

Textural properties in oleogels: synergistic influence of vegetable oils and peppermint essential oil

Propiedades texturales en oleogeles: influencia sinérgica de aceites vegetales y el aceite esencial de menta

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Abstract

Introduction: In recent years, there has been an increase of functional alternatives to conventional fats and oleogels have emerged as a viable option for this purpose.

Objective: In this study, the textural properties of oleogels formulated with soybean (x1), olive (x2), and canola (x3) oils, peppermint essential oil (x4) and sunflower wax (x5), and were evaluated, to determine the synergistic effect of the components on the textural characteristics.

Methods: An I-optimal mixture experimental design was employed for the evaluation of these five ingredients, with firmness (mN) and consistency (mN*s) serving as the response variables that were determined by instrumental textural analysis.

Results: The analysis revealed that sunflower wax had the greatest impact on the oleogel texture, with a minimum gelling concentration of 2% (w/w). The results demonstrated that oleogel firmness exhibited a wide range, spanning from 75.64 to 1754.05 (mN). Furthermore, the data indicated that this parameter could be adjusted to three primary levels: soft (2%), medium (3–5%), and hard (7%), depending on the quantity of sunflower wax utilized. A notable shift in consistency was observed, exhibiting a significant change from 1023 to 17934 (mN*s), as the gelling agent concentration increased from 2% to 5% (w/w).

Conclusions: These findings demonstrate that the oleogels mechanical properties can be precisely adjusted by modifying the vegetable oil type and sunflower wax concentration, offering a potential alternative to traditional fats in various industries, including food, cosmetics, and pharmaceuticals.

Keywords: Fat replacer, Gelators, Waxes, Bioactive Compounds.

Resumen

Introducción: En los últimos años se ha observado un incremento de alternativas funcionales a las grasas convencionales, y los oleogeles han surgido como una opción viable para este propósito.

Objetivo: En este estudio se evaluaron las propiedades texturales de oleogeles formulados con aceites de soya (x1), oliva (x2) y canola (x3), aceite esencial de menta (x4) y cera de girasol (x5) y, con el fin de determinar el efecto sinérgico de los componentes sobre las características texturales.

Metodos: Se empleó un diseño experimental de mezcla I-óptimo para la evaluación de estos cinco ingredientes, utilizando la firmeza (mN) y la consistencia (mN*s) como variables de respuesta, determinadas mediante análisis instrumental de textura.

Resultados: El análisis reveló que la cera de girasol tuvo el mayor impacto en la textura del oleogel, estableciendo una concentración mínima de gelificación del 2% (p/p). Los resultados demostraron que la firmeza del oleogel presentó un amplio rango, oscilando entre 75.64 y 1754.05 mN. Además, los datos indicaron que este parámetro podía ajustarse a tres niveles principales: blando (2%), medio (3–5%) y duro (7%), dependiendo de la cantidad de cera de girasol utilizada. Se observó un cambio notable en la consistencia, evidenciado por una variación significativa de 1023 a 17934 mN*s al aumentar la concentración del agente gelificante del 2% al 5% (p/p).

Conclusiones: Estos hallazgos demuestran que las propiedades mecánicas de los oleogeles pueden ajustarse de forma precisa modificando el tipo de aceite vegetal y la concentración de cera de girasol, lo que ofrece una alternativa potencial a las grasas tradicionales en diversas industrias, incluyendo la alimentaria, cosmética y farmacéutica.

Palabras clave: TReemplazadores de grasa, Gelificantes, Ceras, Compuestos bioactivos.

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Contribution to the literature

Why was it conducted?

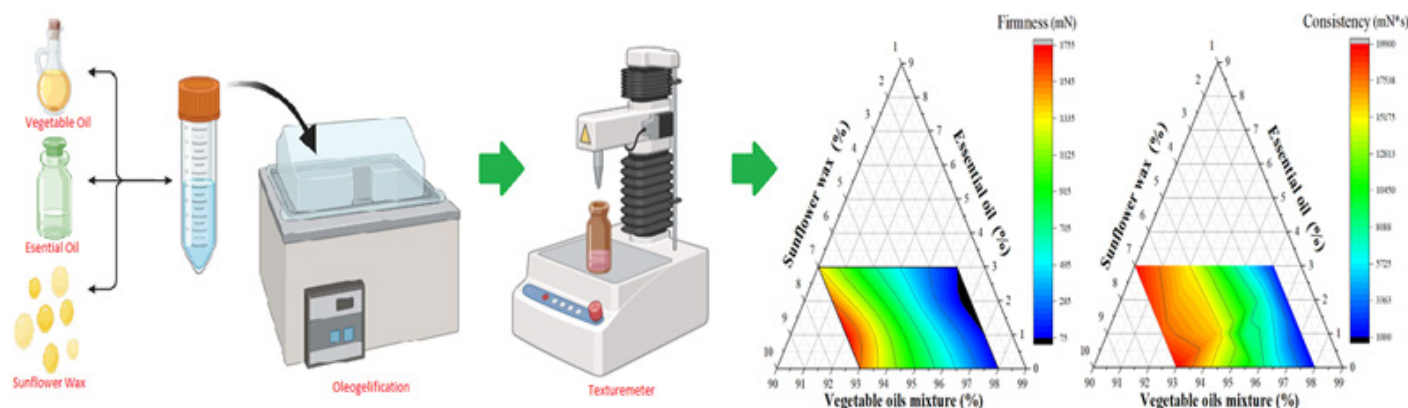
This study was conducted to explore functional alternatives to conventional fats by developing and characterizing oleogels with different vegetable oils and structuring agents. The motivation stems from the growing demand for healthier fat substitutes in various industries, particularly in food, cosmetics, and pharmaceuticals. By investigating the synergistic effects of sunflower wax and peppermint essential oil on the textural properties of oleogels, this research aimed to provide insights into optimizing their mechanical characteristics for different applications.

What were the most relevant results?

Sunflower wax was identified as the primary structuring agent, with a minimum gelling concentration of 2% (w/w). Oleogel firmness varied significantly, ranging from 75.64 to 1754.05 mN, depending on the wax concentration. Firmness could be classified into three levels: soft (2%), medium (3–5%), and hard (7%), based on the percentage of sunflower wax. Consistency increased notably from 1023 to 17934 mN·s when the wax concentration rose from 2% to 5% (w/w). Two mathematical models were developed to predict firmness and consistency, achieving high correlation coefficients ($r^2 = 95.94\%$ and 95.36%).

What do these results contribute?

These findings contribute to the literature by demonstrating that oleogel properties can be precisely tailored through formulation adjustments, particularly by modifying sunflower wax concentration and oil type. The study provides a scientific basis for designing structured lipid systems with controlled mechanical properties, expanding their potential as replacements for conventional fats. This work supports future research on clean-label, functional fat alternatives and encourages innovation in food and non-food industries seeking healthier and sustainable fat replacements.



