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Effect of different hydrocolloids as gelator on the qualitative properties of canola oil oleogel

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ABSTRACT

Considering the special ability of hydrophilic polymers in forming oleogels, in this study, the properties of seven selected hydrocolloids and their ability to form oleogels was investigated. For this purpose, canola oil-based oleogels were prepared using 3% solutions of gum arabic (AG), locust bean gum (LBG), xanthan (XG), guar (GG), hydroxypropyl methylcellulose (HPMC), carboxymethylcellulose (CMC), and carrageenan (CG). The oil retention ability, texture, thermal behavior, rheological properties, crystallinity, and infrared spectra of the produced oleogels were evaluated. Oleogels exhibited solid viscoelastic behavior and their elastic and complex moduli increased with increasing frequency and their complex viscosity decreased, indicating shear thinning behavior. Locust bean gum and the prepared oleogel showed the weakest viscoelastic properties and the highest oil release. In the thermal profile of the oleogels, one to five endothermic peaks were observed, which indicated the different crystallization characteristics and melting behavior of the oleogels and the formation of different polymorphs in them. The oleogel containing AG had the highest and the LBG-containing sample had the lowest texture stiffness and melting enthalpy. Evaluation of the crystal structures showed that all samples except the oleogels containing LBG had a mixture of semi-stable and stable β′ and β crystals. No significant chemical change in the structure of the samples was observed, but an increase in the intensity of hydrogen and van der Waals bonds was confirmed after oleogel formation. Type of hydrocolloids has a significant effect on the oil-polymer interactions and functional properties of oleogels. LBG with the weakest viscoelastic properties and melting temperature was not suitable for oleogelation, while oleogels prepared based on canola oil with AG, followed by HPMC and CMC with suitable oil binding capacity, strength and viscoelastic properties appropriate for food products with low saturated and trans fatty acid content.

1- Introduction

Solid fats have an important role in texture, mouthfeel, oxidative stability, and overall nutritional quality of food products [1]. In bakery products such as bread and cakes, solid fats are particularly important in structure, volume, and sensory acceptability of products [2]. Conventional approaches such as hydrogenation and esterification are commonly used to convert liquid oils to semi-solid or solid fats. The processes by changing chemical properties of products form saturated and trans fatty acids, that are associated with increased risks of cardiovascular diseases, cancer, and diabetes [2]. Therefore, regulatory agencies have imposed restrictions on the consumption of them. According to recommendations of FDA, saturated fat intake should be limited to no more than 10% of total daily energy intake [3]. The alternative strategies are necessary to develop healthier solid fats. In this context, oleogels have been proposed as a suitable alternative with higher content of essential fatty acids and improved nutritional properties [4].

Oleogelation is an innovative approach that enhances the physical properties of edible oils without altering the chemical structure of their fatty acids. It is considered an effective alternative for producing solid-like fats with high content of unsaturated fatty acids [3]. In this process, vegetable oils are structured into thermoreversible, three-dimensional gel networks using organogelators [5]. These gelators may consist of low-molecular-weight organic compounds (LMOGs) or polymeric gelators (PGs), which stabilize the structure through hydrogen bonding, van der Waals forces, and other intermolecular interactions [5,6]. The type and concentration of gelator, along with the characteristics of the oil, are key factors determining the structure and functionality of oleogels. Oleogels are suitable not only in the food industry but also in pharmaceutical and cosmetic formulations. In foods, they reduce saturated fat content, minimize fat migration, and improve the products quality such as cakes, chocolate, and ice cream [7].

Hydrocolloids are long-chain biopolymers with a strong hydrophobicity due to hydroxyl groups They interact with water molecules and create desirable functional properties [8]. Hydrocolloids are widely used in food industry for increasing viscosity, forming gels, stabilizing foams and emulsions, and controlling crystallization [1]. They are derived from plants, microorganisms, animals, and marine sources and play an important role in modifying rheological behavior, texture, and sensory characteristics of food products. Their application has been extensively studied in products such as sauces, jams, jellies, desserts, and low-fat mayonnaise to improve consistency and mouthfeel [8]. Galactomannans are kind of polysaccharides composed of mannose and galactose. One of the most important members of this group is LBG, extracted from the of *Ceratonia siliqua* seeds. The mannose-to-galactose ratio in galactomannans is approximately 4:1 and it has unique physicochemical properties that depend on the distribution of galactose along the backbone and also environmental conditions, such as pH [9,10]. LBG is widely used in dairy and beverage products as emulsifier and stabilizer. In the recent studies, its role as a fat replacer in oil-based cakes was reported. Combination of LBG and XG can significantly improve the physical, rheological, and sensorial properties of cakes [11].

HPMC is a cellulose derivative component obtained by reacting alkaline cellulose with methyl chloride and propylene oxide. It enhances emulsion stability and maintains viscosity at acidic conditions and [12]. Due to its film-forming ability and surface activity, HPMC is widely used in food products. Using Oleogels prepared with HPMC as solid fat substitutes improved structure and textural stability of food products [13]. Another cellulose derivative, carboxymethyl cellulose (CMC), is a linear anionic polymer with high

solubility and ability to form clear, viscous solutions in various foods. Combination of CMC and regenerated cellulose (RC) was used in the oleogel production and showed increasing RC concentration enhanced the emulsion's rheological properties and increased the storage (G′) and loss (G″) moduli of the resulting oleogels [14].

In the Literature review, hydrocolloids were used as gelators in oleogelation, but comparative evaluations of their structural and functional properties in oleogels are limited. In the present study, qualitative properties of seven hydrocolloids (AG, LBG, XG, GG, HPMC, CMC, CG) were evaluated and their effect as gelator on the textural, rheological, and thermal properties of the canola oil oleogels were evaluated. Canola oil was selected because of its favorable fatty acid profile, including balanced of omega-3 and omega-6 fatty acids. Due to the importance of preserving the fatty acids profile in food processing, canola oil oleogelation can produce solid fat–like structure without modifying the oil's fatty acid composition.

2-Materials and Methods

1.1. Materials

Cold-pressed canola oil produced by Behshahr Industrial Company was used as the oil phase for oleogel preparation. Seven different hydrocolloids were obtained from Sigma-Aldrich: locust bean gum, gum arabic, xanthan gum, guar gum, hydroxypropyl methylcellulose, carboxymethyl cellulose, and carrageenan. The selection of hydrocolloids was based on their structural properties, and functional behavior, after reviewing the literature and conducting preliminary screening tests.

