via Origin Pro 7.5 (Yılmaz *et al.*, 2021).

2.2.5. Physicochemical properties

Total phenolic compounds (TPC). In order to determine the total phenolic content (TPC), 1 g of oleogel was dissolved in 5 mL hexane and extracted with 5 mL methanol:water (80:20). The mixture was vortexed and centrifuged (3500 rpm, 10 min, 4 °C), and the supernatant was used for analysis. TPC was measured at 720 nm using Folin–Ciocalteu reagent, and the results were expressed as gallic acid equivalents (mg GAE/kg oil) based on a 0–50 mg/L calibration curve (Canpolat *et al.*, 2023).

***Antioxidant activity analysis*.** Antioxidant activity was assessed using ABTS and DPPH assays. Percent inhibition was calculated from absorbance values, and the results were expressed as μmol TEAC/kg based on Trolox calibration curves (Zhang *et al.*, 2023).

2.2.6. Statistical analysis

Texture, PV, and antioxidant activity data were reported as mean ± standard deviation (SD) based on triplicate analyses. Statistical analysis was performed using SPSS 25 (SPSS Inc., Chicago, IL, USA). One-

way analysis of variance (ANOVA) followed by Duncan's multiple range test. used to assess significant differences among samples ($p < 0.05$).

3. RESULTS AND DISCUSSION

3.1. Model fitting

In the RSM, particularly when applying the BBD, a second-order polynomial model is commonly recommended to describe the effects of experimental factors on response variables. The general form of this model is given in Equation (2):

$$y = \beta_0 + \sum_{i=1}^{k}\beta_i x_i + \sum_{i=1}^{k}\beta_{ii} x_i^2 + \sum_{i<j}\beta_{ij} x_i x_j \quad (2)$$

In this equation, y denotes the predicted response; β_0 is the constant term, β_i denotes the linear coefficient, β_{ii} represents the quadratic coefficient, β_{ij} corresponds to the interaction coefficient, and x_i is the coded factor level. Using this regression model, the optimum response can be predicted by calculating the derivatives of the model (Wang and Wan, 2009).

In the optimization of the BW oleogel enriched with OEO, all response variables OBC, firmness, cohesiveness, and PV were found to fit the second-order polynomial model significantly ($p < 0.0001$). The R^2 were 0.9670 for OBC, 0.9983 for firmness, 0.9989 for cohesiveness, and 0.9971 for PV, indicating excellent model accuracy. The AP values for all models ranged from 17.75 to 84.02, well above the acceptable threshold of 4. Moreover, the LOF p-values were insignificant ($p > 0.05$) for all responses, confirming the models' strong fit to the experimental data.

3.1.1. Oil binding capacity

OBC indicates the amount of oil retained within the oleogel matrix after centrifugation. As oleogels are designed to contain high oil content, high OBC values are essential to prevent oil leakage. ANOVA results for OBC are presented in Table 2, showing a significant model ($p < 0.0001$) with an insignificant LOF ($p > 0.05$), confirming its validity. The model yielded an R^2 of 0.9674, reflecting high predictive accuracy. The coded model equation is given in Equation (3):

$$\%OBC = +99.49 + 1.16A + 0.0862B - 0.2975C - 0.1975AB + 0.2400AC - 0.7845A^2 \quad (3)$$

Here, A, B, and C represent BW, T_H, and T_C, respectively; AB, BC and AC are interaction terms; A^2, B^2, and C^2 are squared terms. The OBC values ranged from 96.87 to 99.88% (Table 1), in agreement with previous studies (Thakur *et al.*, 2022; Çokay *et al.*, 2024). These results confirm that increasing BW concentration and lowering the cooling temperature enhance crystallization and network formation, thereby improving oil retention (Figure 1a). The increase in heating temperature initially exerts a positive effect on OBC due to enhanced wax dissolution and improved network formation at elevated BW levels (Thakur *et al.*, 2022). However, as the temperature exceeds a certain threshold (90 °C), a decline in OBC is observed. Experimental findings suggest that excessive thermal exposure may disrupt the crystalline structure or reduce gelation efficiency, ultimately leading to a decrease in OBC.

Table 2. The results of the variance analysis in optimized oleogel (a) Oil binding capacity (OBC) (b) Firmness (c) Cohesiveness (d) Peroxide value (PV)

Source (a)	Sum of Squares	df	Mean Square	F-value	p-value	Source (b)	Sum of Squares	df	Mean Square	F-value	p-value
Model	14.15	6	2.36	39.55	< 0.0001	**Model**	7.22	6	1.20	776.00	< 0.0001
A-BW	10.70	1	10.70	179.39	< 0.0001	A-BW	6.60	1	6.60	4261.60	< 0.0001
B-T_H	0.0595	1	0.0595	0.9982	0.3470	B-T_H	0.0192	1	0.0192	12.40	0.0078
C-T_C	0.7080	1	0.7080	11.88	0.0087	C-T_C	0.3915	1	0.3915	252.62	< 0.0001
AB	0.1560	1	0.1560	2.62	0.1444	AB	0.0053	1	0.0053	3.45	0.1004
AC	0.2304	1	0.2304	3.86	0.0849	A^2	0.1452	1	0.1452	93.69	< 0.0001
A^2	2.30	1	2.30	38.53	0.0003	C^2	0.0384	1	0.0384	24.75	0.0011
Residual	0.4770	8	0.0596			**Residual**	0.0124	8	0.0015		
LOF	0.2721	6	0.0453	0.4427	0.8143	LOF	0.0110	6	0.0018	2.54	0.3096
Pure Error	0.2049	2	0.1024			Pure Error	0.0014	2	0.0007		
Cor Total	14.62	14				**Cor Total**	7.23	14			
Std. Dev.	0.2442					**Std. Dev.**	0.0394				
Mean	99.07					**Mean**	7.09				
C.V. %	0.2465					**C.V. %**	0.5552				
R^2	0.9674					**R^2**	0.9983				
Adj. R^2	0.9429					**Adj. R^2**	0.9970				
Pre. R^2	0.8666					**Pre. R^2**	0.9916				
AP	17.7532					**AP**	84.0240				

BW: Beeswax; T_H: Heating temperature; T_C: Cooling temperature; LOF: Lack of fit; Adj-R^2: Adjusted coefficient of determination; R^2: Coefficient of determination; Pre-R^2: Predicted coefficient of determination; AP: Adequate precision.