Introduction

Oleogels are defined as liquid organic compounds (vegetable oils) within a three-dimensional network, obtained through a process that occurs through the dispersion of a gelling agent in a continuous oily phase to form a semi-solid. (1, 2). The structures created during the oleogelification process correspond to structures that form an angle within which the liquid is immobilized owing to surface tension, among other types of interactions, giving rise to a gel of variable consistency (3). The gelator substances include salts of fatty acids, steroids, amino acids, carbohydrates, and organometallic compounds. These molecules exhibit amphiphilic behavior, enabling them to assemble in hydrophobic liquids. This assembly achieves a balance between solubility and aggregation forces, primarily through dipolar interactions, covalent complexes, hydrogen bonds, and metal coordination. (4, 5). Structuring agents are generally low-molecular-weight molecules (<3000 Da) that can bind within a crystalline network. This process traps the surrounding oil and occurs as the system's temperature decreases. Modified celluloses, such as ethyl, methyl, and hydroxypropyl celluloses, are currently the dominant gelling agents. (2); however, other compounds, such as waxes, stearic acid, stearyl alcohol, β -sitosterol, oryzanol, lecithin, sorbitan tristearate, and ceramides, are also feasible for oleogel manufacture (6-8).

Oleogelification processes are executed through several mechanisms, which exhibit variations in nature and practical applicability. These mechanisms include: (a) the direct dispersion of the gelling agent within the oily phase, (b) an indirect method that utilizes continuous water emulsions, (c) structuring that is assisted by physical sorption of the liquid oil, and (d) biphasic structuring (3, 9). Oleogels exhibit diminished structural integrity at elevated temperatures and may demonstrate instability over prolonged periods. Interaction between oleophilic groups and oil molecules governs their ability to form structures. Oleogel absorption capacity is influenced by molecular weight and branching of structuring molecules, oil crystallinity, and solubility difference between oleogel and oil (10, 11). The dispersed phase in oleogels results in a variety of colloidal structures. These structures encompass three-dimensional crystalline networks, self-assembled fibrillar networks, structured emulsions, and polymeric networks (1,12).

Defined as products originating from plant material and obtained through diverse physical or chemical mechanisms, essential oils (EO) exhibit volatile liquid fractions. These fractions are generally composed of polymethylene hydrocarbons of the terpene type (C_5H_8), alongside a variety of oxygenated compounds, including monoterpenes, sesquiterpenes, alcohols, ethers, aldehydes, esters, and ketones (13-15). As products of secondary metabolites within plants, essential oils perform critical functions, including chemical defense against insects and predators, and facilitation of plant survival. Storage of these oils occurs in diverse cellular structures. These structures include surface cells, such as glandular hairs located on the plant surface, commonly observed in herbs like oregano, mint, and lavender; cells embedded within tissues, as found in citrus and eucalyptus leaves; and layers of cells surrounding intercellular spaces, such as those present in pine resin canals, secretory canals, glands, and trichomes (16, 17).

Peppermint, scientifically classified as *Mentha piperita*, belongs to the Lamiaceae family. The essential oil derived from this plant is generally obtained through steam distillation, a process that extracts volatile compounds from various plant parts, with leaves yielding a notably higher concentration. This oil presents a pale yellow or pale greenish yellow color, accompanied by a distinctive herbal odor and a fresh, cooling sensation. These characteristics arise from its complex chemical composition, which includes esters, alcohols, ketones, terpenes, and other volatile compounds. Notably, in Belgium, legal regulations stipulate that only the leaves are permissible as the source for this essential oil [\(18\)](#). Peppermint, a medicinal plant with substantial economic importance, is recognized for its comprehensive spectrum of pharmaceutical properties and nutritional value. Scientific investigations have thoroughly examined its traditional medicinal applications, which encompass the treatment of fever, colds, digestive problems, and inflammation. Moreover, studies have demonstrated its diverse biological activities, including antioxidant, antimicrobial, anti-inflammatory, and anticancer effects [\(19\)](#).

Essential oil applications are constrained by their inherent thermal sensitivity, high volatility, and propensity for oxidative degradation. Therefore, the development of adequate transport and dispensing systems is essential to safeguard EOs from external factors that compromise their quality and composition. To achieve successful incorporation of EOs into industrial applications spanning medicine, food, household items, personal care, biotechnology, pharmaceuticals, textiles, and cosmetics [\(20-23\)](#), it is imperative to adapt and modify specific EO properties. This intervention, frequently achieved through polymeric matrix techniques such as encapsulation, microencapsulation, and gelation, serves to mitigate volatility, enhance shelf-life, improve biological effects, and facilitate controlled release [\(16, 24, 25\)](#).

EO applications have some limitations due to its thermal sensitivity, high volatility and easy oxidative degradation, which is why it is necessary to look for adequate transport and dispensing systems that protect EOs from external factors that alter their quality and composition. Therefore, for the successful incorporation of essential oils (EOs) in products with industrial applications in fields such as medicine, food, household items, personal care, biotechnology, the pharmaceutical industry, the textile industry and cosmetics, it is necessary to adapt and intervene in certain properties to favor their performance, as this is the principle most commonly used to reduce the volatility of these compounds, thus improving the shelf-life, biological effects and controlled release of EOs. This improvement can be accomplished via different mechanisms, with the use of a polymeric matrix applied via techniques such as encapsulation, microencapsulation and gelation being the most used and investigated methodologies

In order to take advantage of the extensive technological and functional benefits of oleogels and essential oils, this research aimed to systematically evaluate the combined effect of multiple vegetable oils along with peppermint essential oil on the textural characteristics of the formed oleogels. This approach serves as a strategy to promote a broader application of this technology (oleogelation) and these bioactive compounds in the development of new food, pharmaceutical, or cosmetic products.

Materials and methods

Vegetable oils

Extra virgin olive oil, soybean, and canola oils were purchased from local retailers. To verify the authenticity and initial quality of the vegetable oils, they were subjected to gas chromatography analysis to determine the composition in terms of fatty acids, following a methodology and conditions standardized by the Food and Human Nutrition Laboratory – University of Antioquia (Agilent 6890 N chromatograph with an FID detector, TR-CN100 column, split/splitless injector ratio 100:1, injection volume 1.0 μL , injector temperature 260 $^{\circ}\text{C}$, temperature program: 90 $^{\circ}\text{C} \times 7$ min, increase of 5 $^{\circ}\text{C}$ to 240 $^{\circ}\text{C}$ and hold for 15 min, detector temperature 300 $^{\circ}\text{C}$, carrier gas: helium, flow rate 1.1 mL/min) (26).