1.2. Rheological properties of hydrocolloids

The rheological properties of the hydrocolloids were measured using a rotational rheometer (MCR 301, Anton Paar Instruments). A 3% (w/w) solution of hydrocolloid was prepared, and its flow behavior was evaluated in rotational mode at 25 °C over a shear rate range corresponding to 0.01–100% strain. The experimental data were fitted to the Power-law model (Equation 1) and the Herschel–Bulkley model (Equation 2) to obtain the rheological parameters of the sample [15].

$$\tau = k\dot{\gamma}^n \quad \text{(Equation 1)}$$

$$\tau = k\dot{\gamma}^n + \tau_o \quad \text{(Equation 2)}$$

In these equations, τ is the shear stress, k is the consistency index, $\dot{\gamma}$ is the shear rate, n is the flow behavior index, and τ_o is the yield stress.

1.3. Thermal properties of hydrocolloids

The thermal behavior of the hydrocolloids was analyzed using differential scanning calorimetry (DSC) (Q1000, TA Instruments, USA). Approximately 10 mg of sample was placed in flat-bottomed aluminum pans and heated from 5 to 100 °C at a heating rate of 5 °C/min [16].

1.4. Preparation of oleogels

First, 3 g of hydrocolloid was dispersed in 100 mL of distilled water and stirred on a magnetic stirrer for 20 min to obtain a 3% (w/w) hydrocolloid solution. The solution was stored at 4 °C for 24 h before use. 100 mL of canola oil, 2.5 g of Tween 80, and 2.5 g of mono-diglycerides was added to hydrocolloid and stirred on a magnetic stirrer. The emulsion was homogenized using a high-speed homogenizer for 3 min and cooled to 4 °C. The sample was frozen at −80 °C and subsequently freeze-dried for 18 h in a freeze dryer (model FD-5005-BT) at −70 °C to remove the water from the emulsion. The developed oleogels were coded according to the structuring hydrocolloids: gum arabic (O-AG), locust bean gum (O-LBG), xanthan gum (O-XG), guar gum (O-GG), hydroxypropyl methylcellulose (O-HPMC), carboxymethyl cellulose (O-CMC), and carrageenan (O-CG), and then used for further analyses [6].

1.5. Evaluation of oleogel quality attributes

1.5.1. Oil release

Oil release was determined by applying a centrifugal force. Five grams of oleogel were weighed into Falcon tubes and centrifuged at 9000 rpm for 10 min at 20 °C using a PIT320R centrifuge. The separated oil was removed by inverting the tubes for 5 min, and was weighed [6]. Oil release (%) was calculated using the following equation:

$$Oil \text{ release } (\%) = \frac{\text{released oil}}{\text{initial oil}} \times 100 \quad \text{(Equation 3)}$$

1.5.2. Texture firmness

The firmness of the oleogel was measured using a texture analyzer (Texture Profile Analysis, TPA; model STM20). 20 grams of sample were filled in the instrument's standard container and placed under a cylindrical probe with a diameter of 12.7 mm. A penetration test was performed at a speed of 60 mm/min to a depth of 5 mm using a 6 kg load cell. The maximum force required for probe penetration was recorded as the firmness of the sample [17].

1.5.3. Viscoelastic properties of oleogels

The viscoelastic behavior of the oleogels was evaluated in oscillatory mode. Frequency sweep test was carried out from 0.1 to 100 Hz at a constant strain of 0.1%. The storage modulus (G′), loss modulus (G″), complex modulus (G*), loss tangent (tan δ), and complex viscosity (η*) were determined [1].

1.5.4. Thermal behavior of oleogels

The thermal behavior of the oleogels was characterized by DSC following the method of Valoppi et al. [17] using a differential scanning calorimeter (DSC131-SETARAM, France). The samples were scanned from room temperature to 140 °C at a heating rate of 10 °C/min. The energy required for phase transition and the corresponding enthalpy value were calculated by integrating the area under the DSC curve using the software.

1.5.5. Infrared spectroscopy

Canola oil and oleogels were analyzed separately by Fourier transform infrared (FTIR) spectroscopy using an FT/IR-6300 spectrometer (Jasco, Japan). Spectra were recorded in the range of 7800–350 cm^{-1} at 25 °C [18].

1.5.6. Crystal structure

X-ray diffraction (XRD) was used to study the crystalline structure of oleogels according to the method of Aliasl Khiabani et al. [2]. The sample was placed on the holder, and the surface was leveled before analysis in a D8 ADVANCE diffractometer (Bruker, Germany). Diffraction patterns were collected over a 2θ range of 5–80° with a scan time of 1 s per step at room temperature.

1.6. Statistical analysis

The experiment was designed as a completely randomized design. All measurements were carried out in triplicate. Results are expressed as mean ± standard deviation. One-way analysis of variance (ANOVA) was used to compare means, and differences between means were determined using the least significant difference (LSD) test at a 95% confidence level in SPSS software version 27.0.1.

3-Results and Discussion

1.7. Rheological Properties of Hydrocolloids

The rheological behavior of hydrocolloids was evaluated through dynamic oscillatory measurement. As shown in Figures 1a and 1b, the storage modulus (G′) and loss modulus (G″) varied with frequency at a constant temperature. In most samples, G′ was higher than G″ across the tested frequency range, indicating a predominance of elastic or solid-like response. Therefore, the hydrocolloid dispersions formed weak gel-like structure under the applied test condition.

In samples, guar gum (GG) had the strongest viscoelastic behavior due to the highest values of both storage and loss moduli. In contrast, locust bean gum (LBG) had the lowest G′ and

G″ values, reflecting a comparatively weaker internal structure. A similar trend was observed for complex modulus (G*) and complex viscosity (η*), where GG and LBG showed the highest and the lowest value respectively (Figures 1c and 1d). The differences in rheological properties of the gums are due to their inherent physicochemical characteristics, including molecular weight, spatial structure, type and distribution of functional groups (hydrophilic or hydrophobic), charge density, solubility, and water binding capacity. One important structural factor is the mannose-to-galactose ratio within the galactomannan backbone. This ratio is approximately 2:1 in guar gum and about 4:1 in locust bean gum. Structural difference influences water solubility, chain flexibility, and rheological properties of the polysaccharides. The distinctive rheological behavior of guar gum is due to higher galactose substitution, which promotes better hydration and molecular dispersion in aqueous system. The unique rheological properties of guar gum are due to the presence of its side chains, that form irregular and extended spirals in aqueous medium. This structure traps water molecules and forms a stable three-dimensional network with strong hydrogen bonds [19].