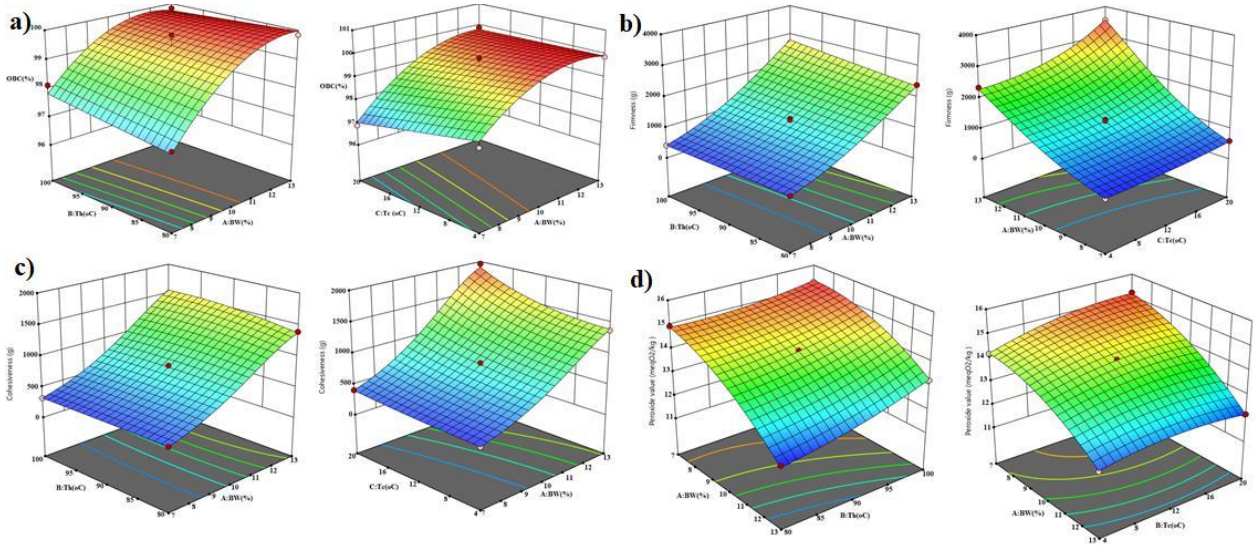

FIGURE 1. Response surface plots showing the effects of beeswax (BW) concentration, heating temperature (T_H) and cooling temperature (T_C) on oleogel properties: a) Oil binding capacity (OBC), b) Firmness, c) Cohesiveness, d) Peroxide value (PV)

3.1.2. Firmness

The analysis of variance (ANOVA) results for firmness are presented in Table 2. The model was statistically significant ($p < 0.0001$), with an insignificant LOF ($p > 0.05$), and exhibited high accuracy ($R^2 = 0.9983$). The estimated model equation is given in Equation (4):

$$\text{In (Sertlik)} = +7.14 + 0.9086A + 0.0490B + 0.2212C + 0.0366AB - 0.1977A^2 + 0.1016C^2 \quad (4)$$

The firmness values ranged from 358.07 to 3534.18 g (Table 1). As can be seen in Figure 1b, increasing the BW concentration significantly enhanced firmness, while higher heating and cooling temperatures generally resulted in reduced firmness. Although higher T_H could theoretically improve wax solubility, excessive heat

might disrupt the microstructure or inhibit the formation of an efficient gel network. These findings are consistent with prior studies which reported that increased gelator content promotes crystal growth and network density, thereby improving structural integrity (Pang *et al.*, 2020).

3.1.3. Cohesiveness

The ANOVA results for cohesiveness (Table 2) confirmed that the model was statistically significant ($p < 0.0001$), with an insignificant LOF ($p > 0.05$). The high R^2 value (0.9989) indicated excellent model reliability. The regression model equation is presented in Equation (5):

$$1/\text{Sqrt}(\text{Cohes.-0.50}) = +0.0348 - 0.0144A + 0.0000B - 0.0029C - 0.0008AB + 0.0010AC + 0.0053A^2 + 0.0005B^2 - 0.0008C^2 \quad (5)$$

Cohesiveness values ranged from −299.23 to −1962.66 g (Table 1); higher absolute values denote stronger internal bonding. According to the response surface plots in Figure 1c, BW concentration was identified as the most influential parameter on cohesiveness.

Increasing BW content significantly enhanced cohesiveness by promoting a denser and more interconnected crystalline network, particularly at medium to high BW levels, whereas temperature effects were limited at low BW concentrations. Higher T_H reduced cohesiveness, suggesting a partial disruption of crystal organization, while lower T_C promoted faster nucleation and the formation of a more compact network. Accordingly, the highest cohesiveness values were obtained at high BW concentrations combined with moderate heating and low cooling temperatures (Pang *et al.*, 2020).

3.1.4. Peroxide value

The analysis of variance (ANOVA) results for PV are presented in Table 2. The model showed high significance ($p < 0.0001$), with an insignificant LOF ($p > 0.05$), confirming its reliability. The R^2 value of 0.9971 indicates a strong correlation between predicted and experimental results. The best-fitted regression equation is given in Equation (6):

$$PV = +14.17 - 1.27A + 0.4587B + 0.2587C + 0.2400AB - 0.2250AC - 0.4929A^2 + 0.1746B^2 - 0.2654C^2 \quad (6)$$

PV values ranged from 11.96 to 15.27 meq O_2/kg (Table 1). The highest PV occurred at 7% BW, 100 °C heating, and 12 °C cooling; the lowest was at 13% BW, 80 °C, and 12 °C. As shown in Figure 1d, PV exhibited a clear decreasing trend with increasing BW concentration, which can be attributed to the natural antioxidant components present in beeswax (Topal *et al.*, 2020) as well as the formation of a denser crystalline network which limits oxygen diffusion into the oil phase. In contrast, increases in both heating (T_H) and cooling temperatures (T_C) at a given BW level led to higher PV. This behavior is consistent with the well-known effect of elevated temperatures in accelerating lipid oxidation by facilitating thermal degradation and enhancing oxygen interaction within the oil matrix (Lim *et al.*, 2017). Overall, these findings indicate that combining high BW concentrations with moderate heating and low cooling temperatures is effective in minimizing oxidative deterioration in BW-based oleogels.

