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Food-grade microemulsions as extractive and delivery systems for corn silks bioactives: phenolic recovery and oxidative stabilization of mayonnaise

Diego E. Carballo
Autonomous University of Chihuahua
Néstor Gutiérrez-Méndez
ngutierrez@uach.mx
Autonomous University of Chihuahua
Johan Mendoza
Autonomous University of Chihuahua
Diana L. Segovia-Guzmán
Autonomous University of Chihuahua
Martha Y. Leal-Ramos
Autonomous University of Chihuahua
Nubia Ivette Amaya-Olivas
Autonomous University of Chihuahua
Yamileth Cruz-Trillo
Autonomous University of Chihuahua

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Diego E. Carballo, Néstor Gutiérrez-Méndez*, Johan Mendoza, Diana L. Segovia-Guzmán, Martha Y. Leal-Ramos, Nubia Ivette Amaya-Olivas, and Yamileth Cruz-Trillo

Faculty of Chemical Sciences, Autonomous University of Chihuahua, Chihuahua 31000, Mexico.

*Corresponding author: Néstor Gutiérrez-Méndez

Chihuahua, México C.P. 31125, Phone: +52(614) 23-66-000 ext. 4245.

E-mail: ngutierrez@uach.mx

Abstract

Corn silk (CS), an underutilized maize by-product, contains bioactive compounds with potential as natural antioxidants. This study evaluated food-grade microemulsions (F-G μEs) as integrated extract-and-deliver systems to recover CS bioactives and transfer them directly into a food matrix. Two microemulsions containing D-limonene and Tween 80 were formulated, with or without acetic acid (AcLiTw and LiTw, respectively), and used to extract bioactive compounds from CS. An 80% ethanol extract was included as a conventional solvent control. Extracts were characterized for phenolics, protein, sugars, and antioxidant capacity using DPPH and ABTS assays. AcLiTw provided the highest total phenolic content (61.68 mg GAE/g DW) and the lowest DPPH IC_{50} (92.05 ppm), while LiTw showed the highest protein recovery (83.50 mg BSA/g DW). Notably, AcLiTw achieved higher total phenolic recovery after only 30 min of extraction, whereas conventional ethanolic extraction was conducted for 24 h. The AcLiTw extract was directly incorporated into mayonnaise at 4% and compared with control and 100 ppm BHA formulations during six months of refrigerated storage. Incorporating this extract modified the mayonnaise's pH, color, and rheological properties while maintaining pseudoplastic behavior and a predominantly elastic structure. At the end of storage, the AcLiTw-mayonnaise formulation showed the lowest TBARS values, indicating reduced secondary lipid oxidation compared with the control and a numerical advantage over the BHA-mayonnaise formulation. Overall, these findings demonstrate the potential of food-grade microemulsions as integrated extract-and-deliver platforms for rapid bioactive recovery and direct application in complex food emulsions.

Key words: corn silk; food-grade microemulsion; bioactive compounds; antioxidant activity; mayonnaise; lipid oxidation

Introduction

Mexico is one of the world's largest maize (*Zea mays*) producers, with annual production surpassing 27.5 million tons in 2023 (SIAP, 2024). This crop is deeply integrated into the country's agricultural sector and plays a vital role in both the national diet and the agricultural economy. Despite high production rates, significant amounts of corn by-products, such as corn silks (CS), are often discarded, presenting an opportunity for sustainable utilization (Maqsood et al., 2025; Zhang et al., 2023). Moreover, CS, the female inflorescence of maize, has been utilized globally as a natural alternative treatment for a variety of illnesses of the urinary tract, as well as conditions like fatigue, anxiety, obesity, diabetes, and cancer (Gulati et al., 2023; Jindal et al., 2022; Mendoza-López et al., 2018). The therapeutic benefits of this by-product have been associated with bioactive compounds like organic acids, saponins, polysaccharides, and phenolic compounds (Dong et al., 2023; Jindal et al., 2022; Lapčík et al., 2023; Mendoza-López et al., 2018; K. J. Wang & Zhao, 2019), which could be extracted and reused for various applications.

