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UTILIZATION OF SUNFLOWER WAX OLEOGELS AS COATING MATERIALS TO EXTEND THE SHELF-LIFE OF FRESH MANDARIN FRUITS

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ABSTRACT
This study aimed to coat mandarin fruits with sunflower oil: sunflower wax oleogel (SOO) and sunflower oil: limonene: sunflower wax (SOLO) oleogel. The peak melting temperatures and storage modulus values of the SOO and SOLO were 61.78 and 59.78 ^{o}C, and 110 and 130 kPa. After storage, L colour values (brightness) of the samples were 28.27, 55.80, and 56.05 for the control, SOO and SOLO-coated samples. Total weight loss values of 49.51%, 38.09%, and 26.72% were recorded after the storage. Total aerobic viable counts were 100, 50, and 3750 CFU/g for the control, SOO, and SOLO-coated samples. After storage, the total soluble solids were 5.55%, 6.17%, and 7.65% for the same samples. Fresh fruit had 14.9% total soluble solids. Overall, oleogel coating, especially limonene-containing oleogel, provided significant protection against colour change, weight loss and microbial activity. Further studies with different oleogels and oleogels with lower oleogelator concentrations are suggested.
Keywords: Mandarin, oleogel, coating, colour, weight loss, microorganism, shelf-life

TAZE MANDALİNA MEYVESİNİN RAF ÖMRÜNÜ UZATMAK İÇİN AYÇİÇEK MUMU OLEOJELLERİNİN KAPLAMA MATERYALİ OLARAK KULLANIMI

ÖZ
Bu çalışmanın amacı, mandalina meyvesini ayçiçek yağı: ayçiçek mumu oleojeli (AAO) ve ayçiçek yağı: limonen: ayçiçek mumu oleojeli (ALAO) ile kaplamaktır. AAO ve ALAO'nun ergime pik noktaları ve depo modül değerleri 61.78 ve 59.78 ^{o}C ve 110 ve 130 kPa olarak ölçülmüştür. Bir aylık depolama sonunda mandalina örneklerinin L renk değerleri (parlaklık) kontrol, AAO-kaplanmış ve ALAO-kaplanmış için 28.27, 55.80, ve 56.05 olarak ölçülmüştür. Toplam ağırlık kayıpları da depolama sonunda %49.51, %38.09, ve %26.72 olarak belirlenmiştir. Aynı örnekler için toplam aerobik sayım değerleri ise 100, 50, ve 3750 CFU/g olarak belirlenmiştir. Depolama sonunda toplam çözünür katılar %5.55, %6.17, ve %7.65 olarak ölçülmüştür. Sonuç olarak oleojel kaplama ve özellikle limonen içeren kaplamanın renk değişimi, ağırlık kaybı ve mikrobiyal aktiviteye karşı önemli koruma sağladığı ortaya konulmuştur. Farklı oleojeller ve daha düşük oleojeletör konsantrasyonuyla hazırlanan oleojeller ile daha ileri araştırmalar önerilmiştir.
Anahtar kelimeler: Mandalina, oleojel, kaplama, renk, ağırlık kaybı, mikroorganizma, raf-ömür

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INTRODUCTION

Mandarin (*Citrus reticulata*), also known as mandarine or tangerine, is a small, rounded fruit belonging to the citrus family (*Rutaceae*). It is a non-climacteric and perishable fruit, which makes it unsuitable for prolonged transit and storage. Mandarin fruits may be preserved for 1 to 2 weeks after harvest, depending on temperature and humidity (Rokaya et al., 2016). Mandarin is one of Turkey's predominant citrus crop producing 1.865.000 tons in 2022, and being third order in the world behind China and EU (Aygören, 2023).

Weight loss and fungal deterioration are the primary factors limiting the storage life of mandarins (Whangchai et al., 2010). Due to the chemical residues, the use of chemical disinfection to mitigate rotting has declined, and the need for ecologically sustainable preservation techniques has heightened (Hong et al., 2007). The use of edible coatings on fruits has garnered global interest, as these coatings safeguard perishables from spoilage by diminishing respiration, enhancing textural quality, preserving volatile flavour compounds, and inhibiting microbial growth (Nasrin et al., 2018). Coatings may be produced from several components, including carbohydrates (starch, cellulose, alginates), proteins (gelatin, whey protein, casein, zein), and lipids (waxes, oils, fats). Consequently, systems like hydrogels, oleogels, or their combination as bigels may serve as edible coverings (Kanelaki et al., 2022).

Oleogels are soft, solid or solid-like substances produced by incorporating an oleogelator into liquid oil under suitable processing conditions. Oleogelators having a crystalline phase are known to concurrently organize into supramolecular structures exhibiting highly asymmetric morphologies akin to fibres or platelets. The asymmetric shape enhances the contact area among self-assembled structures, ultimately resulting in oleogelation (Co & Marangoni, 2012; Ramezani et al., 2024). As far as we have reached, there has been no direct utilization of oleogels as coating materials for food products. Plant waxes and other lipid-based materials were used as composites in edible coatings. In one of them (Khorram et al., 2017), oranges were coated with gelatin, Persian gum and shellac. Shellac is a well-known oleogelator to prepare edible oleogels (Co & Marangoni, 2012). In another study (Motamedi et al., 2018), carnauba wax-nanoclay emulsion was used to coat a film on oranges. Likewise, β-sitosterol-corn oil oleogel was mixed with gelatin to control film water permeability (Xiao et al., 2019). Oyom et al. (2024) reported that oleogel-based coatings substantially reduced moisture loss and fat absorption in chicken nuggets, enhancing lightness (L) and a* values without notable alterations in pH or textural characteristics. Since oleogels have not been directly used as edible coatings for fruits, this study would be essential to point out another use of the oleogels.

To our knowledge, the use of oleogels for preserving mandarin quality has not been investigated yet. The aim of this research was to assess the effectiveness of oleogels composed of sunflower oil and sunflower wax in a 90:10 weight ratio and that composed of sunflower oil, limonene, and sunflower wax in an 80:10:10 ratio to preserve mandarin from spoilage during a 30-day storage period at room temperature.