3.2. Multi-response optimization and model validation

To determine the optimum point, the responses were simultaneously optimized by targeting maximum OBC, firmness, and cohesiveness values, while minimizing PV. Based on these criteria, the optimum point and the corresponding desirability value (D) are presented in Table 3, and the condition with the highest D value was selected as the optimal formulation. Under these optimized conditions, an oleogel sample was prepared and analyzed to validate the predictive capability of the model. The optimum point, predicted response ranges, and experimental validation results are also provided in Table 3. The experimental values of OBC, firmness, cohesiveness, and PV showed strong agreement with the model predictions, confirming the successful performance of the multi-response optimization.

The optimal formulation obtained through the optimization process consisted of 11.40% BW, a heating temperature of 80 °C, and a cooling temperature of 20 °C. The oleogel prepared under these conditions was designated as the optimized oleogel (OBO) for subsequent analyses.

TABLE 3. Optimal points, desirability level obtained according to optimization result, estimated values of the optimum point and responses of the optimized oleogel (OBO)

Sample	BW (%)	T_H (°C)	T_C (°C)	OBC (%)	Firmness (g)	Cohesiveness (g)	PV (meq O_2/kg)	Desirability
OBO	11.40	80	20	99.68	2393.51	-1389.77	12.97	0.708
Responses		**Predicted average**		**95% CI low**		**95% CI high**		**Optimized Oleogel Responses**
OBC		99.68		99.29		100.07		99.58±0.04
Firmness		2393.51		2247.89		2548.57		2542.07±7.01
Cohesiveness		-1389.77		-1284.59		-1508.43		-1503.13±7.23
PV		12.97		12.80		13.14		12.81±0.03

OBC: Oil binding capacity, PV: Peroxide value, BW: Beeswax, T_H: Heating temperature, T_C: Cooling temperature. Each result represents the average of three tests.

3.3. Determination of volatile compounds and quantification of D-limonene

The volatile compounds in OEO were identified as follows: limonene (96.54%), myrcene (1.58%), sabinene (0.50%), α-pinene (0.36%), β-phellandrene (0.22%), δ-3-carene (0.20%), cis-limonene oxide (0.18%), linalool (0.16%), terpinolene (0.14%), and geranyl acetate (0.12%). Our findings align with previously published studies in the literature (Razola-Díaz *et al.*, 2021). The GC-MS chromatogram of orange essential oil is shown in Figure 2.

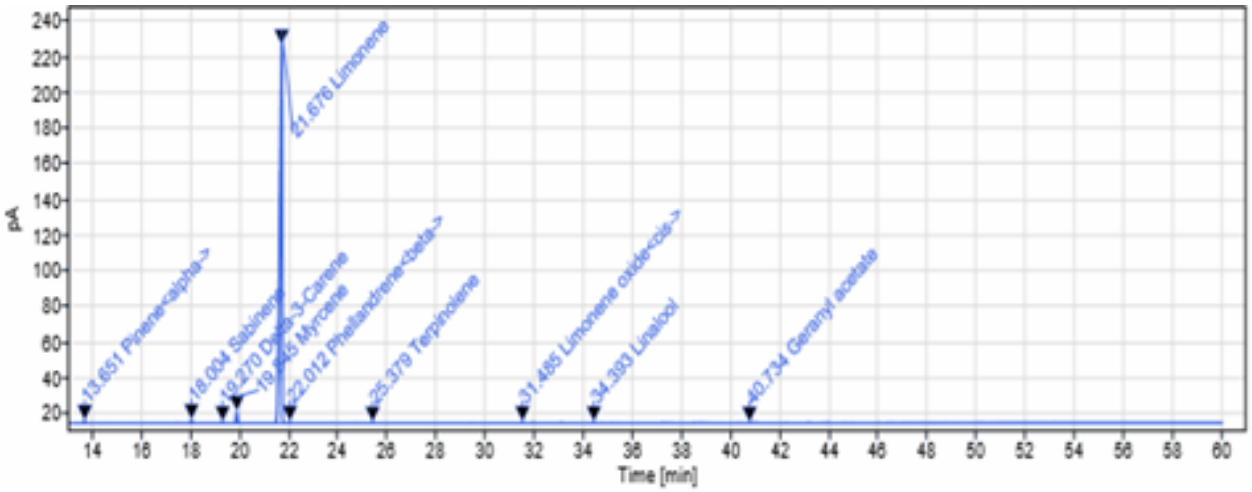

FIGURE 2. GC-MS chromatogram of orange essential oil

After OBO preparation, D-limonene content was monitored during storage (0–90 days). A statistically significant decrease was observed ($p < 0.05$), with concentrations declining from 26.36 to 23.49 mg/kg by day 90 (Table 4), corresponding to an approximately 9–10% loss. This reduction can be attributed to the volatile nature of limonene and its sensitivity to heat, oxygen, and light during processing and storage (Muhoza *et al.*, 2021). Nevertheless, the limited loss indicates that the three-dimensional oleogel network effectively entrapped volatile compounds, restricted oxygen diffusion, and enabled controlled release during storage. In addition, possible interactions between volatile phenolic constituents, particularly phenolic aldehydes, and the beeswax network may have further suppressed volatility and enhanced oxidative stability (Shang *et al.,* 2021; Zhu *et al.*, 2024). Overall, both structural integrity and chemical interactions within the oleogel matrix contributed to the improved retention and stability of D-limonene.