Using food by-products as a source of bioactive compounds is a practical strategy to reduce waste and generate value-added ingredients for the food, pharmaceutical, and cosmetic industries (Mia et al., 2025; Shawky et al., 2025; Soares Mateus et al., 2024). In this context, food-grade (F–G) microemulsions are gaining recognition as a safe, eco-friendly alternative to traditional extraction methods (e.g., organic and inorganic solvents, microwave-assisted extraction, ultrasound-assisted techniques, and pulsed electric field extraction), presenting an efficient approach to recovering bioactive compounds from by-products (Carballo et al., 2025). Microemulsions (μEs) can solubilize and facilitate the extraction of bioactive compounds because of their distinctive properties, including thermodynamic stability, optical transparency, low viscosity, and an amphipathic structure made up of water, oil, surfactants, and often co-surfactants. These properties create a flexible interfacial environment that can interact with compounds of different polarities, facilitating solubilization and recovery from plant matrices while remaining F-G and safe for direct

incorporation into food products (Abbasi & Scanlon, 2023; Chávez-Garay et al., 2025; Jalali-Jivan et al., 2020). From a food-processing perspective, their relevance may extend beyond solubilization, since a properly designed F–G μEs could simultaneously act as an extraction medium and as a carrier for recovered compounds.

Mayonnaise is a low-pH oil-in-water emulsion containing a high proportion of vegetable oil, which makes it susceptible to lipid oxidation during storage. Oxidative deterioration can lead to secondary oxidation products, such as aldehydes and ketones, which may negatively affect the product's sensory and nutritional attributes. Consequently, researchers have investigated incorporating antioxidant compounds into mayonnaise to improve oxidative stability and improve shelf life. In particular, plant-derived extracts rich in phenolic compounds have shown potential to inhibit lipid oxidation in mayonnaise, supporting their use as natural alternatives to synthetic antioxidants (Alizadeh et al., 2019; Ghorbani Gorji et al., 2016). However, the use of F–G μEs as integrated extraction and delivery systems to directly transfer plant-derived bioactives into complex food matrices remains underexplored. This approach could intensify processing by combining bioactive recovery and delivery while avoiding intermediate isolation, solvent removal, and redispersal steps.

In this context, this study aimed to evaluate food-grade microemulsions based on D-limonene, Tween-80, and acetic acid as integrated extract-and-deliver systems for the recovery of bioactive compounds from corn silk and their direct incorporation into mayonnaise, with particular emphasis on extraction performance, food-matrix compatibility, rheological behavior, and oxidative stability of mayonnaise during storage.

Materials and methods

Materials

Chemicals, additives and ingredients

The components of the food-grade micro-emulsions and mayonnaise formulations, including food-grade D-limonene (95%; ROAR Chemicals, Chihuahua, Mexico), polyoxyethylene (20) sorbitan monooleate (Tween-80, 99%; AZUMEX, Puebla, Mexico), vegetable palm oil, vinegar, and dehydrated egg, were obtained from a local market (Chihuahua, Mexico). Acetic acid, 1,1-diphenyl-2-picrylhydrazyl (DPPH), 2,2′-Azino-bis(3-ethylbenzothiazoline-6-sulfonic acid) (ABTS), *Folin-Ciocalteu* phenol reagent, sulphuric acid, phenol, sodium carbonate, glucose, bovine serum albumin, p-coumaric; gallic, chlorogenic, caffeic, vanillic, and trans-ferulic acids, epicatechin, catechin, rutin, and phloridzin were purchased from Sigma-Aldrich (St. Louis, MO, USA).

Raw material

Corn silks (CS) from *Zea mays* were obtained from a local supply center in Chihuahua, Mexico. The collected CS was manually cleaned to remove foreign materials and then dried at room temperature (25 °C) and a relative humidity of 35% for one week in a well-ventilated area, avoiding direct sunlight to prevent degradation of bioactive compounds. After drying, the CS were milled and sieved to obtain a uniform particle size of 420 µm. The powdered CS was then packed in airtight, moisture-resistant containers and stored at −25 °C until further analysis. A proximate composition analysis of the CS powder, including moisture, ash, protein, lipid, and fiber content, was performed following the standard procedures outlined by AOAC (2003).