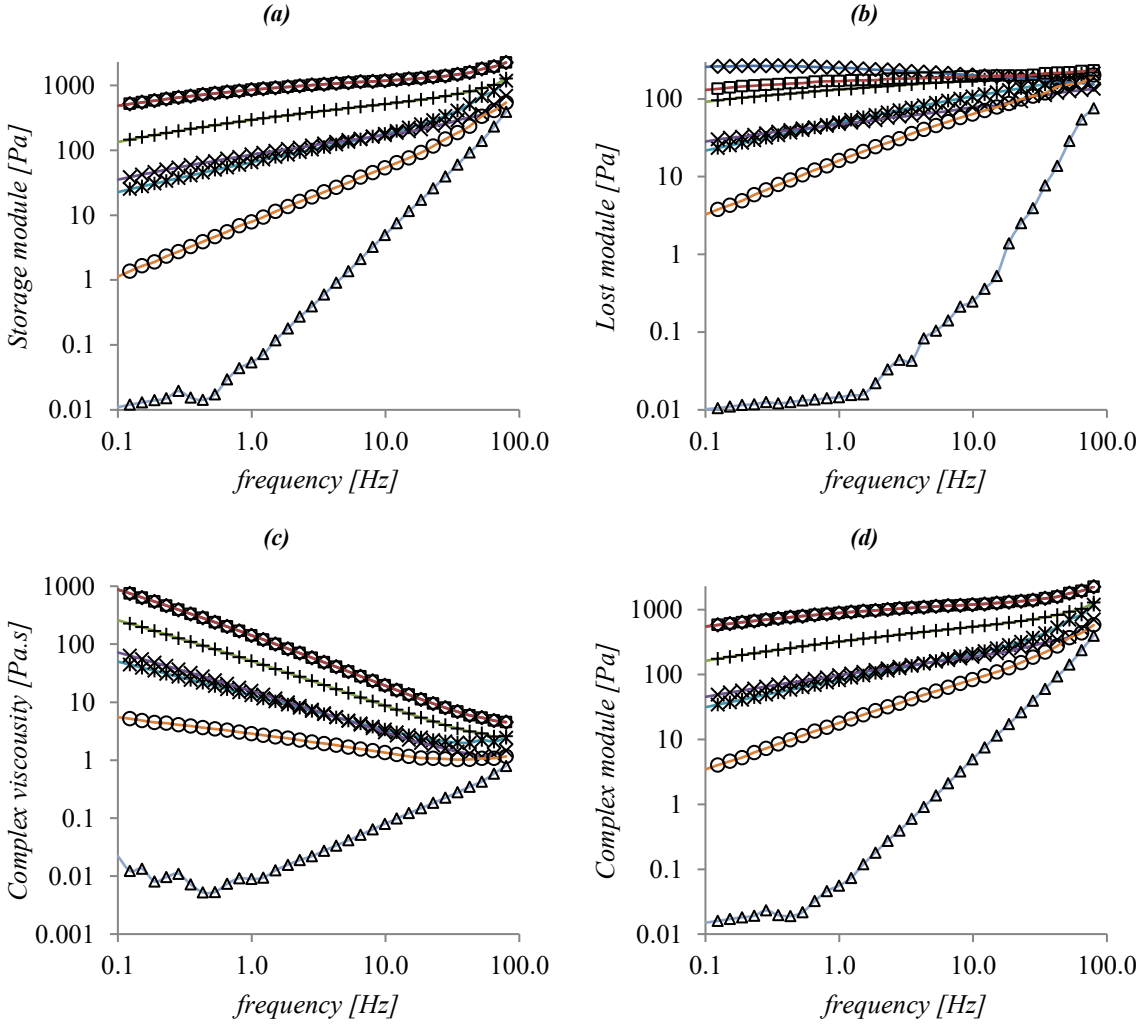


Figure (1) Rheological Properties of Different Hydrocolloids (a): Storage Modulus, (b): Loss Modulus, (c): Complex Viscosity, (d) Complex Modulus

(AG: Arabic Gum, LBG: Locust Bean Gum, XG: Xanthan Gum, GG: Guar Gum, HPMC: Hydroxypropyl methylcellulose, CMC: Carboxymethyl cellulose, CG: Carrageenan Gum)

1.8. Thermal Properties of Hydrocolloids

Thermal characteristics of the hydrocolloids are summarized in Table 1. CMC with the highest melting enthalpy (2813 J/g), had the greatest thermal stability and energy requirement for structural transition. In contrast, GG had the lowest melting enthalpy (1082 J/g). The lowest melting temperature (83 °C) and the weakest thermal resistance was observed in LBG. Variations in molecular and structural features of the hydrocolloids explain the differences in thermal properties of them. Molecular rigidity or flexibility, particle size, chain conformation, and the functional groups, particularly charged or sulfate groups, have important effect on thermal properties of starch [20].

Table (1) Differential Scanning Calorimetry of Hydrocolloids

Sample	Melting Point (℃)	Enthalpy (J/g)
AG	99.291^{c}	1961.203^{b}
LBG	83.283^{e}	1853.052^{c}
XG	98.426^{c}	1600.329^{d}
GG	113.203^{a}	1081.994^{e}
HPMC	95.248^{d}	1928.821^{b}
CMC	98.971^{c}	2813.354^{a}
CG	100.656^{b}	1822.169^{c}

(AG: Arabic Gum, LBG: Locust Bean Gum, XG: Xanthan Gum, GG: Guar Gum, HPMC: Hydroxypropyl methylcellulose, CMC: Carboxymethyl cellulose, CG: Carrageenan Gum)

1.9. Firmness and Oil Loss of Oleogels

The oil-binding capacity of oleogels is presented in Figure 2a. The highest oil release (81%) was observed in the LBG-based oleogel (O-LBG), while the lowest oil release (20%) was recorded for the carrageenan-based sample (O-CG). Hydrocolloids as gelators create a strong three-dimensional continuous gel network with multiple bonds and suitable molecular spatial arrangement that influence the oil retain ability of the produced oleogel [16], [21], [22].

In the Textural analysis (Figure 2b), the highest firmness was observed in the oleogel prepared with gum arabic (O-AG, 15.3 N), whereas the lowest firmness was shown in the LBG-based oleogel (O-LBG, 4.4 N). The formation of a dense and well-connected gel network through intermolecular interactions increased mechanical strength significantly. Differences in molecular weight, intrinsic viscosity, and chain conformation of hydrocolloids are the main reasons of their varied performance as gelators and the resulting differences in textural properties of oleogels [16], [23].