TABLE 4. Texture (firmness and cohesiveness), peroxide values (PVs), total phenolic compounds (TPC), DPPH and ABTS values for olive oil (OO), margarine (MRG), and the optimized oleogel (OBO).

	Firmness (g)				Cohesiveness (g)				PV (meq O_2/kg)			
Sample	**Day 0**	**Day 30**	**Day 60**	**Day 90**	**Day 0**	**Day 30**	**Day 60**	**Day 90**	**Day 0**	**Day 30**	**Day 60**	**Day 90**
OBO	2542.07±7.01^{aC*}	2616.22±16.9^{bC}	3136.6±78.4^{aB}	3840.94±22.6^{aA}	-1503.13±7.2^{bC}	-1751.17±40.5^{aB}	-1669.14±31.3^{bB}	-2131.28±47.9^{aA}	12.81±0.03^{aB}	12.72±0.13^{aB}	13.13±0.11^{aA}	13.19±0.07^{aA}
MRG	2848.98±119.03^{aA}	2814.64±58.6^{aA}	2846.48±91.4^{aA}	2891.66±54.5^{bA}	-2038.32±61.2^{aA}	-2093.21±88.9^{aA}	-2109.01±27.8^{aA}	-2084.1±15.2^{aA}	2.06±0.07^{cD}	2.31±0.064^{cC}	2.65±0.11^{cB}	2.93±0.06^{cA}
OO									8.84±0.1^{bC}	9.71±0.29^{bB}	10.25±0.17^{bA}	10.52±0.01^{bA}
	TPC (mg GAE/kg oil)				**DPPH** (µmol TEAC/kg)**				**ABTS** (µmol TEAC/kg)**			
Sample	**Day 0**	**Day 30**	**Day 60**	**Day 90**	**Day 0**	**Day 30**	**Day 60**	**Day 90**	**Day 0**	**Day 30**	**Day 60**	**Day 90**
OBO	104.9±1.56^{aA*}	101.6±2.69^{aA}	30±0.71^{aB}	29.4±0.14^{aB}	100.04±0.37^{bC}	136.72±0.41^{bB}	152.40±0.61^{aA}	54.91±0.74^{aD}	59.22±0.48^{aA}	29.77±0.12^{aB}	11.88±0.08^{aC}	6.18±0.04^{bD}
OO	40.6±0.28^{bA}	36.4±0.99^{bB}	30.4±0.14^{aC}	28.05±0.21^{bD}	169.30±0.33^{aA}	155.12±1.34^{aB}	61.69±0.46^{bC}	50.33±0.15^{bD}	62.02±0.16^{aA}	25.32±0.12^{bB}	11.99±0.16^{aC}	6.81±0.08^{aD}
	D-Limonene (mg/kg)											
Sample	**Day 0**	**Day 30**	**Day 60**	**Day 90**								
OBO	26.36±0.53^{aA}	26.16±0.64^{aA}	24.74±0.58^{aB}	23.49±0.55^{aC}								

*Each result represents the average of three tests. Different lowercase letters in the same column indicate significant differences among samples, while different uppercase letters in the same row indicate significant differences during storage for each sample ($p < 0.05$). Data are expressed as mean ± standard deviation (SD). Statistical significance was determined by one-way ANOVA and Duncan's multiple comparison test. **2,2-azino-bis (3-ethylbenzothiazoline-6-sulfonic acid) (ABTS), 2,2-diphenyl-1-picrylhydrazyl (DPPH)

3.4. Characterization of the optimized oleogel

3.4.1. Rheological properties

Rheological properties describe the response of oleogels to stress and deformation and their resistance to flow, which are critical for food applications. Oleogels form a three-dimensional crystalline network which governs their elasticity and shear sensitivity. Accordingly, G′ and G″ moduli obtained from frequency sweep tests reflect the elastic and viscous behavior of the oleogel.

G′ represents the elastic behavior of the system, with higher values indicating more elastic, stable, and structurally coherent networks, whereas lower values reflect more flowable and spreadable materials. In contrast, G″ describes the viscous component of the material. When G′ exceeds G″, gel-like behavior is observed. Accordingly, the G″/G′ ratio (tan δ) is commonly used as an indicator of structural robustness, with tan δ < 1 corresponding to firm, margarine-like gel structures (Chai *et al.*, 2022).

Complex viscosity (η) reflects the resistance of the oil phase to deformation and its processability during thermal treatment. Frequency sweep analysis showed that OBO exhibited a stronger structure than MRG, with G′ exceeding G″ and tan δ values below 1 across the entire frequency range, confirming gel-like behavior. OBO displayed higher G′, G″, and η values than MRG, indicating a more rigid, dense, and stable three-dimensional network. Moreover, OBO showed more elastic behavior at low frequencies and a greater viscous contribution at higher frequencies, thus reflecting a frequency-dependent viscoelastic profile (Figure 3a-c). Overall, these results indicate that OBO provides a lipid matrix comparable to margarine, but with enhanced structural robustness.