Food-grade microemulsions (F–G µEs) formulation and characterization

This work used two food-grade microemulsions formulated and characterized in a previous study (Carballo et al., 2025). The first F–G µE was a non-acidic oil-in-water (O/W) system (LiTw), composed of 2% (v/v) D-limonene (Li), 1% (v/v) Tween-80 (Tw), and 97%

deionized water. The second F–G μE was an acidic O/W system (AcLiTw) containing the same ingredients as the non-acidic formulation, but with 40% (v/v) acetic acid (Ac). Both formulations were homogenized for 2 min at 15500 rpm with a high-speed disperser (Ultra-Turrax® T18 IKA Works Inc., Wilmington, NC, USA) and then subjected to 10 min of ultrasonic (70 W, 40 kHz, Branson Ultrasonics Corporation, Danbury, CT, USA) treatment (Piñon-Gómez et al., 2025). After preparation, the systems were incubated at 35 °C to allow equilibration and complete microemulsion formation. During this stage, samples were kept under static conditions until they became transparent and homogeneous, indicating system stabilization. The time required to reach equilibrium was monitored through visual and microscopic observations (B-383PHi trinocular microscope, equipped with a C-P6 digital camera, Optika, Italy). Turbidity during incubation was also monitored with a microplate reader at 595 nm, considered fully transparent when the turbidity values were lower than 5% (Elx808, Biotek, USA). Before their use as extraction media, stabilized microemulsions were characterized in terms of pH (Conductronic, mod pH120, Puebla, Mexico), conductivity (probe number 476501, Corning 441, Corning, NY), density (5 mL pycnometer), and rheological properties (AR-2000ex rotational rheometer; a flat [0◦angle] aluminum plate of 40 mm, and a water trap; TA Instruments, New Castle, DE, USA) to ensure batch-to-batch reproducibility.

Extraction of bioactive compounds from corn silks

The extraction of bioactive compounds from dried corn silks (CS) was performed using LiTw and AcLiTw microemulsions (μEs). Additionally, ethanolic extractions were carried out as a control comparison (Carballo et al. 2025). Briefly, for μEs-based extraction, 1 g of CS was mixed with 10 mL of LiTw or AcLiTw and stirred at 750 rpm and 25 °C for 30 min. For the ethanolic extraction, 1 g of CS was stirred under the same conditions with 10 mL of 80% (v/v) ethanol for 24 h. The ethanolic procedure served as the conventional reference extraction, and each extraction system was evaluated under its previously established operating conditions rather than identical extraction times. After agitation, the

extraction process involved two centrifugation steps (2000 rpm, 15 min), with an additional 5 mL of the corresponding solvent (μEs or 80% ethanol) added before the second centrifugation. The supernatants from each extraction method (μEs or ethanol) were then combined and adjusted to a final volume of 10 mL with the corresponding solvent. The CS extracts from both methods were stored at -20 °C until further analysis.

Corn silk extracts characterization

Physicochemical characterization of corn silk extracts

The *pH* of the extracts was measured in triplicate using a digital pH meter (Conductronic, model pH120, Puebla, Mexico). Total solids extracted were determined gravimetrically by measuring water content in the extracts, following the standard AOAC (2003) procedure. The *total phenolic content* (TPC) in the extracts was analyzed using the Folin-Ciocalteu method (Folin & Ciocalteu, 1927), with gallic acid as the standard (GA; 0–300 ppm), measuring absorbance at 760 nm. *Total sugars* (TS) were analyzed using the phenol-sulfuric acid method (Dubois et al., 1956), with glucose as the standard (G; 0–6 mM), measuring absorbance at 490 nm. *Protein content* (PC) was analyzed using the Bradford assay (Bradford, 1976) with bovine serum albumin as the standard (BSA; 0–1 mg/mL), measuring absorbance at 595 nm. The spectrophotometric readings were carried out using a microplate reader (BioTek Instruments ELx808, Vermont, USA). Results from these analyses were reported as milligrams of equivalents per gram of dry weight: mg GA eq/g DW for phenolics, mg G eq/g DW for sugars, and mg BSA eq/g DW for proteins.

Chromatographic analysis of corn silk extracts

The chromatographic profiles of the different corn silk extracts were evaluated using a UHPLC system (Dionex Ultimate 3000, Thermo Scientific, MA, USA) equipped with a binary pump, an autosampler, a temperature-controlled column compartment, and a diode-array detector (DAD). Chromatographic separation was performed on a Zorbax SB-C18 reverse-phase C18 column (250 × 4.6 mm, 5 μm, Agilent, Santa Clara, CA, USA)

maintained at 35°C. The mobile phase consisted of formic acid 1% in water (solvent A) and acetonitrile (solvent B), delivered at a flow rate of 1.0 mL/min. The injection volume was 10 µL. Chromatographic separation was performed using a gradient elution program starting at 0% solvent B and increasing linearly to 40% over 40 min, and subsequently to 100% over the following 20 min. The column was then maintained at 100% solvent B for 5 min for washing, followed by re-equilibration at 100% solvent A for 10 min before the next injection. Chromatograms were recorded at 280 and 320 nm. Retention times and UV–Vis absorption spectra were compared with those of the phenolic reference standards.