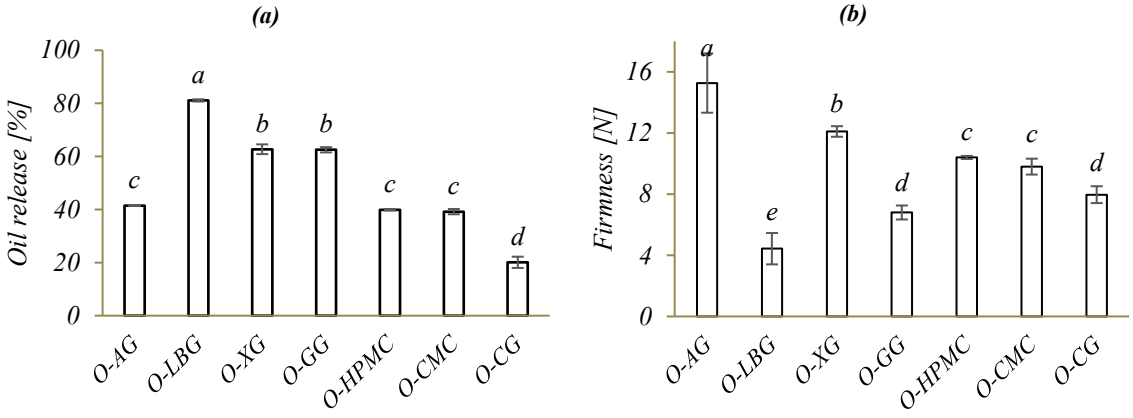


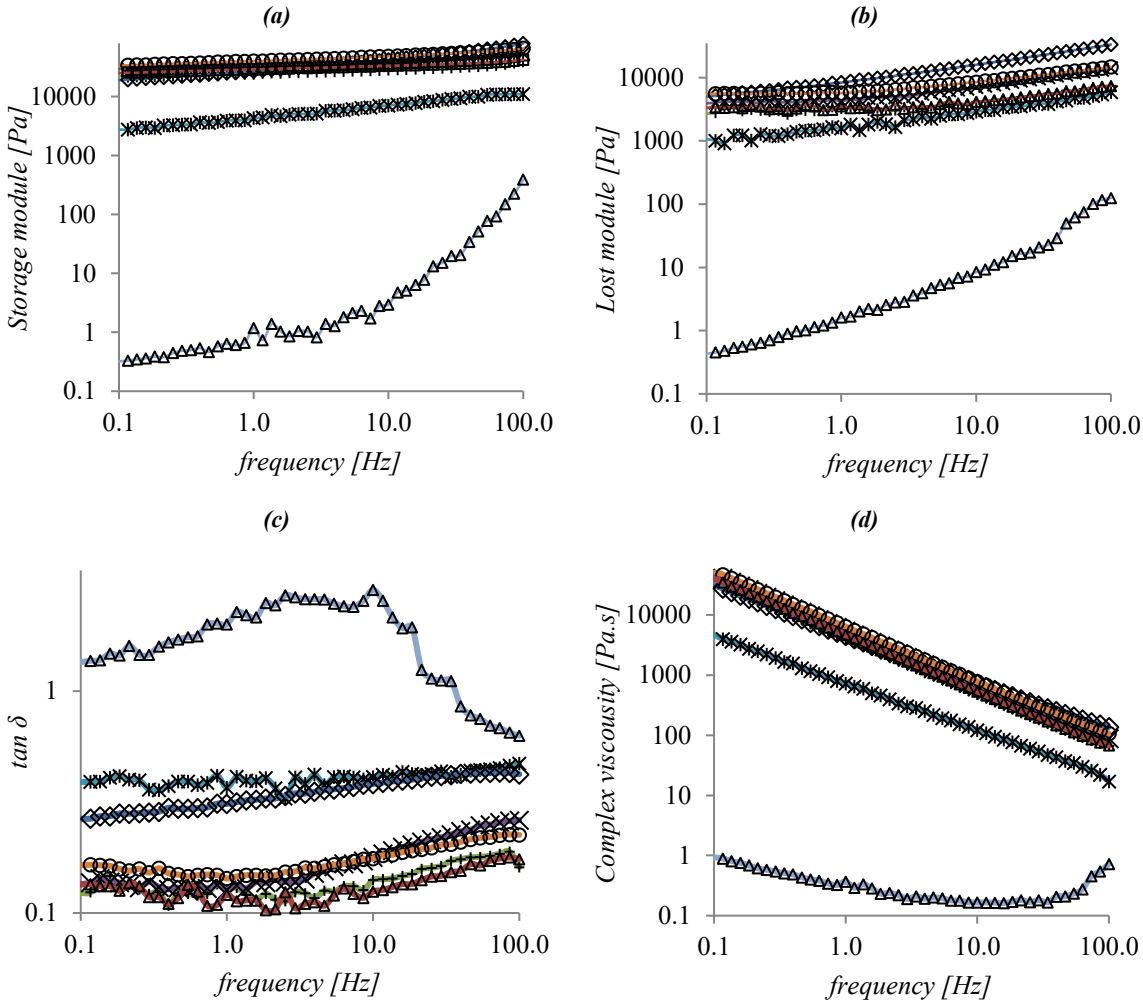
Figure (2) Effect of Hydrocolloids on (a) Oil release, (b) Firmness of Canola Oil Oleogels

(Oleogels with O-AG: Arabic Gum, O-LBG: Locust Bean Gum, O- XG: Xanthan Gum, O-GG: Guar Gum, O-HPMC: Hydroxypropyl methylcellulose, O-CMC: Carboxymethyl cellulose, O-CG: Carrageenan Gum)

(Samples with different lowercase letters indicate significant differences at the 95% confidence level)

1.10. Rheological Properties of Oleogels

Dynamic rheological behavior of the oleogels (O-AG, O-LBG, O-XG, O-GG, O-HPMC, O-CMC, and O-CG) is illustrated in Figures 3a and 3b, storage modulus (G′) and loss modulus (G″) respectively. G′ and G″ increased with increasing frequency due to frequency-dependent viscoelastic behavior of samples. G′ was higher than G″ in oleogels, indicating the solid-like viscoelastic properties of them. The highest values of tan δ, reflecting a greater contribution of viscous to elastic moduli, was observed in O-LBG. Oleogels with high elastic modulus have strong and more stable intermolecular interactions in the linear viscoelastic region [24]. Complex viscosity (η*) and complex modulus (G*) of the oleogels are shown in 3d and 3e Figures. With increasing frequency, the complex modulus increased while complex viscosity decreased in oleogels due to non-Newtonian, shear-thinning behavior, which is commonly observed in structured gels. O-LBG had the lowest values of both complex viscosity and modulus than the other oleogels, confirming its weaker structural integrity. Differences in the rheological properties of polymer-structured oleogels are attributed to variations in polymer chain architecture, molecular complexity, chain length, and molecular weight of the used polymer as gelator [16], [25]. Davidovich-Pinhas et al. [26] reported the rheological properties of ethyl cellulose (EC)-based oleogels are strongly influenced by polymer molecular weight, oil type, oxidation level, and polymer concentration. High molecular weight EC forms strong and stable gels, and increasing EC concentration increases storage (G′) and loss (G″) moduli. On the other hand, increasing molecular weight enhances gel rigidity and structural strength. Hashemi et al. [27] reported different oleogelators form distinct network structures with significant different in rheological properties. Candelilla wax forms small crystals and a dense network, leading to a higher elastic modulus in oleogels. Increasing rice bran wax concentration enhances crystal density and promotes the formation of cohesive network, that enhance hardness and adhesiveness of samples. In contrast, needle-like crystals formed in beeswax-based oleogels weaken viscoelastic properties of samples. A high content of wax esters in sunflower wax promotes the formation of firm and adhesive gels. The findings show oleogelators with different chemical compositions, chain lengths, and intermolecular interactions have significant effect on rheological properties of the produced oleogels.