MATERIALS AND METHODS

Materials

The mandarin fruits (Satsuma variety) used in this research were collected from a summer garden in the Edremit district (Balıkesir, Türkiye) and were promptly transferred to the laboratory. Sunflower oil (100 g product; 100 g fat, 10 g saturated fat, 33 g monounsaturated fat, 57 g polyunsaturated fat, 0 mg cholesterol, 0 g carbohydrate, 0 g protein) used for the preparation of oleogels was procured from Biryağ, Trakya Birlik Oil Factory (Tekirdağ, Türkiye). Sunflower wax (6607L), used as an organogelator, was acquired from Kahlwax Co. (Kalh et al., Trittau, Germany). Aromsa Besin Aroma Katkı Mad (Gebze, Kocaeli) provided liquid limonene. Hypet Media Plate Count Agar (PCA) Ready Medium, Hypet Media Plate Dextrose Agar (PDA) Ready Medium, and Sterile Dry Swab (Without Medium) components used for microbiological counts were acquired from DiaTek Diagnostic Products Technical Co.

(Istanbul, Türkiye). All additional chemicals, consumables, standards, and materials were purchased from Sigma Chem. Co. (St. Louis, USA), Merck (Darmstadt, Germany), and local enterprises.

Preparation of the oleogels as coating solutions
The two kinds of oleogels used for coating mandarin fruits were first made in batches of 500 grams. The first oleogel (SOO) was created using sunflower oil (SO) and sunflower wax (SW) oleogelator in a weight ratio of 90:10. In the second formulation (SOLO), sunflower oil, sunflower wax, and limonene (L) were combined in a weight ratio of 80:10:10. To manufacture the oleogels, the requisite quantity of sunflower oil was measured in a 1 L beaker, followed by the addition of the specified amounts of sunflower wax. The mixture was agitated in a water bath maintained at 80°C to guarantee the complete melting of the wax and its uniform integration with the oil. In the formulation incorporating limonene, the measured limonene was included in the mixture after the melting process and well blended. The resulting homogenous liquid mixture was removed from the water bath and allowed to cool to room temperature autonomously. The oleogels that solidified upon cooling to room temperature were stored in the refrigerator overnight. The oleogels can be observed in Fig. 1.

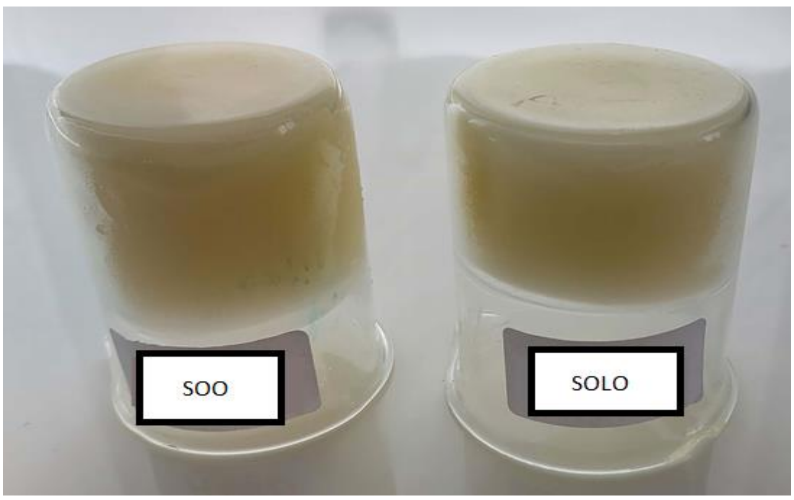


Fig. 1. The sunflower oil-sunflower wax oleogel (SOO) and the sunflower oil-limonen-sunflower wax oleogel (SOLO) used in mandarin coatings.

Thermal and rheological assessment of the prepared oleogels
The thermal characteristics of the oleogels produced were evaluated using a Perkin-Elmer 4000 Series Differential Scanning Calorimeter (DSC) (Groningen, The Netherlands). The equipment was once calibrated using Indium and Zinc. Approximately 5-7 mg of each sample was deposited into aluminum pans and sealed. The temperature protocol included heating samples from 20 °C to 100 °C at a rate of 10 °C/min for complete melting, chilling samples to - 30 °C at the same rate and maintaining that temperature for 3 minutes to ensure complete crystallization, followed by reheating the samples to 100 °C at a rate of 5 °C/min. This thermal cycling methodology, including our published findings, is often used in almost all oleogel research to ascertain crystallization and melting temperatures and enthalpies concurrently. The computations were performed using the Pyris 1 Manager Software (Yılmaz et al., 2021).

A DHR 2 rheometer (TA Instruments, USA) equipped with cross-hatched parallel plate geometry (ϕ = 40 mm, gap 0.9 ± 0.1 mm) and a Peltier system (± 0.1°C) underneath the bottom plate was used to evaluate the rheological parameters of the oleogel samples. Amplitude sweep tests (strain = 0.01–100%) were conducted at 10 °C with a frequency of 1 Hz for each oleogel sample to ascertain the linear viscoelastic region (LVR). The LVR was identified from the stress sweeps as a plateau in the storage (G') and loss (G'') modulus. Following the strain assessment inside the LVR, frequency sweep tests were conducted for each sample at 10 °C, with LVR strains identified by the amplitude sweep test (0.02–0.26%) and frequencies ranging from 0.1 to 100 Hz. A temperature ramp test was conducted from 0°C to 50°C at a rate of 1°C/min and a frequency of 1 Hz inside the linear viscoelastic zone. Each sample underwent three tests, and the findings were reported as average values (Yılmaz et al., 2021).

Coatings of mandarin fruits

Mandarins were randomly divided among three groups after washing and drying under tap water. The first set of samples was neither treated nor coated and served as the control treatment (C). A portion of the second sample group was coated by immersion in SOO (sunflower oil-sunflower wax oleogel). The third group was immersed in SOLO (sunflower oil-limonen-sunflower wax oleogel). The direct coating was administered by immersing the mandarins in each melted oleogel for about 10 seconds at 80 °C temperature, followed by one minute of drainage of excess coating before storage of the samples. The samples prepared were maintained at ambient temperature (20 ± 5°C) for one month. Measurements and analyses were conducted on 1st, 8th, 15th, 22nd, and 30th days of the storage period. Pictures of the samples were also taken during the storage period.