Antioxidant capacity of corn silk extracts

The antioxidant activity of CS extracts, including ethanolic and µEs-based (LiTw and AcLiTw), was assessed using the DPPH (1,1-diphenyl-2-picrylhydrazyl) and ABTS* (2,2′-Azino-bis(3-ethylbenzothiazoline-6-sulfonic acid)) free radical scavenging activity (RSA) assays. For *DPPH-RSA*, extract solutions (0–500 ppm) were mixed with an equal volume of 0.2 mM DPPH in ethanol and incubated in the dark at room temperature for 60 min. Following incubation, a 250 µL aliquot was transferred to a microplate, and absorbance was recorded at 517 nm (Carballo et al., 2025). The *ABTS-RSA* assay was performed following the procedure described by Re et al. (1999), with slight modifications. Briefly, the ABTS radical solution was prepared, and its absorbance was adjusted to 0.700 at 734 nm. Next, 10 µL of each extract solution (10–100 ppm) was added to 190 µL of the ABTS solution in a microplate. The mixtures were kept in the dark for 5 minutes before measuring absorbance at 734 nm. Both assays were performed using a microplate reader (BioTek Instruments ELx808, Vermont, USA).

As blanks, either 1 mL of ethanol (80%) or both plain microemulsions (LiTw and AcLiTw) were used. Finally, the results were expressed as % DPPH/ABTS* Radical Scavenging Activity (RSA) using the following equation 1:

$$\%\, RSA = \left((Abs^{blank} - Abs^{sample}) / \, Abs^{blank} \right) \times 100 \qquad \text{Eq.1}$$

For comparative purposes, the RSA of butylated hidroxinaisole (BHA; 1-10 ppm) was analyzed as formerly described. The half-maximal inhibitory concentration (IC_{50}), defined as the concentration required to reduce 50% of 0.2 mM DPPH or ABTS* radicals, was determined for each extract. IC_{50} values were estimated by fitting the % RSA to a two-parameter rectangular hyperbolic model using SigmaPlot software (version 14.5; Systat Software, Inc.).

Incorporation of corn silk extract into mayonnaise

Based on the antioxidant capacity of the μEs and the pH threshold of 4.4 for controlling pathogenic microorganisms (Chandrapala et al., 2025), the effectiveness of only acidic microemulsions CS extract was assessed in a model food system by evaluating oxidative stability in mayonnaise formulations. The formulation containing acidic μEs-CS extract was compared with two control systems: mayonnaise containing vinegar and mayonnaise formulated with vinegar and the synthetic antioxidant, butylated hydroxyanisole (BHA). All formulations were prepared in triplicate using 7 % (w/w) dehydrated egg as the emulsifying agent, 70 % (w/w) palm vegetable oil as the lipid phase, and 2 % (w/w) salt, following the mayonnaise dehydrated egg manufacturer's instructions. According to the treatment, 4 % (w/w) vinegar (MV), 4 % (w/w) vinegar plus 100 ppm BHA (MVBHA), or 4 % (w/w) acidic microemulsion-CS extract (MAcCS) was incorporated. The remaining proportion was adjusted with distilled water. Mayonnaise was prepared under controlled conditions using a handheld stick mixer (Oster, model FPSTHB2610R, USA) operated at low speed for 1 min, until a stable emulsion was formed. The resulting emulsions were stored in sealed glass containers under refrigeration (4 °C) for up to 6 months.