Essential oils

The peppermint essential oil (*Mentha piperita*) was provided by Health & Beauty Natural Oils Company (HBNO, USA) and has a physical analysis certificate (refractive index (R_D), specific gravity (S_G), optical rotation (O_R), (R_D : 1.4592, S_G : 0.902, O_R : -26.68 $^{\circ}$), which guarantees botanical authenticity and no adulteration. To determine the composition, the following gas chromatographic analysis protocol (GC-FID) was used: Agilent Technologies 7890 chromatograph, FID detector: 250 $^{\circ}\text{C}$, injection: 250 $^{\circ}\text{C}$, split 1:30, volume 1 μL , Agilent 7683B automatic injector, capillary column: DB-5MS, 60 m \times 0.25 mm \times 0.25 μm , stationary phase: 5% diphenyl-95% dimethyl-polysiloxane, carrier gas: helium 1 mL/min. The oven temperatures were 45 $^{\circ}\text{C}$ - 150 $^{\circ}\text{C}$ (3 $^{\circ}\text{C}/\text{min}$), 220 $^{\circ}\text{C}$ (4 $^{\circ}\text{C}/\text{min}$), and 275 $^{\circ}\text{C}$ (10 $^{\circ}\text{C}/\text{min}$). Samples: 3 μL of essential oil dissolved in diethyl ether (1000 ppm). The percentage composition of each component in the essential oil was determined via automatic integration of the area of each peak generated by the GC-FID signal, which was compared with calibration curves (Chem-Station, Agilent Technologies, USA).

Gelling agent

Sunflower wax (*Helianthus annuus*) with melting point: 70 $^{\circ}\text{C}$, acidity: ≤ 8 mg KOH/g, saponification value: 75 - 95 mg KOH/g, and iodine value ≤ 12 g 100/g (Praan Naturals, USA).

Experimental design

An optimal mixture type experimental plan was established via Design Expert 10[®] software (Stat-Ease, USA). This design was created with the D-optimality criterion (integrated variance), with which it was intended to have the lowest predicted variance in the entire experimental region. Within experimental design, the D-optimality criterion aims to minimize the generalized variance of the parameter estimates. This is equivalent to minimizing the volume of the confidence ellipsoid and is achieved by maximizing the determinant of the Fisher information matrix. The study examined the effects of five formulation ingredients: olive oil (0–90%), soybean oil (0–90%), canola oil (0–90%), essential oil (0–3%), and gelling agent (2–7%). From these parameters, 25 mixtures were generated, including 15 base model points, 5 replicates, 4 verification points, and 1 mixture for lack-of-fit analysis. The point exchange algorithm was used to fit a Scheffe model. All percentages (%) refer to

the weight/weight percentage (% w/w) and were measured in grams (g). The experimental unit was 10 g per mixture, and the response variables Firmness (Fr) and Consistency (Cn) were determined by instrumental textural analysis.

Oleogels fabrication

All ingredients were measured in terms of weight to avoid errors due to volume and variation in density. Glass vials (15 ml) with screw caps were used, into which each of the formulation components given by the experimental design was dosed. The mixtures were then subjected to hydrothermal heating at 70 ± 1 °C and continuous ultrasonic stirring (Precision GP02, Thermo Scientific, USA) to avoid the formation of nonuniform temperatures zones that produce incomplete solubilization of sunflower wax. The vials always remained with the lid closed to avoid losses due to evaporation. The heating time was 15 minutes, after which each mixture was left to rest for a period of 24 hours at room temperature (24 ± 2 °C) before texture analysis was performed. The temperature and time used guaranteed complete dissolution of the gelling agent. These parameters were selected based on gelator physicochemical characteristics and a previous experimental design carried out to standardize the procedure (data not shown).

Textural analysis

For each vial, a downward force was applied at the oleogel center with a texture analyzer (TA-Xtplus, Stable Micro Systems, USA) equipped with a 5 kg load cell and a measuring accessory finished in a spherical tip with a 5 mm diameter (P/5S). The following configuration was used in the method, mode: compression force measurement; option: return to start; pretest speed: 2.0 mm/s; test speed: 1.0 mm/s; posttest speed: 1.0 mm/s; penetration distance: 20 mm; trigger type: Auto–5 g; test mode: Auto; data acquisition rate: 200 pps. The data were captured with Exponent® software Version. 6.1.15 (Stable Micro Systems, USA), where a macro was programmed to calculate the firmness (mN, maximum positive force) and consistency (mN*s, area under the positive force curve).

Statistical analysis

The results were studied via analysis of variance (ANOVA) by applying a significance level ($p \leq 0.05$) to estimate the significant effects of the factors (ingredients) on the response variables Firmness and Consistency (Design Expert 10®, Stat-Ease, USA). The nonsignificant terms were eliminated from the final predictive mathematical models to simplify the equations in terms of real values.

Results and discussion

Vegetable oils composition

The soybean oil (Glycine max) exhibited the following fatty acid ratios 1:1.51:3.82 (saturated : monounsaturated : polyunsaturated), normalized to saturated fat. The saturated fraction (15.5%) was primarily composed of palmitic (10.4%) and stearic acid (4.13%). Monounsaturated fatty acids

constituted 23.5% of the total, with oleic acid (23.4%) being the predominant component within this portion. In contrast, polyunsaturated fatty acids represented 59.4% of the total, with linoleic acid (51.6%) being the most abundant (Figure 1 – Peak 20). It is noteworthy that soybean oil contains the highest proportion of this type of fatty acid in comparison to olive and canola oils. The composition found is consistent with that reported in previous studies of soybean oil, which have identified major and minor components [\(27\)](#), in comparison, High-oleic acid soybean oil (H-OSBO) contains over 70% oleic acid, enhancing stability and shelf life, this composition reduces the requirement for hydrogenation and the production of trans-fatty acids [\(28\)](#).