Mandarin analyses during the storage period

Peel hue

Five fruits were randomly picked from each group to assess the exterior (peel) colour diversity. The CIELab coordinates L* (lightness), a* (green-red component), and b* (blue-yellow component) were calculated as the mean of three measurements per fruit using a Minolta CR400 (Minolta, Japan). The L*, a*, and b* values were computed using the D65 illuminant by the CIELab scale (Strano et al., 2021).

Weight loss of the fruits

The reduction in fresh weight was calculated as a percentage by deducting the beginning weight from the final weight at designated sample intervals. Five fruits per replication were used for weight loss assessment (Ali et al., 2021).

Quantification of aerobic plate counts, total yeasts and molds

The aerobic plate counts (APC) and total yeasts and moulds (YM) for fresh mandarins were assessed using the methodology of Nam et al. (2019). Five grams of mandarin peels were shredded and combined with 45 mL of sterilized peptone water. Serial dilutions were prepared in peptone water and then plated on plate count agar and potato dextrose agar for aerobic plate count (APC) and yeast-mold (YM) enumeration, respectively (Difco Lab., Franklin Lakes, NJ, USA) for the enumeration of APC at 35 °C for 24 to 48 hours and YM at 25 °C for 5 to 7 days. The plates were incubated under the specified conditions. The colonies were quantified and represented as colony-forming units per gram (CFU/g). Plating and counting were conducted in triplicate to assure precision.

Assessment of total soluble solids (Brix), titratable acidity (TA), and pH

Randomly selected, four mandarin fruits were squeezed to get the fruit juice, and the juice was used for the pH and total soluble solid (°Brix) measurements. A pH meter probe (PB-11) was put into the juice, and the pH value was read and recorded several times. An Abbe 5 refractometer (Bellingham and Stanley, UK) calibrated against ultrapure water was used to read the Brix value of the fruit juice at room temperature several times. The titratable acidity of the juice sample was assessed according to Kashyap et al. (2020) using titration with 0.1 N NaOH, reaching a pH endpoint of 8.2, as observed by a pH meter. It was confirmed that the burette contained no air before the titration commenced. The beaker was continually agitated with an electronic stirring bar while NaOH was gradually introduced to the solution. The data was presented as a percentage of citric acid.

Statistical analysis

In this study, mandarin coatings were conducted two times, and for each coating, the listed analyses were completed in triplicate. The data provided were the means and standard deviations of six measurements. Comparison of the treatment groups was achieved by the Analysis of Variance with means comparison of Tukey's test. Minitab ver. 16.1 (Minitab, 2010) software was used with a 95% level of confidence.

RESULTS AND DISCUSSION

Thermal and rheological properties of the oleogels used as coatings

This study assessed two oleogels (SOO and SOLO) prepared and used for mandarin coatings for DSC-determined thermal properties (Table 1). In addition to the oleogels, the sunflower oil (SO) and sunflower wax (SW) used to create them were also analyzed. The melting peak temperatures of SO and SW were - 18.26 and 67.78 °C and were quite in agreement with the literature (Yılmaz et al., 2021). The SOO oleogel, including 10% of wax, had a 61.78 °C peak melting temperature, while the SOLO oleogel, including 10% of each SW and limonene, had a 59.78 °C peak melting temperature. The presence of limonene caused a slight reduction in peak melting temperature. Fully melted oleogels started crystalizing at 51.51 and 49.99 °C, respectively (Table 1). Eventually, both oleogels were solid at room temperature, and the coated mandarins were stored.

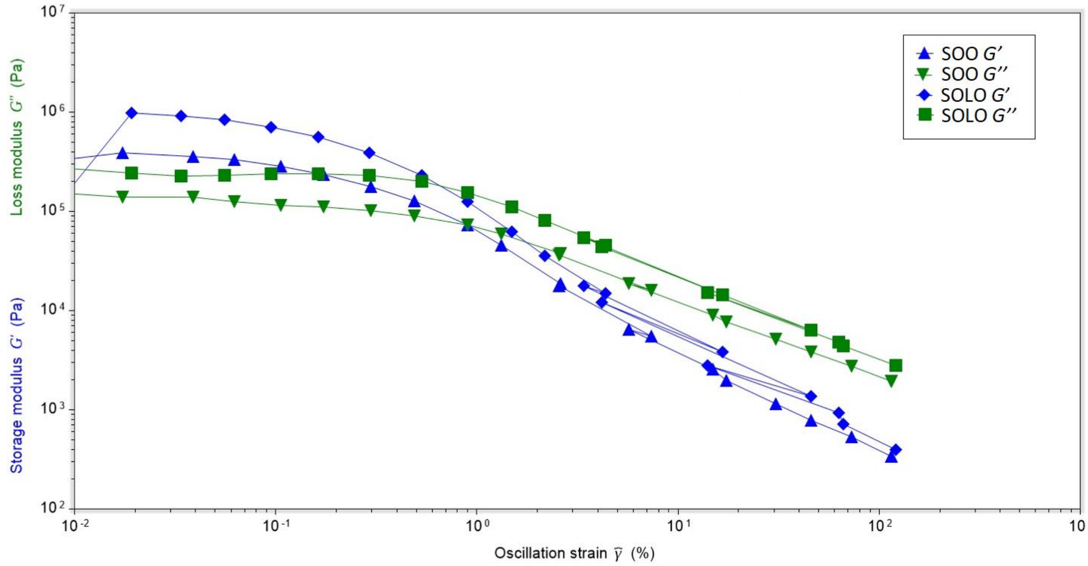
Table 1. Thermal properties of the sunflower oil, sunflower wax, and the oleogels (SOO and SOLO) used in mandarin coatings.