Physicochemical characterization of mayonnaise

pH, *color*, and *rheological behavior* of the mayonnaise were analyzed in triplicate in freshly prepared samples and after 6 months of refrigerated storage (4 °C). pH was determined

using a digital pH meter (Conductronic, model pH120, Puebla, Mexico). The mayonnaise color (L^*, a^*, and b^*) were measured with a calibrated colorimeter (CR-400/410 Konica-Minolta, Osaka, Japan). Before the analysis, the samples were transferred into transparent plastic containers and placed on a white surface. The apparent viscosity and viscoelastic behavior of the mayonnaise samples were evaluated using an AR-2000ex rotational rheometer (TA Instruments, New Castle, DE, USA) fitted with a 40 mm flat aluminum plate. About 1 mL of each sample was carefully placed on the plate and allowed to stabilize for 2 min at 10 °C prior to testing. The distance between plates was maintained at 1000 μm. Apparent viscosity (η_{ap}; Pa*s) measurements were carried out at share rates between 1 and 200 s^{-1} (2 min; 10 °C). Viscoelastic properties were assessed through an oscillatory strain sweep test (5-25 %; 10 °C; 7 Hz) to identify the linear viscoelastic region (LVR).

Lipid oxidation of mayonnaise during storage

The lipid stability of mayonnaise was evaluated at 2, 4, and 6 months after preparation using the thiobarbituric acid-reactive substances (TBARS) assay following the procedures described by Nam & Ahn (2003). Results were expressed as mg of malondialdehyde (MDA) per kilogram of mayonnaise.

Statistical analysis

Data from micro-emulsion systems (LiTw and AcLiTw), ethanolic corn silk extracts, mayonnaise characterization, and oxidative stability assays were analyzed using a general linear model (one-way ANOVA), with extraction method, mayonnaise formulation type or storage time as fixed factors. When significant effects were observed, mean comparisons were performed using the Tukey-Kramer *post-hoc* test at $P < 0.05$. All experiments were conducted at least in triplicate, and results are expressed as mean values. Statistical analyses were performed using SPSS Statistics software (version 26; IBM, Somers, NY, USA).

Results and discussion

Food-grade microemulsions (F–G μEs)

Both LiTw and AcLiTw systems formed clear, homogeneous dispersions after the stabilization period at 35 °C, confirming successful microemulsion formation. Consistent with the characteristic properties of microemulsions — namely, their isotropic and transparent nature, with turbidity values typically below 0.5–1 % (Cho et al., 2008) — both systems showed low turbidity at equilibrium (≈ 0.20 for LiTw and ≈ 0.14 for AcLiTw). Turbidity changed over time, indicating differences ($P < 0.05$) in self-assembly kinetics between formulations (Fig. 1). The acidic system (AcLiTw) reached a stable state more rapidly (≈ 11 days) than the non-acidic (LiTw), which requires nearly 14 days to attain comparable stability (Fig. 1). This behavior suggests that acetic acid promotes the organization of interfacial structures during microemulsion formation. The faster stabilization observed in the AcLiTw agrees with previous reports indicating that acidic conditions can enhance microemulsion stability. Aum et al. (2021) demonstrated that acidic environments improve the stability of oil-in-water microemulsions formulated with nonionic surfactants. This effect may relate to changes in interfacial interactions and surfactant packing, which promote the formation of more stable, rapidly organized structures.