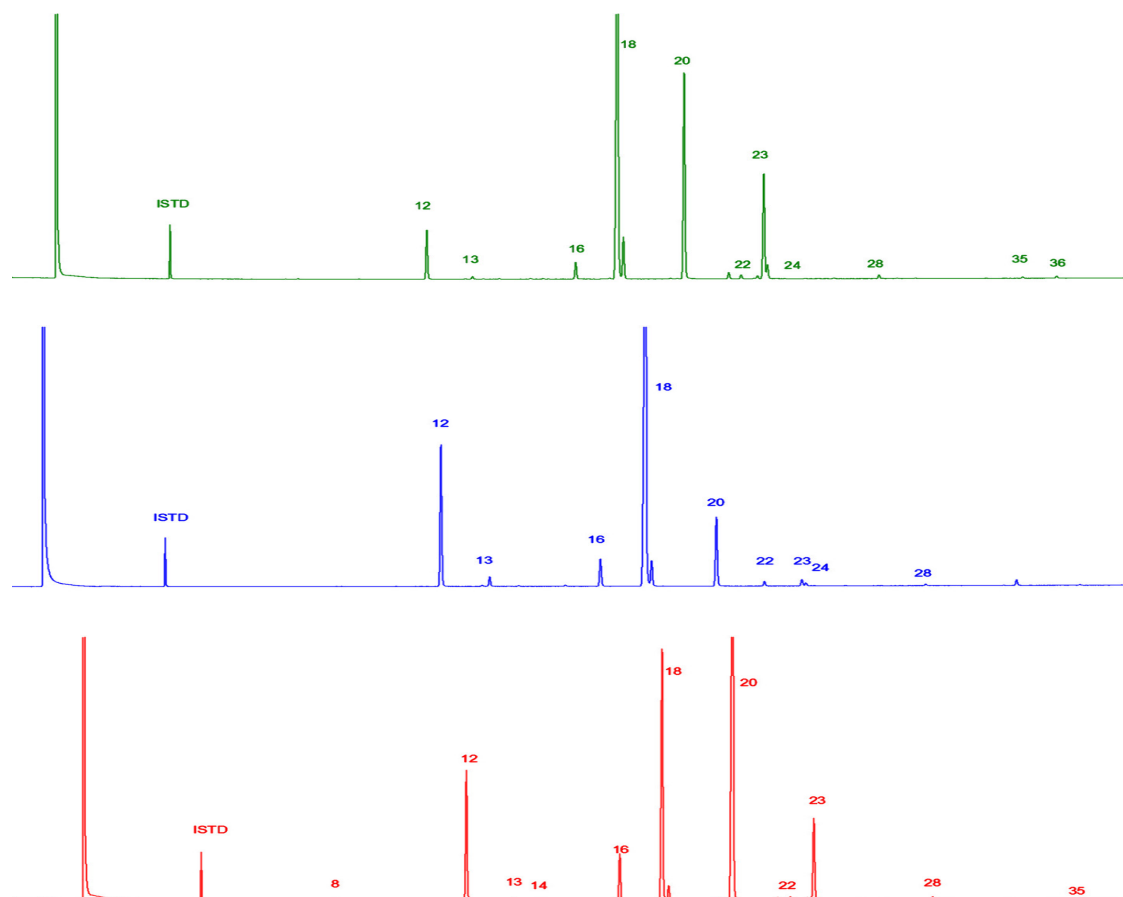


Figure 1. Chromatographic fatty acid profiles present in the vegetable oils of soybean (red), olive (blue), and canola (green) used in oleogel manufacturing. ISTD: Internal standard. See Table 1 for the fatty acids peaks assignment.

The analyses indicate that for olive oil (*Olea europaea*), the fat ratio was 1 : 4.64 : 0.48, and saturated fatty acids accounted for 16.1%, with palmitic acid (12.8%) and stearic acid (2.6%) being the most representative. The palmitic acid content is a significant factor in oxidation kinetics, elevated palmitic acid levels confer increased resistance to oxidation compared to unsaturated fatty acids, leading to a diminished and more consistent oxidation rate, oils containing less than 10% palmitic acid demonstrate a higher susceptibility to oxidation [\(29\)](#). The monounsaturated fatty acid component constituted 74.7% of the oil, wherein oleic acid represented the predominant constituent at 73.4%. The polyunsaturated fatty acid fraction comprised 9%, with linoleic

acid accounting for approximately 7.1% and olive oil exhibited the lowest concentration of polyunsaturated fatty acids relative to soy and canola oils. Literature data on olive oil fatty acid composition revealed oleic acid levels ranging from 70.3% to 87.4% and linoleic acid levels ranging from 4.4% to 12.6%, the results from our work are in line with these reports (30, 31). The favorable nutritional profile and established health benefits of olive oil render it a prime candidate for saturated fat replacement. Nevertheless, the escalating price of this raw material impedes its industrial-scale application in oleogel formulations (32).

Canola oil (*Brassica napus*) presents a saturated : monounsaturated : polyunsaturated fatty acid ratio of 1:8.78:4.57. Saturated fatty acids constitute 6.5%, with palmitic (4.0%) and stearic (1.5%) acids as the primary components; canola oil exhibits the lowest saturated fat content. Monounsaturated fatty acids account for 57.4%, predominantly oleic acid and polyunsaturated fatty acids comprise 29.9%, consisting of linolenic (10.2%) and linoleic (19.7%) acids. Canola oil demonstrates an intermediate profile for monounsaturated and polyunsaturated fatty acids relative to soybean and olive oils, while maintaining the lowest saturated fat content, consistent with established literature (33). The consistent availability, moderate cost, and favorable nutritional properties of canola oil contribute to its widespread application in the food industry to produce margarine, spreads, mayonnaise, salad dressings, and various processed foods. The vegetable oils examined adhered to established quality and composition standards, confirming the absence of adulteration or chemical alteration that could compromise oleogel formation (34, 35).

Table 1. Fatty acid profiles (g/100 g) present in the soybean, olive and canola oils used in the manufacturing of oleogels.

Peak	Fatty acid	Soy		Olive		Canola	
		(g/100 g)	±	(g/100 g)	±	(g/100 g)	±
8	C14:0 (Myristic acid)	0.07	0.005	-	-	-	-
12	C16:0 (Palmitic acid)	10.48	0.004	12.82	0.028	4.02	0.016
14	C17:0 (Heptadecanoic acid)	0.11	0.006	-	-	-	-
16	C18:0 (Stearic acid)	4.13	0.001	2.69	0.009	1.52	0.008
22	C20:0 (Arachidic acid)	0.33	0.004	0.45	0.007	0.55	0.001
28	C22:0 (Behenic acid)	0.33	0.006	0.14	0.005	0.31	0.001
35	C24:0 (Lignoceric acid)	0.12	0.008	-	-	0.1372	0.14
	Total Saturated Fat	15.56	0.005	16.10	0.048	6.54	0.020
13	C16:1 (Palmitoleic acid)	0.13	0.001	0.94	0.002	0.23	0.001
18	C18:1n9c (Oleic acid)	23.42	0.005	73.48	0.136	55.66	0.169
24	C20:1n9 (Cis-11-eicosenoic acid)	-	-	0.29	0.29	1.3189	1.32
36	C24:1n9 (Nervonic acid)	-	-	-	-	0.1963	0.002
	Total Monounsaturated Fat	23.55	0.004	74.71	0.143	57.40	0.176
20	C18:2n6c (Linoleic acid)	51.65	0.040	7.13	0.006	19.73	0.052
23	C18:3n3 (α-linolenic acid)	7.75	0.220	0.68	0.000	10.20	0.042
	Total Polyunsaturated Fat	59.40	0.261	7.81	0.209	29.93	0.302
	Total Fat	98.51	0.259	98.62	0.196	93.87	0.289