	Crystallization			Melting		
	$Onset_c$ (ºC)	Peak (Tc, ºC)	ΔHc (J/g)	$Onset_m$ (ºC)	Peak (Tm, ºC)	ΔHm (J/g)
Sunflower Oil (SO)	-39.30 ± 0.5^{d*}	-35.22 ± 0.5^{c}	1.39 ± 0.5^{d}	-16.54 ± 0.5^{c}	-18.26 ± 0.2^{c}	-2.30 ± 0.5^{a}
Sunflower Wax (SW)	69.31 ± 0.2^{a}	78.34 ± 0.4^{a}	201.65 ± 0.2^{a}	73.81 ± 0.5^{a}	67.78 ± 0.5^{a}	-206.94 ± 0.2^{d}
Sunflower Oil Oleogel (SOO)	51.51 ± 0.2^{b}	65.07 ± 0.1^{b}	16.04 ± 0.2^{c}	63.34 ± 0.0^{b}	61.78 ± 0.0^{b}	-16.52 ± 0.2^{b}
Sunflower Oil-Limonen Oleogel (SOLO)	49.99 ± 0.5^{c}	64.72 ± 0.5^{b}	19.54 ± 0.0^{b}	63.74 ± 0.5^{b}	59.78 ± 0.5^{b}	-19.49 ± 0.0^{c}

*Small letters within each column indicate significant differences among the samples for the mean ± SD values by one-way analysis of variance and Tukey's test ($p \leq 0.05$).

The amplitude and frequency sweep test graphics of the oleogel samples are presented in Fig. 2. The amplitude tests were completed first to determine the linear viscoelastic region (LVR, the non-destructive deformation range) of each sample. All further rheological measurements were done within the LVR strains per the requirement of correct rheology. The LVR strains were 0.1% and 0.15% for SOO and SOLO samples. Further, the sinusoidal response signals (phase-shift angle) were between 0^{o} and 45^{o} during the amplitude tests, indicating an actual gel-like state. The amplitude sweep tests also suggested that the gel structure could be destroyed at around 0.9% and 1.0% oscillatory strains for SOLO and SOO samples since the crossover point (G´ = G´´) was observed (Mezger, 2014).

To understand the time-dependent behavior of the oleogels at LVR, oscillatory frequency sweep tests are completed by stepwise enhancement of angular frequency from 0.1 rad/s to 1000 rad/s (Fig. 2). Practically, frequency sweep tests provide information about the inner structure and behavior, and long-term stability for the gels (Mezger, 2014). Both samples showed higher storage moduli (G´) values than loss moduli (G´´) values throughout the applied frequency range. Consequently, both samples had true-gel and stable gel structures within the range. The G´ component of gel viscosity indicates the solid-like properties of samples (elastic portion), while G´´ indicates the liquid-like (viscous portion) properties. Further, the G´´ / G´ value is named the loss (damping) factor and describes the viscoelastic behavior (Mezger, 2014). Consequently, the graphic in Fig. 2 shows that both samples had higher storage modulus (around 110 and 130 kPa) values than those of the loss modulus (around 100 and 101 kPa) for the SOLO and SOO samples, respectively. Both samples were clearly strong gels, and SOLO gel stiffness was slightly higher than the SOO sample. The limonene component might have created additional network structures or enhanced wax crystal networks. Similar phenomena were reported for other wax oleogels (Yılmaz et al., 2021).



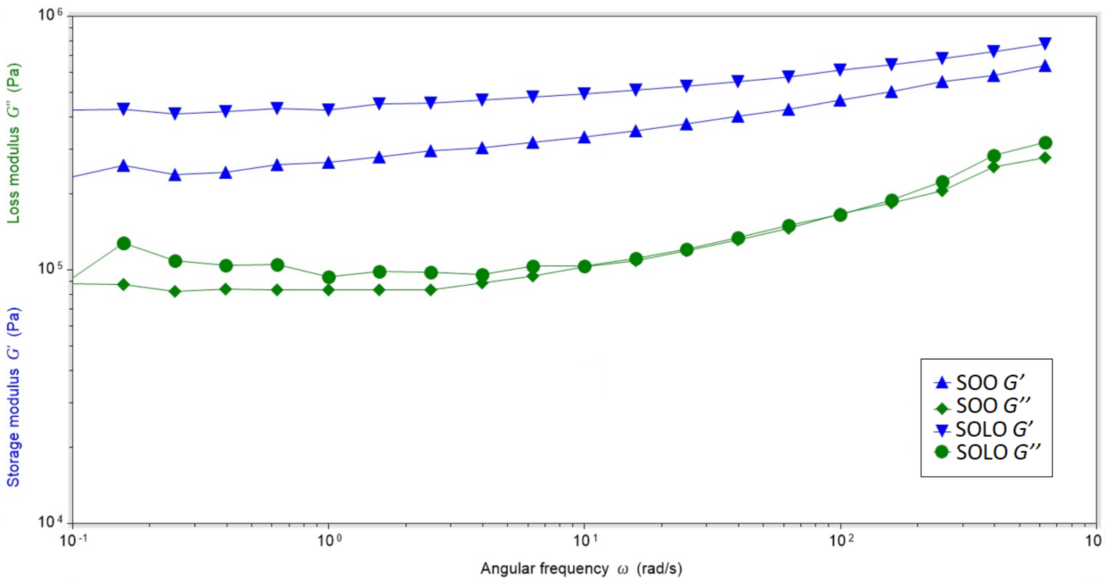


Fig. 2. The amplitude and frequency sweep graphics of the prepared oleogels used in mandarin coatings (SOO: sunflower oil-sunflower wax oleogel, SOLO: sunflower oil-limonen-sunflower wax oleogel).

Lastly, the temperature-ramp test graphics are given in Fig. 3. Both samples kept their gelled state intact from 5 to 50 ^{o}C, enhancing temperature, since within the range, the G´ value was always higher than the G´´ value. The SOO sample had around 140 kPa G´ value at 5 ^{o}C and decreased to 105 kPa at 50 ^{o}C. However, at 50 ^{o}C, the G´´ value was much lower at around 12 kPa. A similar situation existed for the SOLO sample, but at the endpoint (50 ^{o}C), it had a 130 kPa G´, proving that the SOLO sample was stiffer at even elevated temperatures. None of the samples showed a crossover point, keeping their actual gel structure up to 50 ^{o}C surrounding temperatures. Consequently, oleogel-coated mandarins would not have any melting and drainage problems at room temperature.