(a)
Storage module [Pa]
10000
1000
100
10
1
0.1
0.1
1.0
10.0
100.0
frequency [Hz]
(b)
Lost module [Pa]
10000
1000
100
10
1
0.1
0.1
1.0
10.0
100.0
frequency [Hz]
(c)
tan δ
1
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frequency [Hz]
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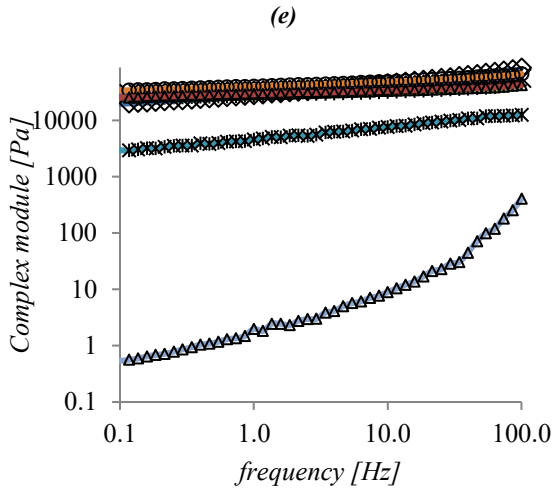

Figure (3) Rheological Properties of Different Oleogels

(a): Storage Modulus, (b): Loss Modulus, (c): tanδ, (d): Complex Viscosity, (e): Complex Modulus

(O-AG: Arabic Gum Oleogel, O-LBG: Locust Bean Gum Oleogel, O-XG: Xanthan Gum Oleogel, O-GG: Guar Gum Oleogel, O-HPMC: Hydroxypropyl Methylcellulose Oleogel, O-CMC: Carboxymethylcellulose Oleogel, O-CG: Carrageenan Gum Oleogel)

O-AG O-LBG O-XG O-GG O-HPMC O-CMC O-CG

1.11. Thermal Properties of Oleogels

One to five endothermic peaks were shown in the thermal profile of the oleogels (Table 2). The presence of multiple peaks indicates differences in crystallization behavior, melting transitions, as well as the formation of various crystalline polymorphic in the oleogels [15]. The xanthan-based oleogel (O-XG) had five distinct endothermic peaks, suggesting the presence of crystalline domains with different melting requirements. In the samples, the melting temperature of the first peak ranged from 25 to 31 °C, while the second peak appeared between 58 and 108 °C. In most cases, the enthalpy associated with the first peak was higher than that of the second, indicating a greater energy contribution from the primary crystalline fraction. O-XG had the highest melting temperature (127.1 °C) and melting enthalpy (20.41 J/g). Its related to the presence of sulfate groups in the ordered superhelical structure, which promote strong intermolecular interactions and enhance structural stability. Furthermore, prepared oleogels with guar and locust bean gum had higher melting temperatures compared the carrageenan-based oleogels. With considering sum of enthalpies, the oleogel prepared with gum arabic required the highest energy for melting. It's related to differences in chemical composition, molecular weight, solubility, functional groups, and the hydrocolloid interactions properties in the gel network [28].

Table (2) Thermal Properties of Different Oleogels

	Peak #1		Peak #2		Peak #3		Peak #4		Peak #5		
Sample	Melting Point (℃)	Enthalpy (J/g)	Melting Point (℃)	Enthalpy (J/g)	Melting Point (℃)	Enthalpy (J/g)	Melting Point (℃)	Enthalpy (J/g)	Melting Point (℃)	Enthalpy (J/g)	Enthalpy (Total) (J/g)

O-AG	30.25	41.49	107.91	1.74							43.23
O-LBG	31.19	21.54	107.23	0.21							21.75
O-XG	28.55	20.41	60.00	0.16	103.15	3.45	119.38	0.67	127.10	2.62	27.31
O-GG	29.76	20.82	65.24	0.19	72.82	0.86	81.62	0.59			22.46
O-HPMC	29.33	23.82	61.08	0.59							24.41
O-CMC	28.52	37.85	60.14	0.49	91.52	0.44	103.98	2.83			41.61
O-CG	25.73	26.75	58.07	1.01	116.13	0.81					28.57

(O-AG: Arabic Gum Oleogel, O-LBG: Locust Bean Gum Oleogel, O-XG: Xanthan Gum Oleogel, O-GG: Guar Gum Oleogel, O-HPMC: Hydroxypropyl Methylcellulose Oleogel, O-CMC: Carboxymethylcellulose Oleogel, O-CG: Carrageenan Gum Oleogel)

Changes in the molecular weight and the presence of hydrophilic groups such as hydroxyl influence gelator solubility in oil, crystallization kinetics, and ultimately thermal properties of samples [6,7,15]. Hashemi et al. [27] reported the thermal behavior of oleogels is varied due to difference in gelator chemical structure, oil type, intermolecular interactions, and crystal size. Long-chain compounds form suitable network with stronger intermolecular bonds and higher thermal stability compared to short-chain systems. In this regard, Davidovich-Pinhas et al. [26] reported the high molecular weight of ethyl cellulose improved crystalline organization, increased melting, glass transition temperatures, and energy requirements for phase transitions of structured organogels. The formation of small and uniform crystals enhances thermal stability of product and in the practical perspective, stable oleogels at high temperatures are suitable candidate for replacing conventional solid fats in food products.