Essential oil composition

The chemical analysis of peppermint oil revealed that the top 10 components accounted for 94.50% of the total, which were Menthol, Menthone, 1,8-Cineole, Menthyl acetate, Menthofuran + Isomenthone, Neo-Menthol, Limonene, Sabinene + β -Pinene, β -Caryophyllene, and α -Pinene, identifying a total of 54 molecules in the oil used (Table 2). The essential oil was characterized by menthol (45.98%, $C_{10}H_{20}O$, 156.3 g/mol) and menthone (21.96%, $C_{10}H_{18}O$, 154.25 g/mol) as the two primary components, with their respective proportions falling within the established quality standard ranges for this essential oil. The ratio of 1,8-cineole to limonene was calculated to be 2.74, satisfying the minimum threshold of 2. According to the United States Pharmacopeia (USP) guidelines, the quality standards for *Mentha Piperita* oil mandate a minimum presence of 5% total esters, expressed as methyl acetate, and the oil utilized in this study exhibited a content of 5.36%, thus meeting the required specifications (36).

Menthol, the principal component identified in the oil, is categorized as a cyclic monoterpene, a class of natural compounds with extensive industrial applications and documented biological activity in both in vitro and in vivo studies (37). Menthol exerts a significant influence on the structural integrity of fungal cell membranes, inducing depolarization and subsequent chemical or physical alterations that disrupt metabolic activities. Furthermore, minor components, such as limonene, β -pinene, β -caryophyllene, and α -pinene, contribute to the antimicrobial properties of *Mentha* essential oil, potentially through synergistic interactions that amplify their individual effects (38).

Table 2. Chemical Composition of *Mentha piperita* essential oil incorporated into Oleogels.

Peak	Compound	RT (min)	(%)	Peak	Compound	RT (min)	(%)
1	cis-2-Hexenol	7.60	0.04	29	Neo-Menthol	25.64	3.78
2	α-Thujene	8.97	0.03	30	Menthol	26.86	45.98
3	α-Pinene	9.23	0.91	31	Isomenthol	27.17	0.28
4	Methylcyclohexanol	9.76	0.07	32	Neoisomenthol	27.70	0.05
5	Camphene analog	9.96	0.02	33	α-Terpineol	27.79	0.24
6	Methylcyclohexanone	10.09	0.13	34	Pulegone	31.60	0.68
7	Sabinene + β-Pinene	11.54	1.73	35	Carvone	32.04	0.08
8	1-Octen-3-ol	12.10	0.05	36	Piperitenone	32.80	0.25
9	1-Octen-3-ol	12.53	0.03	37	neo-Menthyl acetate	34.92	0.10
10	β-Myrcene	12.74	0.30	38	Dihydroedulan I	35.48	0.06
11	3-Octanol	13.10	0.29	39	Dihydroedulan II	35.90	0.02
12	α-Phellandrene	0.01	0.04	40	Menthyl acetate	36.61	5.36
13	α-Terpinene	14.19	0.20	41	Isomenthyl acetate	37.48	0.06
14	p-Cymene	14.75	0.12	42	Bicycloelemenene	39.62	0.04
15	Limonene	15.04	2.13	43	α-Copaene	42.57	0.06
16	1,8-Cineole	15.16	5.84	44	β-Bourbonene	43.19	0.09
17	(Z)-β-Ocimene	15.98	0.10	45	β-Elemene	44.07	0.05
18	(E)-β-Ocimene	16.69	0.03	46	β-Caryophyllene	45.85	1.63
19	γ-Terpinene	17.24	0.35	47	β-Copaene	46.68	0.06
20	Sabinene hydrate	0.02	0.23	48	α-Humulene	48.48	0.07
21	Octanol	18.63	0.03	49	(E)-β-Famesene	49.72	0.11
22	α-Terpinolene	0.02	0.06	50	Germacrene D	50.70	0.54
23	Sabinene hydrate	20.21	0.03	51	Bicyclogermacrene	51.91	0.08
24	Linalool	20.66	0.10	52	δ-Cadinene	54.25	0.04
25	Amyl isovalerate	21.07	0.03	53	Caryophyllene oxide	58.14	0.02
26	Isopulegol	23.90	0.14	54	Viridiflorol	58.82	0.06
27	Menthone	24.82	21.96	Total		99.96	
28	Menthofuran + Isomenthone	25.46	5.18				

Pulegone is a key compound for assessing peppermint oil quality, found in higher concentrations in young leaves, it is metabolically converted to menthol during leaf maturation, a significant pulegone presence signifies oils derived from plants in early developmental stages, correlating with lower quality, conversely, high-quality oils exhibit pulegone concentrations below 1% (39). In this study, the pulegone was 0.68%, and menthofuran concentration 5.18%, both values conforming to the ISO 856 standard, which delineates quality parameters for peppermint oils from United States (menthofuran: 1.5 - 6.0%; pulegone: 0.5 - 2.5%) and other origins (menthofuran: 1.0 - 8.0%; pulegone: 0.5 - 3.0%). Furthermore, the Cosmetic Ingredient Review Expert Panel (CIR) has concluded that peppermint oil is safe for use in cosmetic formulations when pulegone content remains below 1.0%, establishing a Total Daily Intake (TDI) = 0.1 mg/kg body weight, for the combined intake menthofuran + pulegone (40).

Textural analysis

Table 3 provides the data derived from the textural analysis conducted on the oleogel formulations. The results indicate that all mixtures successfully formed oleogels, thereby establishing a 2% (w/w) critical sunflower wax concentration for the studied system. Waxes have been demonstrated to be highly efficient gelating agents, capable of forming well-defined, structured networks (long fibers) that immobilize vegetable oil through surface tension, achieving this effect even at very low concentrations of 0.5% (w/w) (41).

Table 3. Mixture experimental design applied to analyze the oleogels incorporated with peppermint essential oil (all ingredients: % w/w).