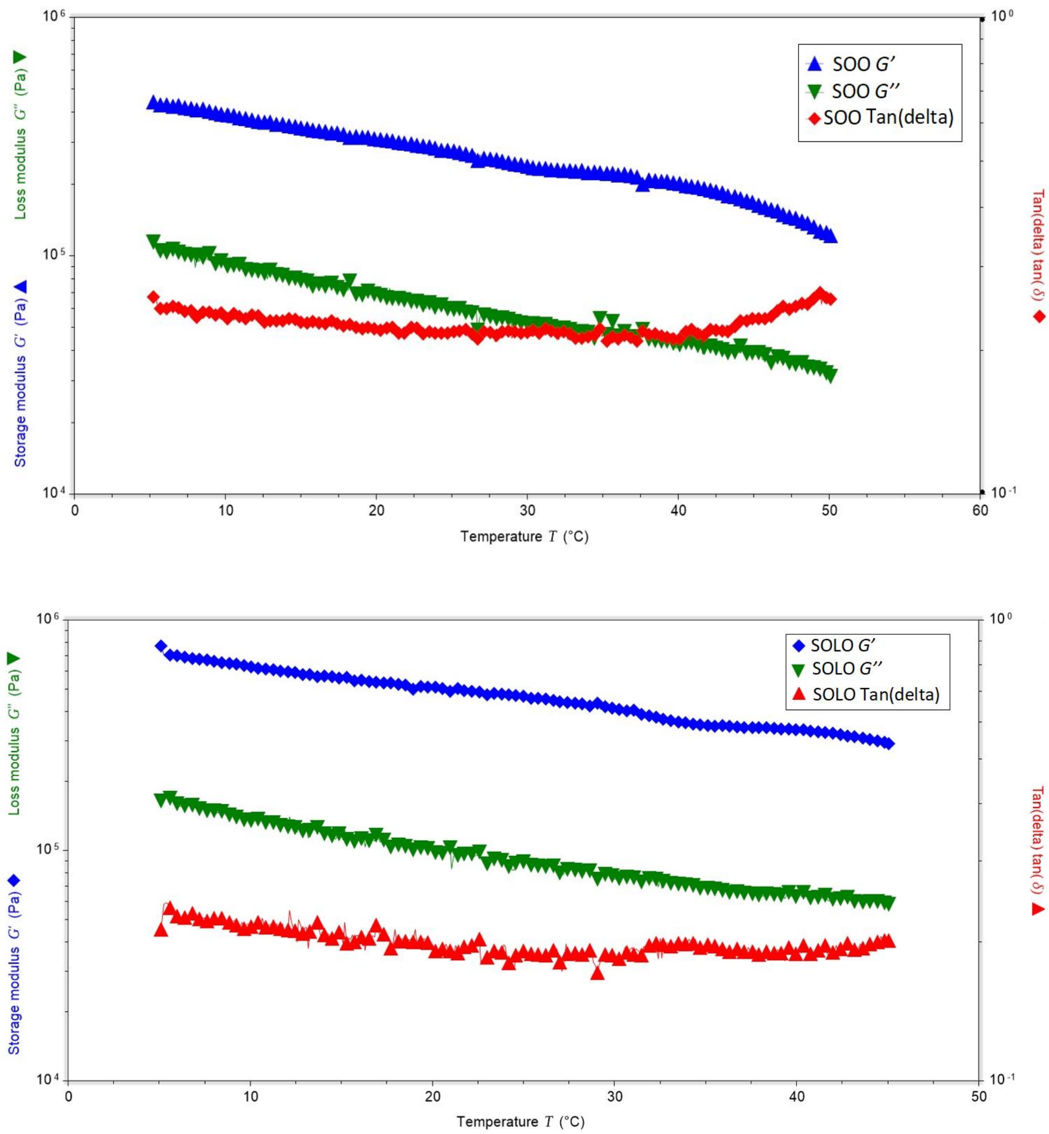


Fig. 3. The temperature ramp test graphics of the oleogels used in mandarin coatings (SOO: sunflower oil-sunflower wax oleogel, SOLO: sunflower oil-limonen-sunflower wax oleogel).

Colour values of the control and oleogel coated mandarins stored for a month

The development of fruit pigmentation is significantly influenced by environmental factors, including light and temperature; mandarin fruits also exemplify considerable variety in colour (Strano et al., 2021). The SOO and SOLO oleogels coated and control mandarins were stored for one month at room temperature (controlled by an air-conditioner strictly at 20 ± 5°C), and their pictures were taken on the 1st, 8th, 15th, 22th, and 30th days (Fig. 4). Further, the peel colour values (L*, a* and b*) were measured (Table 2). The surfaces of coated mandarins were clearly shiny on the first day due to the lipid-based coating. As the storage was prolonged, the oleogel-coated surface became dull due to the solidification of sunflower oil. Also, some crusting occurred. After 8 days, the control mandarins became darker, and their sizes were reduced, possibly due to excessive water loss (Fig. 4). The L* levels in mandarins generally decline across all groups during storage. This trend is more pronounced in control groups but is detected at diminished levels in coated samples. The diminishing impact of reduced L* values may be attributed to fruit dehydration (Hong et al., 2007) and an elevated respiratory rate (Bang et al., 2020) in control samples. The measured colour values agree with the observed facts in the picture (Fig. 4). The L* values of samples on the first day were 61.32, 66.28, and 78.25 for the control, SOO, and SOLO-coated samples, respectively. The fresh oleogel coating was bright. As storage time extended, samples' L* values decreased since the control sample had colour change by darkening, while oleogel-coated samples had a solid oleogel appearance dominating. All samples had positive a* values indicating red components of the colour. Likewise, positive b* values show the observed yellow colours of the mandarins. While the a* values were usually decreased during storage, the b* values were generally enhanced. Most importantly, there were some fluctuations in the colour values of the oleogel-coated samples. The reason for this was the crusting of the oleogel coating. After 8 days, the oleogel crusts started to crumble. These pictures and colour data proved that coated oleogel could be thinner with lower oleogelator concentration. Consequently, experiments with wax oleogelators that are less than 10 wt% used could be suggested. Overall, the oleogel-coated mandarin samples kept their surface colour and shape better than the control sample (Fig. 4). As the storage period lingered, extensive wrinkling and darkening occurred in the control samples. It was also essential to follow the weight changes in the samples.