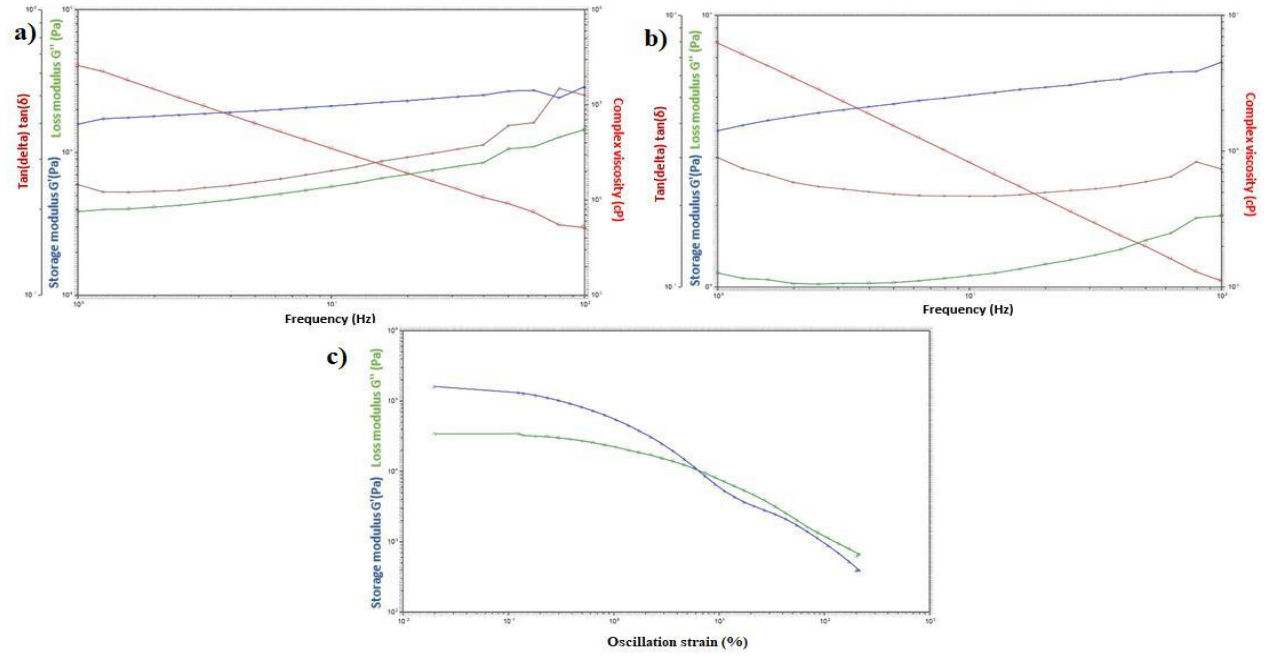

FIGURE 3. Rheological characterization of optimized oleogel (OBO) and margarine (MRG): **a)** Frequency sweep of OBO, **b)** Frequency sweep of MRG, **c)** Amplitude sweep (0.01–100% strain).

Overall, OBO shows strong potential as a fat-structuring alternative in a wide range of food applications, including bakery products, spreads, sauces, creams, fillings and coatings, and meat products.

3.4.2. X-ray diffraction

The polymorphic forms of fats (α, β, and β′) greatly influence structural attributes and spreadability. Among these, the β′ form is preferred for its smooth texture and pleasant mouthfeel (Chai *et al.*, 2022). The XRD

pattern of the OBO (Figure 4a) showed peaks at 4.12 Å and 3.70 Å, corresponding to the β′ polymorph, and a peak at 4.55 Å indicative of the β form. A peak at 21.46 Å was attributed to the lamellar spacing of triglyceride layers. The β′ form, associated with an orthorhombic perpendicular (O⊥) structure, is particularly favorable for margarine and other spreadable applications due to its uniform and creamy texture (Yılmaz *et al.*, 2021).

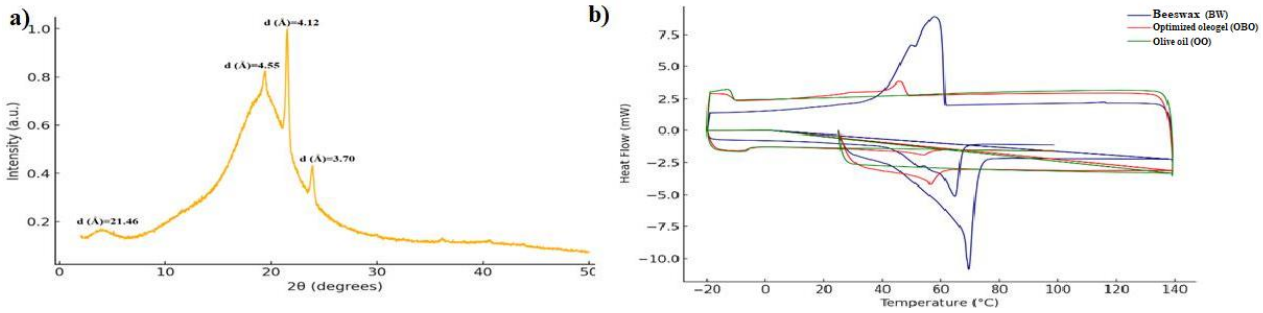

FIGURE 4. a) X-ray diffraction pattern of optimized oleogel (OBO), **b)** Differential scanning calorimeter thermogram of optimized oleogel (OBO), olive oil (OO) and beeswax (BW)

3.4.3. Thermal properties

DSC analysis was performed to evaluate the crystalline network structure and thermal behavior of the oleogel (Çokay and Öğütçü, 2023). Figure 4b presents the melting and crystallization thermograms of the OBO sample. The onset (T_{om}) and peak melting temperatures (T_{pm}) were 54.28 °C and 56.31 °C, respectively, which were higher than those of olive oil (OO), but lower than those of beeswax (BW). The melting enthalpy (ΔH_m) was 8.91 J/g, a value lower than both BW (181.8 J/g) and OO (23.45 J/g). The onset and peak crystallization temperatures (T_{oc} and T_{pc}) were 48.50 °C and 45.79 °C, respectively, while the crystallization enthalpy (ΔH_c) was 6.4 J/g. These relatively low enthalpy values indicate partial crystallization in the oil phase and dilution of the wax structure (Table 5).

TABLE 5. Thermal properties of beeswax (BW), olive oil (OO) and optimized oleogel (OBO)

Sample	Melting			Crystallization		
	T_{om} (°C)	T_{pm} (°C)	ΔH_m (J/g)	T_{oc} (°C)	T_{pc} (°C)	ΔH_c (J/g)
BW	65.96	69.55	181.8	61.43	57.82	180.1
OBO	54.28	56.31	8.91	48.5	45.79	6.4
OO	-5.32	-1.78	23.45	-21.67	-16.94	18.92

T_{om}: Onset melting T_{pm}: Peak melting ΔH_m: enthalpy of melting T_{oc}: Onset crystallization T_{pc}: Peak crystallization ΔH_c: enthalpy of crystallization

Although studies on the thermal properties of volatile oil–enriched oleogels are limited, comparable findings have been reported (Çokay and Öğütçü, 2023; Yılmaz and Öz, 2023). Some researchers have noted that essential oils may weaken the gel network and lower melting–crystallization temperatures (Çokay and Öğütçü, 2023). However, in the present study, the OEO level was as low as 0.5%, and its impact on the thermal transitions appeared minimal.