Mixture	Vegetal Oil			x_4 Essential oil	x_5 Sunflower wax	Firmness (mN)	Consistency (mN*s)
	x_1 Olive	x_2 Soy	x_3 Canola				
1	32	32	32	0	4	624.63	7792.3
2	48	47	0	3	2	84.63	1751.0
3	0	47	47	1	5	986.69	17934.6
4	4	4	90	0	2	77.89	1082.6
5	32	32	31	1	4	607.40	8424.8
6	0	3	90	0	7	1507.37	19580.8
7	0	90	3	0	7	1585.23	19681.1
8	61	18	16	2	3	199.97	7549.1
9	32	32	32	0	4	625.36	7636.3
10	45	0	45	3	7	1355.31	19870.2
11	0	2	90	3	5	567.71	14702.2
12	47	0	47	1	5	808.87	13628.4
13	47	47	0	1	5	989.76	14674.3
14	90	6	0	0	4	781.91	10948.0
15	47	0	47	1	5	1017.08	15285.0
16	47	47	0	1	5	919.66	16233.4
17	0	47	47	1	5	967.65	17227.9
18	90	0	2	1	7	1754.05	18017.7
19	47	47	0	1	5	981.88	16473.1
20	90	0	2	3	5	1021.57	15000.4
21	0	45	45	3	7	1348.87	19058.0
22	0	90	8	0	2	75.64	1023.5
23	90	0	7	1	2	77.14	1284.7
24	0	48	47	3	2	84.67	1770.6
25	2	90	0	3	5	561.72	15681.7

Firmness refers to the structural product strength and indicates how much it can withstand an external force, exceeding maximum force results in oleogel internal structure rupture. It is an imitative test that simulates the ease with which a human finger would deform the oleogel during application, for example, on the skin, as the breaking force increases, the gel becomes more resistant and more fragile as the firmness decreases. The mixtures with which the oleogels were prepared presented values ranging from 75.64-1754.05 (mN) and can be grouped into three firmness categories: soft (less than 495 mN - blue), intermediate (495-1150 mN - green), and hard (> 1150 mN - Red), these three proposed empirical categories were based on data analysis and contour plots in the Figure 2 (ternary diagram), which also shows that the gelator amount was the most influential factor (trace plot). These firmness categories correspond to sunflower wax levels of 2, 3-5, and 7% (w/w), respectively, analysis of variance revealed no significant interaction between ingredients (Table 4).



The main effect of the gelling agent on firmness can be observed in the trace graph (Figure 2); thus, the importance of the different components present in the oleogel can be evaluated. This graph starts with a reference mixture or centroid of the experimental region (0.0), observing the way in which the response (Firmness) changes as the sunflower wax increases or decreases, when one of the ingredients changes, the remaining components adjust proportionally. Wax-based oleogels, as in this research, can be considered supramolecular gels containing a mixture of different gelling molecules, which produce different types of self-organizing bonds, resulting in diverse morphologies within the oleogel. High tailoring capacity in the crystal lattice is offered by these coassembled systems (32). Therefore, resulting oleogels' macroscopic properties can be widely manipulated by changing wax type and concentration, solvent polarity, and working conditions, including cooling rate, mixing velocity, or gelation temperature.

Table 4. Analysis of variance (ANOVA) examining the ingredients influence on the firmness oleogels with peppermint essential oil.

Source	Sum of Squares	df	Mean Square	F-value	p-value
Model	6.01E+06	14	4.29E+05	40.2	< 0.0001
Linear Mixture	5.82E+06	4	1.45E+06	136.13	< 0.0001
$x_1 * x_2$	295.05	1	295.05	0.0276	0.8713
$x_1 * x_3$	17755.85	1	17755.85	1.66	0.2263
$x_1 * x_4$	6906.33	1	6906.33	0.6467	0.4412
$x_1 * x_5$	23237.75	1	23237.75	2.18	0.1709
$x_2 * x_3$	35250.58	1	35250.58	3.3	0.0993
$x_2 * x_4$	6418.55	1	6418.55	0.6011	0.4561
$x_2 * x_5$	25496.22	1	25496.22	2.39	0.1533
$x_3 * x_4$	6438.06	1	6438.06	0.6029	0.4554
$x_3 * x_5$	25837.75	1	25837.75	2.42	0.1509
$x_4 * x_5$	920.24	1	920.24	0.0862	0.7751
Lack of Fit	67653.49	5	13530.7	1.73	0.2813
Total	6.12E+06	24			

The analyzed firmness is directly related to the gelation phenomenon of the monocrystalline particles present in sunflower wax, such as n-alkanes, acids and fatty alcohols. When heated, these particles dissolve, and as the temperature decreases, they form solid nuclei. This growth process results in strong intercrystalline interactions, which lead to the formation of supramolecular structures. These structures act as a framework, effectively capturing liquid vegetable oil and creating a three-dimensional network. The sintered primary connections are characterized by their strength, which, together with the (weaker) secondary Van der Waals interactions, act as the main driving forces responsible for the formation of this network structure in oleogels.

Sunflower wax, the gelator used in this work, has low polarity, long chains and a high melting point, which contributes to the excellent crystallization properties of vegetable oils. The predominant components of waxes also influence their morphology and crystalline structure. Generally, waxes present three morphologies in their natural state: an orthorhombic structure, common in aliphatic compounds; another tricyclic phase, present in secondary alcohols; and a hexagonal structure,



which exists in beta-diketones. The proportion of these different structures is what contributes most to the consistency of the oleogel formed (42).

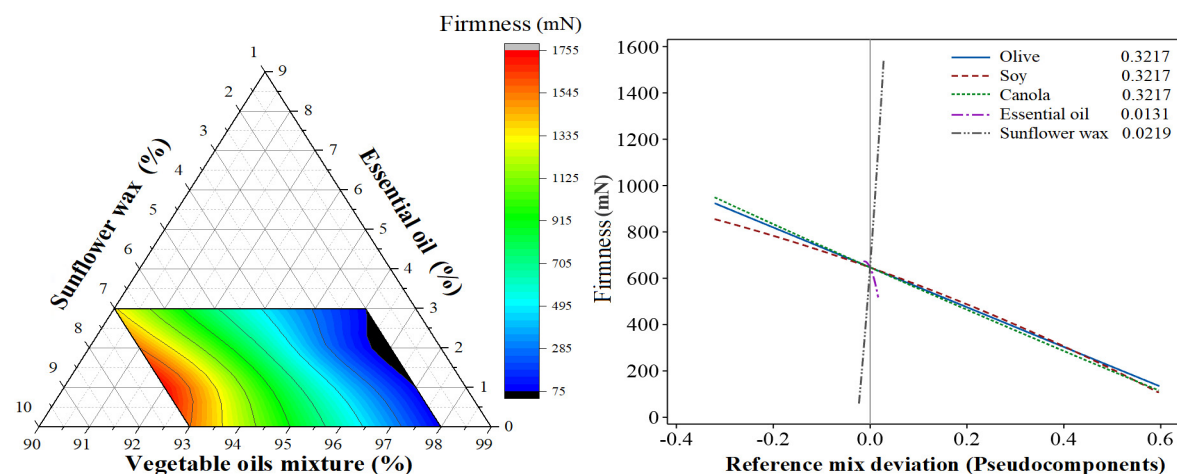


Figure 2. Ternary diagram (left) and trace plot (right) of oleogel firmness (mN) as a composition function.