Weight loss values of the control and oleogel coated mandarins stored for a month

The weight loss values of control and oleogel-coated mandarin samples were summarized in Fig. 5. The loss of moisture often results in observable symptoms of wilting and/or wrinkling in a fresh product, eventually causing considerable economic losses (Ali et al., 2021). The weight loss of mandarin fruits significantly increased from the 8th to the 30th days, regardless of the coating and control groups (Fig. 5). Nonetheless, the augmentation in fresh weight loss of mandarin fruits was much greater in the control group compared to the coated groups. There were significant differences among the samples on each measurement day from 8th days towards 30th days. During the storage days, the control sample always had higher weight loss values than the SOO and SOLO-coated samples. The weight loss values of 13.49%, 29.89%, 41.81% and 49.51% were recorded at the 8th, 15th, 22th, and 30th days of storage for the control sample. For all measurement days, these values were significantly higher than the values for SOO and SOLO coated samples (3.16%, 13.52%, 29.17%, 38.09% and 5.59%, 13.63%, 20.58%, 26.72%, respectively). Further, weight loss values were the lowest in SOLO-coated samples. SOLO coating prevented weight loss more efficiently, possibly due to slowing the water evaporation rate. It was previously stated that the peak melting temperature of SOLO was lower than that of the SOO (Table 1), but SOLO was stiffer than SOO (Fig. 2). Further, the SOLO sample had better temperature resistance once heated (Fig. 3). Also, it could be observed from Fig. 4 that SOLO coated mandarins had a little less crusting. Since it included limonene in addition to sunflower oil and wax, the stiffer and better coating ability might be due to the limonene. Further mechanistic studies can be suggested to clear this phenomenon. The reduction in fresh weight of coated fruits was likely reduced owing to the establishment of a semi-permeable barrier between mandarin fruits and their storage environment (Ali et al., 2021), as well as decreased rates of respiration and transpiration (Baswal et al., 2020). These operations aim to decrease moisture from fresh fruit, eventually compromising citrus fruit quality (Haider et al., 2020). Consistent with our findings, Khorram et al. (2017) observed that coating treatments substantially mitigated weight loss in mandarin fruits.

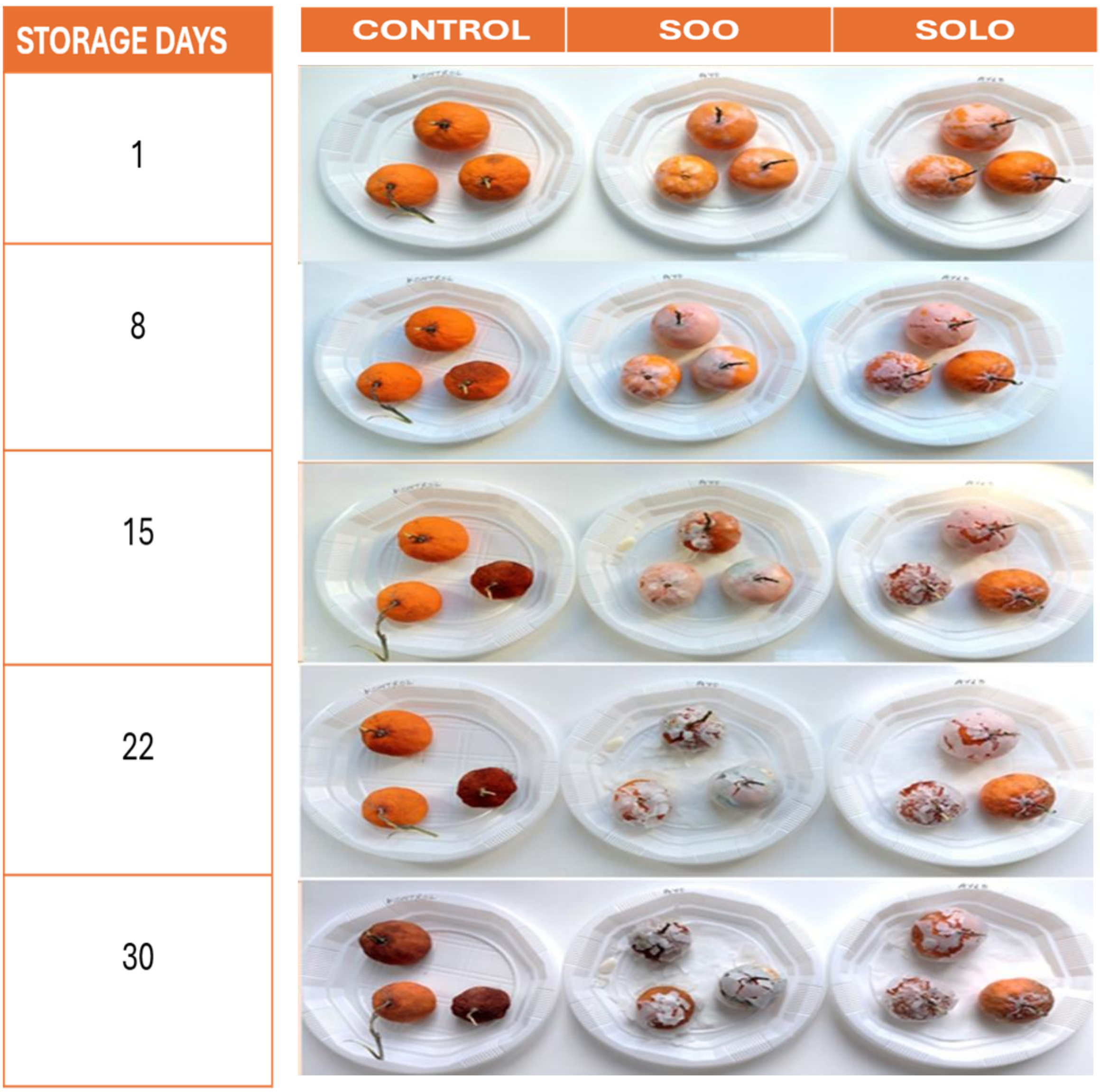


Table 2. The color values of the control and oleogel coated mandarin samples during one month storage period.