Yılmaz and Öğütçü (2014) reported the melting temperature of commercial solid fats to be 41.86 °C. The higher melting temperature of OBO indicates that the product remains stable at room temperature and melts more slowly in the mouth, resulting in a slightly waxy texture. Meanwhile, its crystallization temperatures support its thermo-reversible nature and demonstrate that OBO maintains structural stability across thermal cycles (Yılmaz and Öz, 2023).

3.4.4. Textural properties

Firmness and cohesiveness values for the OBO changed significantly during storage ($p < 0.05$), whereas MRG remained stable ($p > 0.05$) (Table 4). At each time point, significant differences were observed among

the samples. The oleogel retained a stable, plastic, and homogeneous structure over 90 days. These results are consistent with previous reports on BW-based oleogels using various oils (Lim *et al.*, 2017). Initially, the oleogel's firmness and cohesiveness were lower than MRG, but by day 60, firmness surpassed MRG, and by day 90, both values were the highest. This indicates that the oleogel developed a robust network over time. The storage period had a more pronounced effect on OBO than MRG. The increased firmness in OBO may result from microstructural changes such as crystal growth, and interactions between long-chain hydrocarbons and esters (Ojijo *et al.*, 2004).

3.5. Physicochemical properties

3.5.1. Peroxide values

During storage, a statistically significant increase ($p < 0.05$) in PV values was observed in OBO, MRG, and OO (Table 4). Similar increases have been reported in beeswax-based oleogels prepared with olive oil (Pang *et al.*, 2020). Among the samples, MRG exhibited the highest rate of increase (42.23%), whereas OBO showed only a 2.97% rise. This behavior can be attributed to the natural antioxidant compounds present in BW (Topal *et al.*, 2020) as well as the antioxidant activity of OEO (Razola-Díaz *et al.*, 2021; da Silva *et al.*, 2023). Previous studies have additionally demonstrated that higher BW concentrations contribute to lower PV by forming a denser network which restricts oxygen diffusion (Lim *et al.*, 2017).

Pang *et al.* (2020) observed a slow yet steady increase in PV over 90 days at 5 °C in wax-based oleogels prepared with different vegetable oils. Likewise, Lim *et al.* (2017) noted a consistent rise in PV as storage progressed. These findings collectively demonstrate that both time-dependent changes and structural differences among samples significantly influence this quality parameter.

At the beginning of the experiment, all oleogel samples exhibited higher PV values compared to olive oil and margarine. This was attributed to exposure of the oils to high temperatures during oleogel preparation, as well as processing-related conditions associated with production (Lim *et al.*, 2017).

3.5.2. Total phenolic compounds and antioxidant activity analysis

A significant decrease in total phenolic content (TPC) was observed during storage ($p < 0.05$), with a pronounced decline after day 60 (Table 4). OBO showed higher initial TPC than OO; however, its TPC decreased from 104.9 to 29.4 mg GAE kg^{-1} oil by day 90. This substantial reduction may be attributed to oxidative degradation, volatility, and the limited stability of phenolic compounds within the oleogel matrix. Although oleogels can partially protect phenolics by restricting oxygen exposure, high preparation temperatures and prolonged storage still promote losses (Shang *et al.*, 2021; Zhu *et al.*, 2024). Nevertheless, previous studies have reported that phenolic compounds generally remain at higher levels in oleogels than in liquid oils despite storage-related declines (Puşcaş *et al.*, 2020).

These reductions may also be associated with interactions between phenolic compounds and the oleogel matrix, leading to partial entrapment and reduced antioxidant availability. Although studies on TPC dynamics in oleogels during storage remain limited, previous findings indicate that phenolic–matrix interactions can reduce extractability and consequently lower the measured phenolic content (Gao *et al.*, 2024). Similarly, Puşcaş *et al.* (2020) reported phenolic losses of up to 50% after prolonged storage, attributing these reductions to structural instability and oxidative degradation. Therefore, phenolic stability in oleogels should be interpreted not only based on total content but also considering extractability, analytical methodology, and interactions between phenolics and the oleogel network.

Despite the decline in TPC, DPPH and ABTS activities showed distinct storage-dependent trends in OBO: DPPH increased until day 60 before decreasing, while ABTS steadily declined throughout storage (Table 4). The initial increase in DPPH activity may be attributed to the ability of the oleogel matrix to encapsulate volatile compounds within its crystalline network, thereby protecting them against oxidation and enabling controlled release (Pinto *et al.,* 2021; Çokay and Öğütçü, 2023). The continued rise in DPPH activity until day 60, despite decreasing phenolic content, suggests that phenolic degradation

products may retain antioxidant capacity and that matrix reorganization may enhance radical-scavenging activity over time. In contrast, ABTS values declined steadily from 59.22 to 6.18 μmol TEAC g^{-1} during storage, likely due to the higher sensitivity of the ABTS assay to hydrophilic antioxidants and the progressive loss of hydrophilic phenolics. While DPPH primarily reflects lipophilic antioxidant activity, ABTS responds to both hydrophilic and lipophilic compounds. Consistent with this, wax-based oleogel structures have been reported to improve the stability of lipophilic antioxidants (Xie *et al.*, 2014; Silva *et al.*, 2022).