1.12. Fourier Transform Infrared (FTIR) Spectroscopy

FTIR spectra of canola oil and oleogels (Figures 4 and 5) showed absorption bands in the high-energy region of 400–3500 cm^{-1}. No new functional groups were detected in the formed oleogels, and the spectra of oleogels were similar to that of pure canola oil, with differences observed mainly in peak intensity rather than peak position. Therefore, oleogelation don't induce chemical modifications but involve physical interactions of components. A weak band around 3473 cm^{-1} was attributed to O–H stretching vibrations, associated with intra- and intermolecular hydrogen bonding of polysaccharide chains [29]. After oleogelation, the intensity of this interaction decreased that the reduction followed the order: O-CG > O-AG > O-XG > O-LBG > O-GG > O-CMC > O-HPMC. A relatively strong band near 3007 cm^{-1} corresponded to =C–H stretching vibrations. Strong absorption peaks at approximately 2920 and 2854 cm^{-1} were assigned to asymmetric and symmetric stretching vibration of aliphatic =CH(CH2) groups, respectively. Another intensified interaction at 1746 cm^{-1} was attributed to C=O stretching of ester group. Additional peaks were observed at 1654 cm^{-1} (cis C=C bending), 1462 cm^{-1} (–CH(CH2) bending), 1375 cm^{-1} (symmetric –CH(CH3) bending), and 1237 cm^{-1} (C=O stretching). A medium-intensity band at 1164 cm^{-1} corresponded to C–O stretching, while weak bands at 965 cm^{-1} and 723 cm^{-1} were associated with trans and cis –CH=CH– bending vibrations, respectively. The weak signals were related to trans –CH=CH bending (~968 cm^{-1}), ester stretching (~1092 cm^{-1}), carbonyl stretching (~1238 cm^{-1}), and methyl C–H bending (~1375 cm^{-1}). In contrast, the strongest absorptions were observed in methylene C–H bending (~1745 cm^{-1}) and C–H stretching vibrations at 2854 and 2925 cm^{-1}. The increase in these bands intensity in oleogels than the pure canola oil is due to enhanced non-covalent interactions, including hydrogen and van der Waals interactions in the oleogel

components. The interactions promote the formation of inclusion complexe and semi-crystalline structure in the oleogels. The bands at 2920, 2854, and 1746 cm^{-1} showed particularly strong intensity, reflecting the dominant role of hydrogen and van der Waals interactions in network formation. The interactions were strong in O-CG, followed by O-XG, and weak in O-LBG.