Storage Day	L* Value	a* Value	b* Value
	Control Mandarin		
1	61.32 ± 1.02[a*]	34.98 ± 0.90[a]	58.15 ± 3.24[a]
8	63.50 ± 5.45[a]	31.64 ± 7.50[b]	47.32 ± 9.63[a]
15	51.56 ± 8.55[b]	30.46 ± 8.25[b]	45.79 ± 6.25[b]
22	48.78 ± 4.95[b]	27.59 ± 9.30[bc]	39.38 ± 8.70[c]
30	28.27 ± 9.02[c]	28.10 ± 10.50[b]	36.59 ± 10.30[c]
	SOO Coated Mandarin		
1	66.28 ± 1.35[a]	25.02 ± 1.05[a]	41.96 ± 8.55[a]
8	58.44 ± 7.44[b]	15.18 ± 9.33[b]	24.47 ± 12.15[b]
15	56.99 ± 8.45[b]	17.68 ± 8.55[b]	23.09 ± 10.22[b]
22	45.95 ± 10.00[b]	9.78 ± 7.83[c]	14.93 ± 5.65[c]
30	55.80 ± 15.90[b]	10.83 ± 9.55[c]	20.75 ± 10.55[b]
	SOLO Coated Mandarin		
1	78.25 ± 3.70[a]	9.72 ± 3.20[b]	12.72 ± 6.34[b]
8	61.77 ± 4.90[b]	22.09 ± 7.63[a]	24.66 ± 20.03[a]
15	57.23 ± 6.00[c]	18.51 ± 10.04[a]	26.98 ± 16.83[a]
22	23.78 ± 3.15[d]	10.55 ± 2.55[b]	14.67 ± 2.80[b]
30	56.05 ± 5.80[c]	18.33 ± 10.10[a]	24.05 ±13.61[a]

*Small letters within each column indicate significant differences among the samples for the mean ± SD values by one-way analysis of variance and Tukey's test ($p \leq 0.05$).

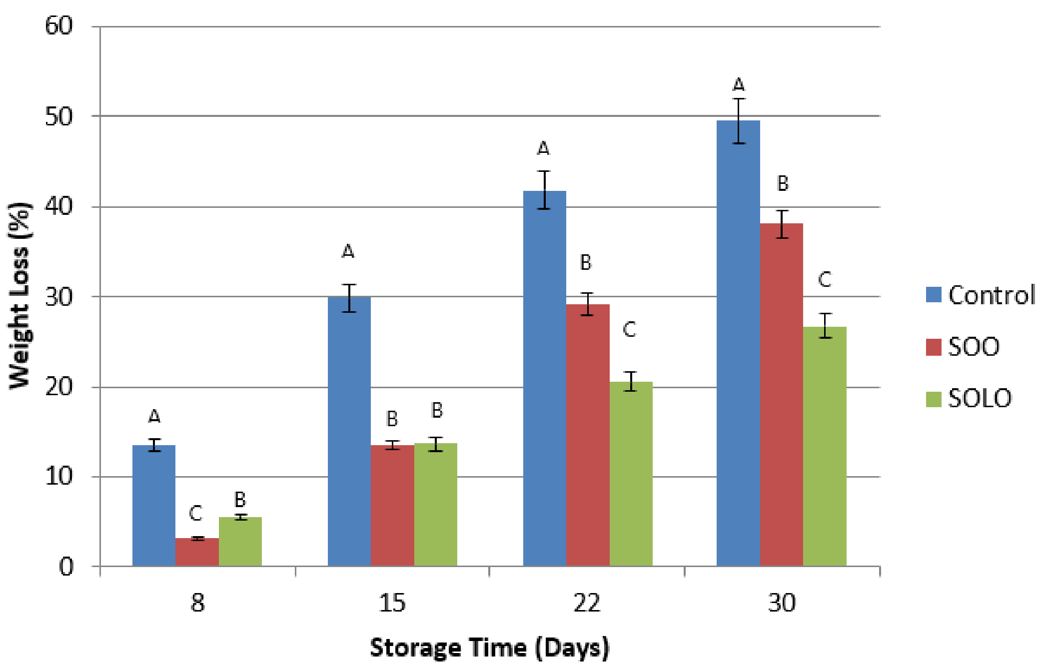


Fig. 5. The weight loss values of the control and oleogel coated mandarin samples during the storage period at room temperature (SOO: sunflower oil-sunflower wax oleogel, SOLO: sunflower oil-limonen-sunflower wax oleogel).

Aerobic plate counts, total yeasts and molds of the control and oleogel coated mandarins stored for a month

The microbiological quality of mandarins subjected to edible coatings during storage is shown in Table 3. Aerobic plate counts (APC), and yeasts and molds (YM) were quantified every 7 days throughout a 30-day storage period. The microbial burden of total viable count decreased at 8th and particularly on the 22nd days for the control group. The outer surface of the control group may have begun to desiccate, resulting in a reduced quantity of microorganisms during these drying phases. The total microbial load assessed from fruit peels indicated that the control group was statistically significant, differing from the other samples in all measures conducted at the various periods. SOO-coated samples had no microbial enumeration until the 22nd day and SOLO-coated samples had no count until the 30th days. Oleogel coating significantly enhanced the microbial quality of mandarins. As explained in the method sections, mandarins were dipped into melted oleogel at 80 ^{o}C and kept for 10 seconds before taking out and cooling to coat the surface. Even though the time was short, at 80 ^{o}C, a melted oleogel environment might kill all microorganisms. This operational benefit could be accounted for as a positive for oleogel coatings. Further, coating itself might slow down microbial proliferation due to limited oxygen access. Also, a much better protective effect of SOLO coating could be attributed to the limonene in the oleogel formulation. Limonen is well known for its anti-microbial activity (Riberio et al., 2023). Overall, oleogel coating for mandarins could be very beneficial in preventing microbial spoilage.

Table 3. The total viable, total fungi and yeast counts of the control and oleogel coated mandarins during the storage period.