Oleogel consistency is critical for functionality and acceptability in practical applications. In food products, it influences mouthfeel and quality perception. In cosmetics, appropriate consistency enhances application and user experience, for pharmaceuticals, it affects active ingredient stability and controlled release. The formulated oleogels show consistency values ranging from 1023-19870 (mN*s) and can be classified into four quantitative ranges: less than 1770 (mN*s), 7549-10948 (mN*s), 13628-15681 (mN*s), and greater than 16233 (mN*s), these four empirical consistency categories were established based on data analysis and contour plots presented in Figure 3. The gelling agent was identified as the primary factor influencing oleogel consistency. Sunflower wax concentrations of 2, 3-4, 5, and 7% (w/w) correlated with each consistency category.

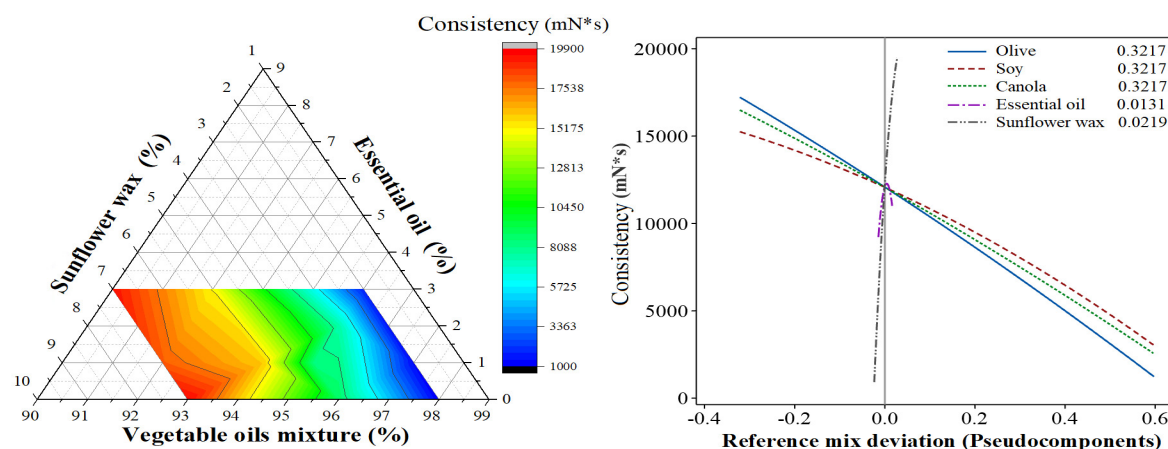


Figure 3. Ternary diagram (left) and trace plot (right) of oleogel consistency (mN*s) as a composition function.

A more detailed analysis of the data generated revealed that a great change in consistency occurred at low concentrations of the gelling agent, especially when the concentration increased from 2 to 3%, where the consistency increased by more than 5000 mN*s (mixtures 24 and 8); when the concentration changed from 4 to 5%, this effect was reduced by almost half, 2600 mN*s (mixtures 12 and 14). Finally, when the concentration of the gelling agent increased from 5 to 7%, an average increase of 1000 mN*s was produced. Finally, analysis of variance (Table 5) revealed that the linear mixture of the ingredients was significant, with interactions between some of the ingredients (soy, olive, and canola oils: gelling agent). If firmness and consistency are maximized, the three vegetable oils (olive, soybean, and canola) should be present in similar proportions (~32% each), the gelling agent should be present at a medium-high level (4-5%), and the peppermint essential oil should be present at low levels (0-1%).

No significant effect ($p \geq 0.05$) of peppermint essential oil on oleogel consistency was detected, which can be explained by the low concentration used (0–3% w/w) to comply with international regulations. It is important to mention that peppermint essential oil is not considered a new food or novel ingredient, meaning “any food that has not been used to a significant extent for human consumption in the European Union”; therefore, it falls outside the scope of Regulation (EU) 2015/2283 on novel foods that came into force in 2018, and the US Food and Drug Administration (FDA) assigns the GRAS (Generally Recognized as Safe) status.

Table 5. Analysis of variance (ANOVA) of the effects of the ingredients on the consistency of the oleogels incorporated with peppermint essential oil.

Source	Sum of Squares	df	Mean Square	F-value	p-value
Model	1.02E+09	14	7.29E+07	25.3	< 0.0001
Linear Mixture	9.58E+08	4	2.39E+08	83.03	< 0.0001
$x_1 * x_2$	2.25E+06	1	2.25E+06	0.7787	0.3982
$x_1 * x_3$	8.52E+06	1	8.52E+06	2.96	0.1163
$x_1 * x_4$	1.38E+07	1	1.38E+07	4.8	0.0532
$x_1 * x_5$	3.14E+07	1	3.14E+07	10.91	0.0084
$x_2 * x_3$	1.29E+06	1	1.29E+06	0.4482	0.5184
$x_2 * x_4$	1.36E+07	1	1.36E+07	4.73	0.0547
$x_2 * x_5$	3.20E+07	1	3.20E+07	11.11	0.0076
$x_3 * x_4$	1.36E+07	1	1.36E+07	4.72	0.0549
$x_3 * x_5$	3.18E+07	1	3.18E+07	11.03	0.0077
$x_4 * x_5$	2.50E+07	1	2.50E+07	8.67	0.0547
Lack of Fit	2.53E+07	5	5.06E+06	7.14	0.2251
Total	1.05E+09	24			

Given its status as the major constituent of soybean oil, linoleic acid ($C_{18}:2n6c$) warrants detailed analysis, particularly concerning its technological implications in soybean oil oleogels. The polyunsaturated nature of this omega-6 fatty acid leads to an intermediate level of oxidative stability, double bonds inherent in linoleic acid negatively impact oleogel quality and reduce shelf

life under continuous exposure to oxygen, light, and heat. Linoleic acid contributes to alterations in the texture and consistency of food products. Specifically, in baked goods, it affects the softness and structural integrity of dough, leading to an increase in volume while concurrently decreasing its overall stability (43). Polyunsaturated fatty acids, including linoleic acid, are recognized for their capacity to form stable emulsions, which is advantageous in the production of sauces and dressings. They also contribute to food flavor and palatability, although the specific sensory perception is dependent on concentration and other formulation compounds (44).

The impact of oleic acid (C18:1n9c) on olive oil oleogels has been extensively studied, especially regarding technological properties in food applications. Research on replacing traditional fat with oleic acid-rich oleogels in Bologna sausages demonstrated that increasing oleic acid content to 20% preserves oxidative stability and enhances nutritional profile without significant sensory differences (45). Oleic acid is more stable than polyunsaturated fatty acids are, and oleogels rich in oleic acid are less prone to oxidation (it is slower and more constant) when exposed to heat. In addition, increasing its proportion helps stabilize the rheological properties of semisolid products such as butter and improves the texture of baked goods such as cakes while reducing the use of saturated fats (46).