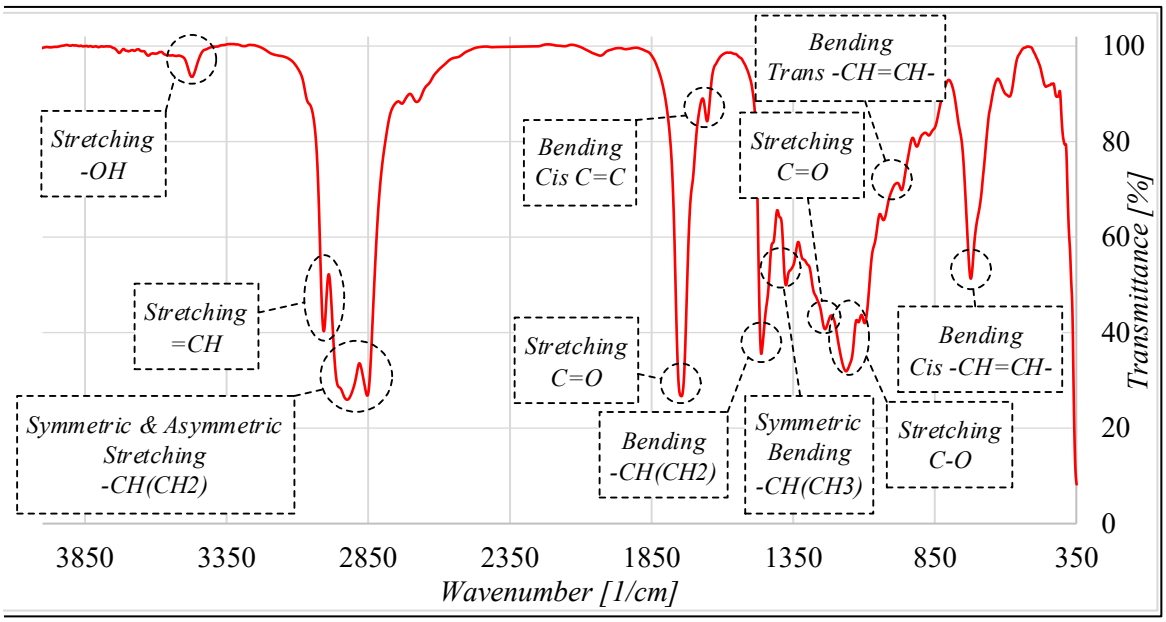

Figure (4) Schematic of Functional Group Positions in Canola Oil

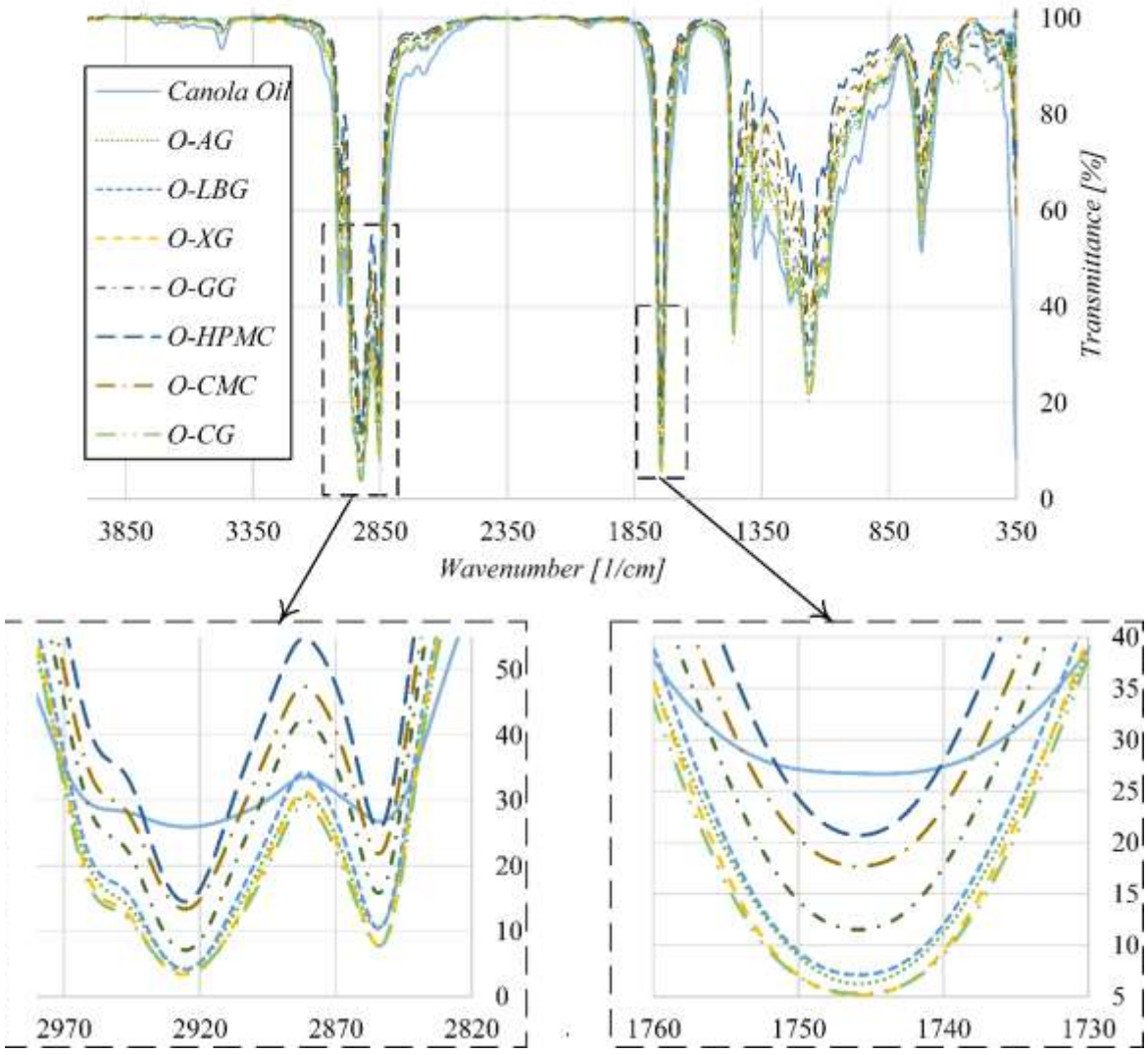

Figure (5) FTIR Spectra of Canola Oil and Oleogels Formulated with Different Hydrocolloids

(O-AG: Arabic Gum Oleogel, O-LBG: Locust Bean Gum Oleogel, O-XG: Xanthan Gum Oleogel, O-GG: Guar Gum Oleogel, O-HPMC: Hydroxypropyl Methylcellulose Oleogel, O-CMC: Carboxymethylcellulose Oleogel, O-CG: Carrageenan Gum Oleogel)

Canola Oil *O-AG* *O-LBG* *O-XG*
O-GG *O-HPMC* *O-CMC* *O-CG*

1.13. Crystalline Structure

The X-ray diffraction (XRD) patterns of pure canola oil and the prepared oleogels are presented in Figure 6. XRD analysis provides valuable insight about the crystalline properties of lipid-based systems. Fatty acids can crystallize to three principal polymorphic forms: α (hexagonal), β′ (orthorhombic), and

β (triclinic). Melting point, density, packing order, and thermodynamic stability increase from α to β crystals [30,31]. β form crystals are large and coarse, whereas β′ crystals are finer than them and impart a smooth and creamy texture in food products. Therefore, in products such as margarine, shortening, and spreads, a higher proportion of β′ crystals than β is generally preferred to achieve desirable textural properties [32,33]. Prepared oleogels with gum arabic (O-AG), xanthan gum (O-XG), guar gum (O-GG), carboxymethyl cellulose (O-CMC), hydroxypropyl methylcellulose (O-HPMC), and carrageenan (O-CG) had mixed polymorphic structures with β′ and β crystals. While, only the β crystalline was observed in the LBG-based oleogel (O-LBG). Diffraction peaks observed in the range of 3.64–4.13 Å corresponded to β′ crystals, while other peaks in 4.47 to 4.55 Å were attributed to the β polymorph. The findings are consistent with previously reported patterns for structured oleogels [32,34]. Therefore, type of hydrocolloids changed the intensity of the diffraction peaks, due to differences in fatty acid packing and molecular organization under varying crystallization conditions.