Storage Day	Total Aerobic Plate Count (CFU/g)		
	Control Mandarin	SOO Coated Mandarin	SOLO Coated Mandarin
1	6800	-	-
8	4500	-	-
15	5000	-	-
22	2000^{a*}	50^{b}	-
30	3750^{a}	100^{b}	50^{c}
	Total Fungi/Yeast Count (CFU/g)		
1	Nct.	-	-
8	500000	-	-
15	150000	-	-
22	4000^{a}	50^{b}	-
30	4500^{a}	200^{b}	50^{c}

*Small letters within each row indicate significant differences among the samples for the mean ± SD values by one-way analysis of variance and Tukey's test ($p \leq 0.05$). Nct.: Countless

Total soluble solids (Brix), titratable acidity (TA), and pH of the fresh and control and oleogel coated mandarins stored for a month

Total soluble solids (TSS or Brix) are a crucial determinant of quality, primarily influencing sweetness and consumer acceptance of fruits (Selcuk & Erkan, 2015). The TSS of all groups shown in Table 4 decreased after storage. The fruit coated with SOLO exhibited a significantly higher TSS (7.65%) compared to the control (5.55%) and SOO-coated samples (6.17%) after the completion of one month of storage. The fresh fruit TSS was measured as 14.9% at the beginning. TSS loss was significant in all samples, but the SOLO coating was better overall. The coating may alter the gas composition inside the fruit (oxygen and carbon dioxide), thereby influencing respiratory metabolism, postponing the synthesis and breakdown of carbohydrates, and slowing the microbial activity to reduce sugar usage in the fruit (Gao et al., 2018). It can be inferred that the SOLO coating effectively sealed the pores/stomata and regulated the respiration process of fruits more efficiently than the SOO coating.

Table 4. Some physico-chemical measures of the fresh, and after 30 days of storage for the control and oleogel coated mandarines.

	Fresh Mandarin	After 30 Days of Storage		
		Control Mandarin	SOO Coated Mandarin	SOLO Coated Mandarin
Total Soluble Solids (%)	$14.9 \pm 0.01^{a*}$	5.55 ± 0.00^{d}	6.17 ± 0.01^{c}	7.65 ± 0.00^{b}
Titratable Acidity (% Citrate)	0.14 ± 0.01^{a}	0.12 ± 0.05^{b}	0.11 ± 0.04^{c}	0.11 ± 0.01^{c}
pH	4.33 ± 0.01^{a}	3.72 ± 0.01^{b}	3.64 ± 0.01^{c}	3.61 ± 0.01^{d}

*Small letters within each row indicate significant differences among the samples for the mean ± SD values by one-way analysis of variance and Tukey's test ($p \leq 0.05$).

The primary constituents of titratable acidity (TA) are organic acids, which often serve as substrates in the enzymatic activities of respiration. Consequently, TA content is considered a significant indication of the respiration rate of fruits (Xu et al., 2018). After 30 days, significant alterations in TA were seen in both control (0.12% citrate) and coated fruits (0.11% citrate for each) relative to their original levels (0.14% citrate) in fresh fruits (Table 4). All coating treatments and the control significantly reduced TA while prolonging the storage duration. The decrease in TA concentration while preserving citrus fruit may be attributed to using organic acids for energy generation. Acids may have been used for alcoholic fermentation in harvested citrus (Roongruangsri et al., 2013). Consequently, it may be inferred that coatings were ineffective in decelerating the degradation of organic acids during storage.

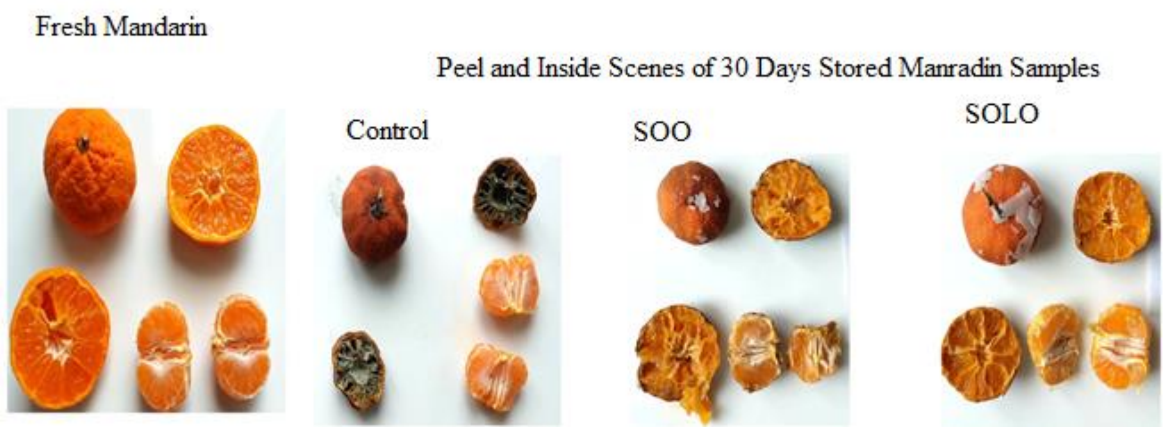


Fig. 6. The fruit peel and flesh scenes of the control and oleogels coated mandarin samples after 30 days of storage period (SOO: sunflower oil-sunflower wax oleogel, SOLO: sunflower oil-limonen-sunflower wax oleogel).

The impact of coating treatments on the pH of mandarin flesh during storage is also shown in Table 4. The initial pH and total acidity values were 4.33 and 0.14%, respectively. The findings of this investigation indicate that the coating treatments influenced the pH of the mandarin flesh in comparison to control samples. The pH values for coated samples ranged from 3.61 to 3.64 significantly lower than the fresh fruit and lower than the stored control sample ($p > 0.05$). Clearly, coatings have protected organic acids from degradation to provide a lower pH value. This situation might cause taste and aroma differences (Roongruangsri et al., 2013).

CONCLUSIONS

This study is a pioneer investigation of oleogel coatings on fresh mandarin fruits to prolong their shelf-life. Sunflower oil: sunflower wax (10 wt%) and sunflower oil: sunflower wax: limonene (10 wt% each) oleogels were used. For one month storage at room temperature, some indices were followed. Oleogel coatings preserved fruits from excessive moisture loss and colour changes. Oleogel coatings provided significant microbiological protection to mandarin fruits. After one-month, coated samples had lower total soluble solids and titratable acidity than fresh fruits, but the values were better than the control sample stored for one month. Overall, oleogel coatings for mandarin fruit prolonged the shelf-life to almost one month, which is considered an essential achievement. Further studies with different oleogel formulations and other oleogelator levels are suggested to improve the applicability of the technique.

CONFLICT OF INTEREST STATEMENT

No potential conflict of interest was reported by the author(s).

AUTHOR CONTRIBUTIONS

Emin Yılmaz: supervision, project administration, writing-review & editing. Merve Karataş: investigation, resources, formal analysis. Mehmet Seckin Aday: visualization, writing-original draft.

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