Throughout storage, OBO exhibited higher antioxidant activity than OO. This behavior can be attributed to the presence of natural antioxidant compounds in beeswax (Topal *et al.*, 2020), the oxygen-barrier effect of the beeswax crystalline network which enhances oxidative stability, and the ability of the oleogel matrix to retain volatile antioxidants such as limonene, thereby reducing evaporative losses. In addition, the higher initial phenolic content in OBO likely contributed to its consistently greater antioxidant activity compared to OO. Although studies on antioxidant behavior in oleogels remain limited, enhanced radical-scavenging activity has been reported in systems containing essential oils (Silva *et al.*, 2022; Oyom *et al.*, 2024). Overall, oleogel systems provide an effective delivery matrix for the stabilization of volatile and lipophilic bioactive compounds against oxidation.

4. CONCLUSIONS

This study successfully developed and optimized a beeswax-structured olive oil oleogel enriched with orange essential oil, offering a promising alternative to saturated fat-based systems such as margarine. Response Surface Methodology identified beeswax concentration and processing temperatures as key parameters influencing oil binding capacity, textural attributes, and oxidative stability. The optimized formulation exhibited excellent oil retention, desirable firmness and cohesiveness, and a stable crystalline network.

Rheological analyses confirmed dominant elastic behavior ($G' > G''$) and $\tan \delta < 1$, indicating a well-structured, rigid gel matrix, while DSC results demonstrated thermo-reversible melting and recrystallization, ensuring structural integrity under temperature variations. The presence of the β' polymorph further supported its suitability for spreadable fat applications.

During storage, the oleogel maintained its structural stability and exhibited slower peroxide formation compared to olive oil and margarine. In particular, D-limonene showed high volatile stability, with a low degree of loss over 90 days, demonstrating that the beeswax crystalline network effectively entrapped volatile bioactive compounds and limited oxygen diffusion. This highlights the dual functionality of the system as both a structuring matrix and a carrier for volatile and antioxidant compounds.

Overall, the combination of olive oil, beeswax, and orange essential oil produced an oleogel with favorable physicochemical, structural, thermal, and oxidative properties. These results support its potential use in bakery products, spreads, coatings, and other applications requiring solid-fat functionality.

In addition, the findings underscore the need for further investigation into phenolic stabilization mechanisms within oleogel systems, particularly the interactions between wax networks, essential oil components, and phenolic compounds that govern long-term oxidative protection. Future studies should assess sensory properties, consumer acceptance, and real-food performance while exploring different essential oil types or concentrations to further tailor functional characteristics.

RESEARCH DATA POLICY

DATA AVAILABILITY

The data supporting the findings of this study are available from the corresponding author upon reasonable request.

ACKNOWLEDGMENTS

We would like to thank the Ege University Scientific Research Council for their financial support (Project number: FM-GAP-2023-31862). We would also like to express our gratitude to the General Directorate of Agricultural Research and Policies, Quality and Technology Laboratory of the Aegean Agricultural Research Institute for providing the facilities necessary for conducting this research.

DECLARATION OF COMPETING INTEREST

The authors declare that they do not have any conflict of interest.

FUNDING SOURCES

Ege University Scientific Research Council (Project number: FM-GAP-2023-31862).

AUTHORSHIP CONTRIBUTION STATEMENT

N. Yaman: Writing-original draft, Methodology, Conceptualization, Investigation, Formal analysis, Writing–review & editing, Validation;

Ö. Özdestan Ocak: Supervision, Writing -review & editing, Methodology, Formal analysis, Validation.

REFERENCES

Canpolat K, Bilenler Koç T, Karabulut İ. 2023. Physicochemical properties of oleogels produced using wheat germ oil and natural waxes. *Firat Univ. J. Eng. Sci.* **35** (2), 647-656. https://doi.org/10.35234/fumbd.1247374

Chai X, Zhang Y, Shi Y, Liu Y. 2022. Crystallization and structural properties of oleogel-based margarine. *Molecules,* **27,** 8952. https://doi.org/10.3390/molecules27248952

Çokay H, Öğütçü M. 2023. Determining the structure and stability of essential oil-sunflower wax and beeswax oleogels. *J. Am. Oil Chem. Soc.* **100** (12). https://doi.org/10.1002/aocs.12745

Çokay H, Çelebi Uzkuç NM, Yüceer YK, Öğütçü M. 2024. Comparative analysis of essential oil oleogels containing beeswax and sunflower wax with petrolatum gels. *Eur. J. Lipid Sci. Technol*. **126** (5). https://doi.org/10.1002/ejlt.202300055

da Silva FT, dos Santos FN, Fonseca LM, de Souza EJD, dos Santos Hackbart HC, da Silva KG, Biduski B, Gandra EA, Dias ARG, da Rosa Zavareze E. 2023. Oleogels based on germinated and non-germinated wheat starches and orange essential oil: application as a hydrogenated vegetable fat replacement in bread. *Int. J. Biol. Macromol.* **253**. https://doi.org/10.1016/j.ijbiomac.2023.126610

Gao P, Liu Y, Wang S, Huang C, Zhong W, Yin J, Hu C, He D, Wang X. 2024. Effects of different oleogelators on the structural properties and composition of iron walnut-oil oleogels. *Ultrason. Sonochem*. **102**, 106729. https://doi.org/10.1016/j.ultsonch.2023.106729

Giacomozzi AS, Carrın ME, Palla CA. 2018. Muffins elaborated with optimized monoglycerides oleogels: from solid fat replacer obtention to product quality evaluation. *J. Food Sci.* **83** (6), 1505-1515. https://doi.org/10.1111/1750-3841.14174

Guo J, Cui L, Meng Z. 2023. Oleogels/emulsion gels as novel saturated fat replacers in meat products: a review. *Food Hydrocoll.* **137**, 108313. https://doi.org/10.1016/j.foodhyd.2022.108313

Lim J, Hwang HS, Lee S. 2017. Oil-structuring characterization of natural waxes in canola oil oleogels: rheological, thermal, and oxidative properties. *Appl. Biol. Chem.* **60** (1), 17-22. https://doi.org/10.1007/s13765-016-0243-y

Manzoor S, Masoodi FA, Naqash F, Rashid R. 2022. Oleogels: Promising alternatives to solid fats for food applications. *Food Hydrocoll. Health*, **2**. https://doi.org/10.1016/j.fhfh.2022.100058