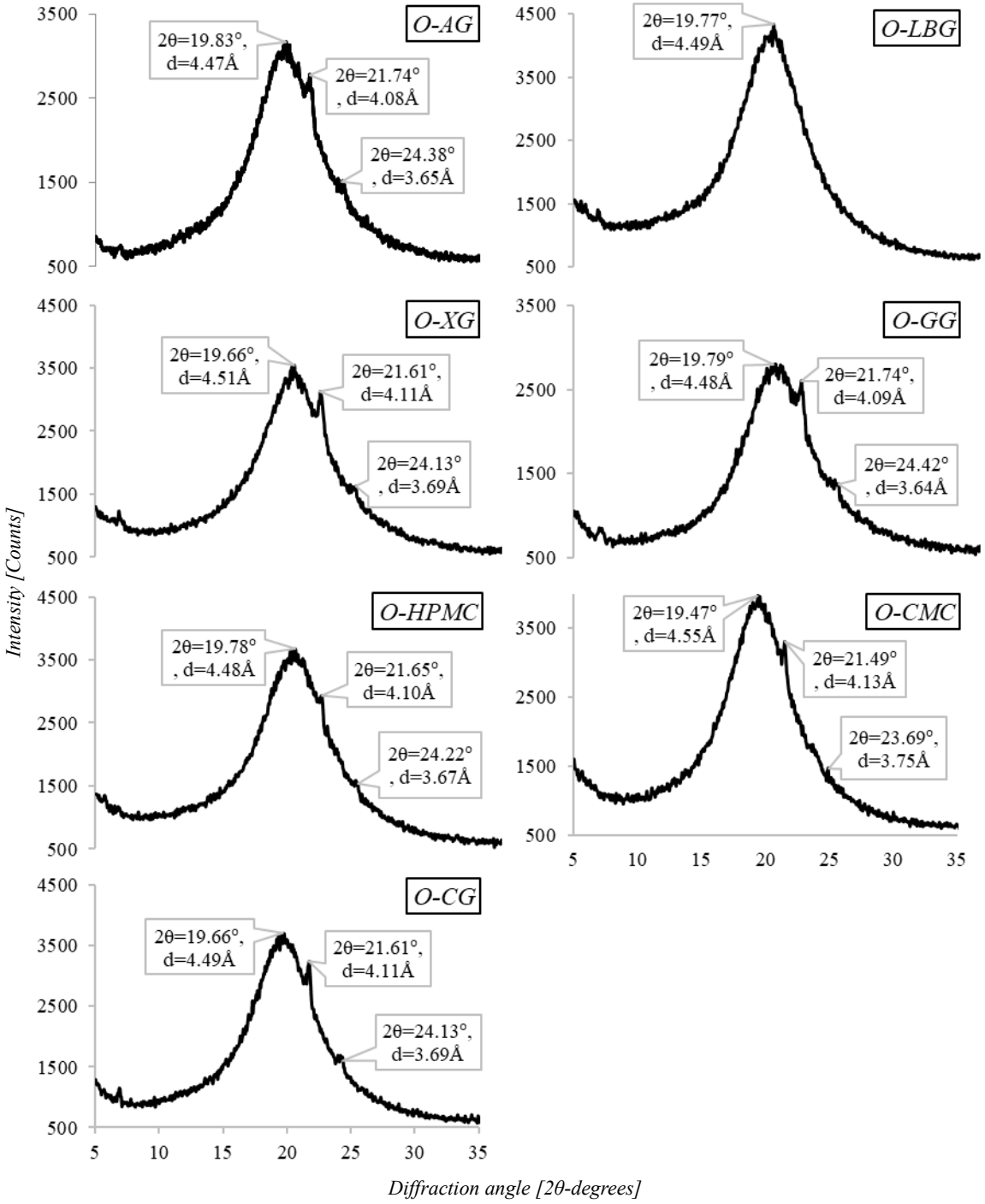

Figure (6) XRD Patterns of Canola Oil and Oleogels Formulated with Different Hydrocolloids

(O-AG: Arabic Gum Oleogel, O-LBG: Locust Bean Gum Oleogel, O-XG: Xanthan Gum Oleogel, O-GG: Guar Gum Oleogel, O-HPMC: Hydroxypropyl Methylcellulose Oleogel, O-CMC: Carboxymethyl Cellulose Oleogel, OCG: Carrageenan Gum Oleogel)

4-Conclusion

Type of hydrocolloids as a gelator has an important role on oil–polymer interactions and consequently, the structural network, rheological behavior, thermal stability, and crystalline characteristics of the oleogels. The

produced oleogel with gum arabic had the highest textural firmness and oil retention capacity, indicating formation of a well-developed and stable three-dimensional network. In contrast, the LBG-based oleogel had the weakest viscoelastic properties, the lowest elastic and viscous moduli, melting temperature, and enthalpy, reflecting limited structural integrity due to low suitability of LBG for oleogelation. FTIR analysis confirmed no new chemical bonds were formed in gelation, indicating the oleogel formation is driven primarily by physical interactions rather than chemical modification. The enhanced intensity of specific absorption bands such as hydrogen and van der Waals interactions have a major role on stabilizing the gel network. X-ray diffraction analysis showed the presence of mixed β′ (metastable) and β (stable) polymorphics in oleogels except O-LBG with the β form. The coexistence of β′ and β crystals is particularly desirable in food products requiring balanced structural stability and favorable textural properties. Consequently, structured oleogels with hydrocolloids have strong potential as alternatives to conventional solid fats in the development of food products with reduced saturated and trans fatty acid content. Further research should explore on other hydrocolloids and their combination with other biopolymers for enhancing functional performance. They can be used as a protective, delivery, and controlled release matrices for micronutrient, opening a new opportunity for improving the nutritional quality of formulated foods.

Data Availability

Data will be made available on request.

Conflict of Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Authors Contributions

Arezou Tavakoli Najafabadi: Data curation, Writing- Original draft preparation, Investigation, Visualization; Hajar Abbasi: Conceptualization, Methodology, Supervision, Validation, Writing- Reviewing and Editing.

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بررسی تأثیر به‌کارگیری هیدروکلوئیدهای مختلف به عنوان ژلاتور بر ویژگی‌های کیفی اولئوژل روغن کانولا

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چکیده

نظر به قابلیت ویژه پلیمرهای آب‌دوست در تشکیل اولئوژل‌ها، در این پژوهش، بررسی ویژگی‌های هفت هیدروکلوئید منتخب و بررسی تولنایی آن‌ها در تشکیل اولئوژل مورد نظر قرار گرفت. به این منظور اولئوژل‌های مبتنی بر روغن کانولا با استفاده از محلول‌های ۳٪ وزنی صمغ عربی *(AG)*، صمغ دانه لوکاست *(LBG)*، زانتان *(XG)*، گوار *(GG)*، هیدروکسی پروپیل متیل سلولز *(HPMC)*، کربوکسی متیل سلولز *(CMC)* و کاراگینان *(CG)* تهیه شدند. قابلیت حفظ روغن، سفتی بافت، رفتار حرارتی، خصوصیات رئولوژیکی، ساختار کریستالی و طیف مادون قرمز اولئوژل‌های تولید شده مورد ارزیابی قرار گرفتند. تمامی اولئوژل‌ها رفتار ویسکوالاستیک جامد از خود نشان دادند و مدول‌های الاستیک و کمپلکس آن‌ها با افزایش فرکانس افزایش و ویسکوزیته کمپلکس آنها کاهش یافت که نشان از رفتار رقیق‌شونده با برش دارد. در این میان، صمغ دانه لوکاست و اولئوژل تهیه شده از آن ضعیف‌ترین ویژگی ویسکوالاستیک و بیشترین میزان نشت روغن را دارا بود. در پروفلیل حرارتی اولئوژل‌ها، یک تا پنج قله لندوترمیک مشاهده شد که نمایانگر ویژگی‌های تبلوری متفاوت اولئوژل‌ها و تشکیل پلی‌مورف‌های مختلف در آنهاست. اولئوژل حاوی *AG* بالاترین و نمونه دارای *LBG* کمترین سفتی بافت و آنتالپی ذوب را دارا بود. ارزیابی ساختارهای کریستالی نشان داد که همه نمونه‌ها به جز اولئوژل حاوی *LBG* دارای ترکیبی از کریستال‌های نیمه پایدار و پایدار β' و β هستند. تغییر شیمیایی قابل توجهی در ساختار نمونه‌ها مشاهده نگردید ولی افزایش شدت پیوندهای هیدروژنی و واندروالسی پس از تشکیل اولئوژل تأیید شد. درمجموع نوع هیدروکلوئید تأثیر به‌سزایی بر کنش‌های روغن-پلیمر و ویژگی‌های عملکردی اولئوژل‌ها نشان داد. *LBG* با ضعیف‌ترین خواص ویسکوالاستیک و دمای ذوب برای اولئوژلاسیون مناسب ارزیابی نشد، درحالیکه اولئوژل‌های تهیه شده بر پایه روغن کانولا با *AG*، و پس از آن *HPMC* و *CMC* با ظرفیت اتصال روغن، استحکام و خواص ویسکوالاستیک مناسب برای کاربرد در محصولات غذایی با محتوی اسیدهای چرب اشباع و ترانس اندک مناسب بودند.