It was also found that oleogels with high canola contents (90%) tend to have lower firmness and consistency values. Specifically, for canola oil in the food industry, industrial applications have been developed for spreads and gummies (47), breads (41), and low-fat cheeses (48). These studies have established that the ultimate texture of oleogels is highly contingent upon the ratio of oleic to linoleic fatty acids and the selection of the gelling agent, which in turn governs the type of fiber formed during the crystallization process. These investigations also verified that oleogels formulated with canola oil exhibit intermediate oxidative stability and can be precisely engineered to fulfill the specific texture requirements of the food product into which they are incorporated. Following analysis of variance and multiple regression, nonsignificant effects ($p \geq 0.05$) were removed. Equations predicting firmness and consistency, based on actual ingredient quantities, were generated (Figure 4). These equations exhibited correlation coefficients (R^2) of 95.94% and 95.36%, respectively, significance and lack-of-fit criteria for the models were presented in Tables 4 and 5.

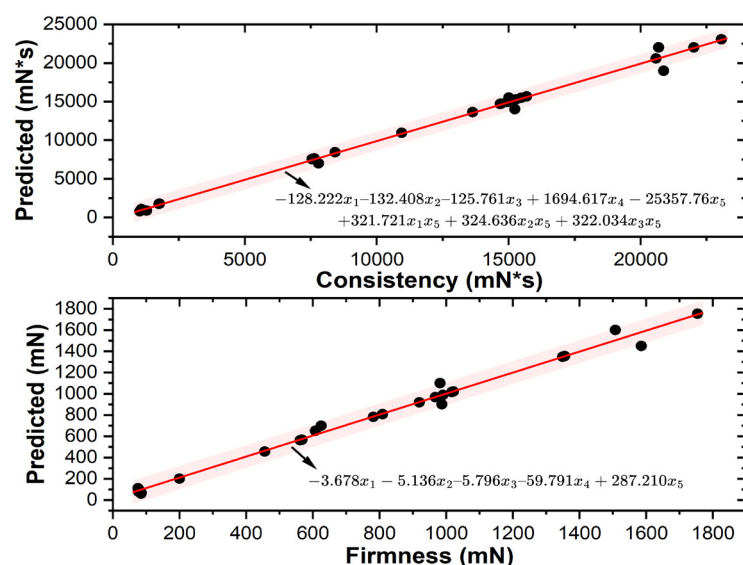


Figure 4. Scatterplot of firmness (mN) and consistency (mN*s) vs. predicted values of the oleogels as a function of composition (shaded area = 95% confidence interval).

The predictive model demonstrates that all three oils (x_1 , x_2 , x_3) exert a negative influence on the firmness of the oleogels, with canola oil exhibiting the most pronounced negative effect, this observation is likely correlated with the relatively high concentrations of unsaturated fatty acids present in canola oil, which tend to form less rigid structural networks compared to saturated fatty acids. Conversely, olive oil, characterized by the highest saturated fat content among the three oils, displayed the least negative impact on firmness, a result consistent with this expectation. Soybean oil, with an intermediate level of polyunsaturated fatty acids, exhibited a moderate reduction in firmness. The model also indicates that all three oils reduce oleogel consistency, with similar magnitudes of coefficient values. This suggests that factors other than unsaturation type, or potential interactions between oils and other oleogel components, may obscure the individual effects of their respective fatty acid profiles on consistency.

A positive interaction terms noted between each oil and sunflower wax concerning consistency suggest that the presence of unsaturated fatty acids from the oils may modulate the structuring effect of sunflower wax, potentially resulting in increased consistency at higher wax concentrations. While the specific fatty acid profiles likely contribute to the nature and magnitude of these interactions, current linear model, even with interaction terms, may not comprehensively capture the subtle nuances of these relationships. Furthermore, the arrangement and type of fatty acids within the triacylglycerols of each oil can influence their interaction with the wax and resultant oleogel structure. For instance, the distribution of saturated and unsaturated fatty acids within individual molecules can alter the oil's polarity and its compatibility with the wax. Additionally, the presence of minor components within the oils, which are not reflected in the fatty acid profile, could also contribute to the observed textural properties.

Given the high correlation observed among the predictive models presented, it is reasonable to infer that these models will enable the prediction of how the varying quantities of ingredients affect the oleogels final properties, specifically in terms of firmness (Nm) and consistency (Nm*s). These mathematical expressions may be applied to streamline the formulation process, optimizing outcomes while reducing the costs associated with trial and error tests. Oleogelation strategies are designed to not only alter the final product's structure but also to improve its functional attributes. This is particularly relevant in the cosmetic and pharmaceutical sectors, where oleogels are utilized as "release vehicles" to facilitate the delivery of humectants and functional essential oils to the skin's surface. Additionally, the structured oil matrix provided by oleogels serves to inhibit syneresis on the surface of cosmetic products, a phenomenon caused by oil migration [\(20\)](#).

Conclusion

All vegetable oils (soybean, olive, canola) and peppermint essential oil met established compositional standards. Oleogel firmness and consistency were directly correlated with gelling agent concentration, with 2% (w/w) identified as the critical concentration. Oleogel firmness was empirically categorized into three distinct levels: soft (2%), intermediate (3-5%), and hard (7%), based on the sunflower wax amount. This classification was determined by the quantity of sunflower wax utilized as a gelling agent, facilitating the texture control according to the intended application. Furthermore, the consistency exhibited a notable increase with rising gelling agent concentrations, particularly between 2% and 5%. The two mathematical models developed to predict the oleogels firmness (mN) and consistency (mN*s) based on their composition were statistically significant ($p \leq 0.05$). The incorporation of peppermint essential oil did not result in statistically significant modifications to the textural characteristics of the oleogels. This outcome is likely attributable to the low concentration of peppermint essential oil employed, specifically up to 3% w/w, which permits the attainment of functional enhancements, as documented in scientific literature, without causing substantial alterations to the product's structural integrity. The ability to modulate oleogel firmness and consistency demonstrates their adaptability, providing significant customization potential across food, cosmetic, and pharmaceutical industries, where these mechanical properties are paramount for final product acceptance and efficacy.

CRediT authorship contribution statement

Conceptual Idea: Zapata-Betancur, F.; Forero-Longas, F.; Methodology design: Zapata-Betancur, F.; Forero-Longas, F.; Data collection: Forero-Longas, F Pulido-Diaz A.; Data analysis and interpretation: Zapata-Betancur, F.; Forero-Longas, F.; Pulido-Diaz A.; and Writing and editing: Zapata-Betancur, F.; Forero-Longas, F.; Pulido-Diaz A.

Conflict of interest: does not declare

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