Muhoza B, Qi B, Harindintwali J, Koko M, Zhang S, Li Y. 2021. Encapsulation of cinnamaldehyde: an insight on delivery systems and food applications. *Crit. Rev. Food Sci. Nutr.* **63**, 2521-2543. https://doi.org/10.1080/10408398.2021.1977236

Ojijo NKO, Neeman I, Eger S, Shimoni E. 2004. Effects of monoglyceride content, cooling rate and shear on the rheological properties of olive oil/monoglyceride gel networks. *J. Sci. Food Agric.* **84** (12), 1585-1593. https://doi.org/10.1002/jsfa.1831

Oyom W, Mahmud N, Islam J, Valizadeh S, Awuku RB, Ibrahim SA, Tahergorabi R. 2024. Enhancing The Oxidative Stability, Physicochemical and Sensory Quality of Deep-fat Fried Chicken Nuggets Using Thyme Essential Oil-Loaded Oleogel Coatings. *Prog. Org. Coat.* **186**. https://doi.org/10.1016/j.porgcoat.2023.107977

Özek G, Demirci F, Özek T, Tabanca N, Wedge DE, Khan SI, Başer KHC, Duran A, Ergin Hamzaoglu E. 2010. Gas chromatographic–mass spectrometric analysis of volatiles obtained by four different techniques from Salvia rosifolia Sm., and evaluation for biological activity, *J. Chromatogr. A*, **1217** (5), 741-748. https://doi.org/10.1016/j.chroma.2009.11.086

Pang M, Shi Z, Lei Z, Ge Y, Jiang S, Cao L. 2020. Structure and thermal properties of beeswax-based oleogels with different types of vegetable oil. *Grasas Aceites* **71** (4), 380. https://doi.org/10.3989/gya.0806192

Pinto TC, Martins AJ, Pastrana L, Pereira MC, Cerqueira MA. 2021. Oleogel-based systems for the delivery of bioactive compounds in foods. *Gels* **7** (3). doi:10.3390/gels7030086

Pușcaș M, Socaciu C, Muste S, Muresan V. 2020. Phenolics dynamics and infrared fingerprints during the storage of pumpkin seed oil and thereof oleogel. *Processes* **8** (11), 1412. https://doi.org/10.3390/pr8111412

Razola-Díaz M, del C., Guerra-Hernández EJ, García-Villanova B, Verardo V. 2021. Recent developments in extraction and encapsulation techniques of orange essential oil. *Food Chem.* **354**, 129575. https://doi.org/10.1016/j.foodchem.2021.129575

Shang J, Zhong F, Zhu S, Huang D, Li Y. 2021. Formation, structural characteristics and physicochemical properties of beeswax oleogels prepared with tea polyphenol loaded gelators. *Food Func.* **12**, 1662–1671. https://doi.org/10.1039/D0FO02772C

Silva SS, Rodrigues LC, Fernandes EM, Lobo FCM, Gomes JM, Reis RL. 2022. Tailoring natural-based oleogels combining ethylcellulose and virgin coconut oil. *Polymers* **14**, 2473-2489. https://doi.org/10.3390/polym14122473

Thakur D, Singh A, Prabhakar PK, Meghwal M, Upadhyay A. 2022. Optimization and characterization of soybean oil-carnauba wax oleogel. *LWT–Food Sci. Technol.* **157**. https://doi.org/10.1016/j.lwt.2022.113108

Topal E, Ceylan Ö, Kösoğlu M, Mărgăoan R, Cornea-Cipcigan M. 2020. Structure, usage areas and main problems of bees wax. *U.Arı.D.-U.Bee.J.* **20** (2), 209-220. https://doi.org/10.31467/uluaricilik.781259

Wang J, Wan W. 2009. Experimental design methods for fermentative hydrogen production: A review. *Int. J. Hydrogen Energy* **34** (1), 235–244. https://doi.org/10.1016/j.ijhydene.2008.10.008

Xie J & Schaich K. 2014. Re-evaluation of the 2,2-diphenyl-1-picrylhydrazyl free radical (DPPH) assay for antioxidant activity. *J. Agric. Food Chem*. **62** (19), 4251-60. https://doi.org/10.1021/jf500180u

Yılmaz E, Öğütcü M. 2014. Properties and stability of hazelnut oil organogels with beeswax and monoglyceride. *J. Am. Oil Chem. Soc.* **91** (6), 1007–1017. https://doi.org/10.1007/s11746-014-2434-1

Yılmaz E, Keskin Uslu E, Öz C. 2021. Oleogels of some plant waxes: Characterization and comparison with sunflower wax oleogel. *J. Am. Oil Chem. Soc.* **98** (6), 643-655. https://doi.org/10.1002/aocs.12490

Yılmaz E, Öz C. 2023. Preparation and characterization of cold-pressed hazelnut oil oleogels with sunflower wax and polyglycerol stearate organogelators. *Akademik Gıda* **21** (1), 38-48, https://doi.org/10.24323/akademik-gida.1274003

Zhang A, Wang X, Zhong R, Li C, Chen F, Zhang D, Cao Y, Lan Y. 2024. Engineering crystal network of supramolecular Oleogel via kinetical regulation for improved lutein bioaccessibility. *Food Chem.* **463** (4), 141444. https://doi.org/10.1016/j.foodchem.2024.141444

Zhang H, Yang X, Zhong R, Huo Y, Zhu Y, Liang P. 2023. Antioxidative properties of fish roe peptides combined with polyphenol on the fish oil oleogel. *J. Sci. Food Agric.* **103** (4), 1714-1726. https://doi.org/10.1002/jsfa.12336

Zhu J, Tian D, Chen X, Huang T, Chen X. 2024. Preparation of Chitosan-phenolic Aldehyde Fragrance Oleogels and Comparative Study of their Structure and Properties. *Food Bioprocess Technol.* **17**, 4204-4214. https://doi.org/10.1007/s11947-024-03390-4