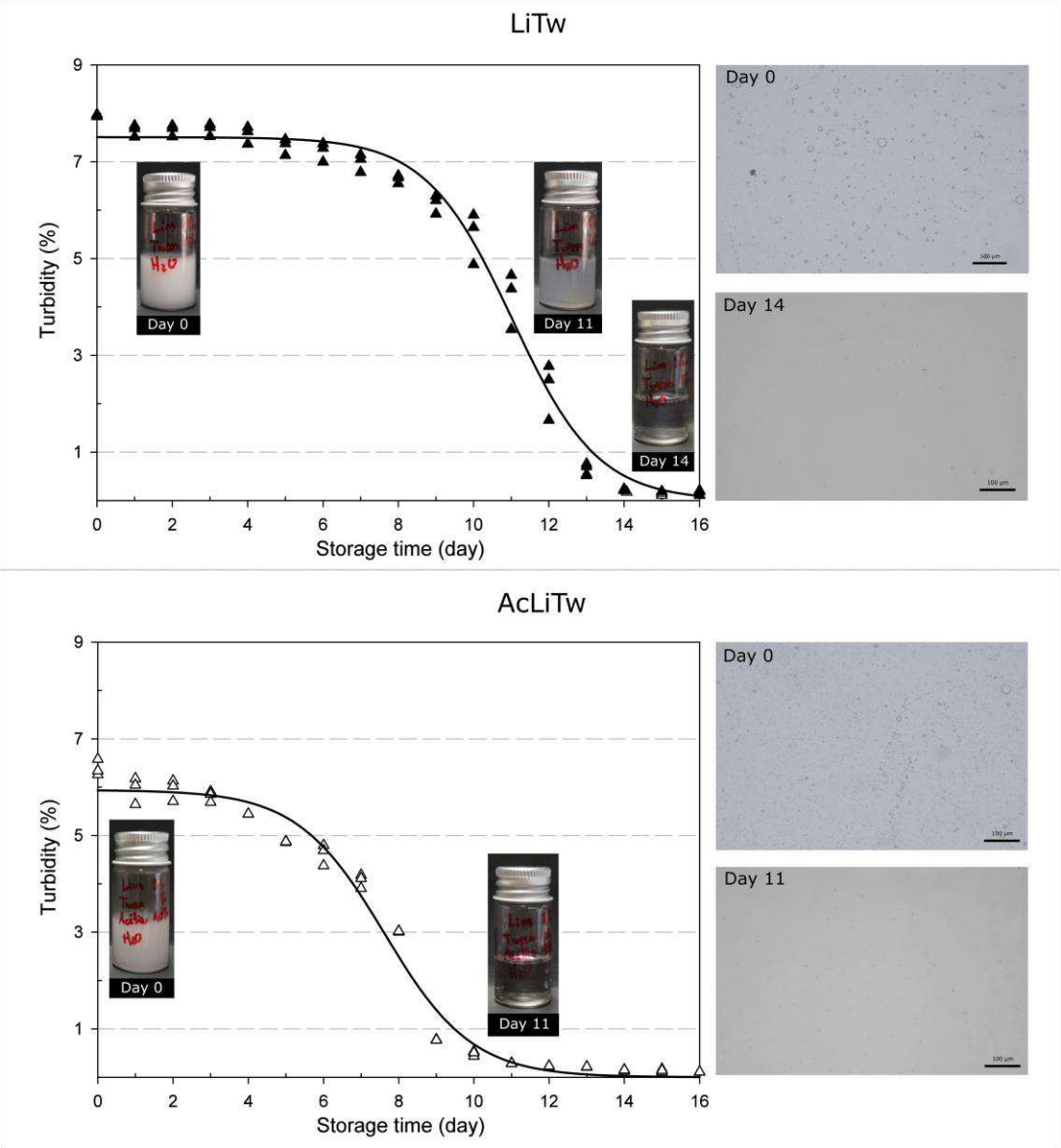


Fig. 1 Turbidity kinetics and micrographs showing the formation of food–grade microemulsions over time, at 35 ºC. The LiTw formulation included 2% D-limonene, 1% Tween-80, and 97% water. The formulation AcLiTw included 2% D-limonene, 1% Tween-80, 40% acetic acid, and 57% water.

Physicochemical characterization of F–G μEs revealed clear differences between formulations. LiTw exhibited a near-neutral pH (5.22 ± 0.01), a conductivity of 1.14 ± 0.02 mS/cm, and a density of 1.01 ± 0.01 g/cm³, whereas AcLiTw presented a highly acidic environment (pH 0.71 ± 0.02), with higher conductivity (1.24 ± 0.04 mS/cm) but a similar density (1.01 ± 0.01 g/cm³) to the LiTw formulation. These differences markedly

influenced the rheological behavior of the F–G μEs systems. Both microemulsion formulations exhibited typical non-Newtonian pseudoplastic behavior, with viscosity decreasing as shear rate increased. However, the incorporation of acetic acid led to a slight increase ($P < 0.1$) in apparent viscosity (3.43 ± 0.42 mPa·s for AcLiTw vs. 2.47 ± 0.07 mPa·s for LiTw).

Overall, these findings indicate that acetic acid not only accelerates microemulsion formation but also modifies the interfacial and structural properties of the system, potentially enhancing its performance as an extraction medium for bioactive compounds. These characteristics are technologically relevant because the physicochemical environment of the extraction phase may influence not only compound recovery but also its subsequent compatibility with the food matrix.

Recovery of bioactive compounds from corn silks using microemulsions

The ability of food–grade acidic and non–acidic microemulsions to recover bioactive compounds was evaluated using corn silks (CS), a locally sourced agro-industrial by-product, as the extraction matrix. Prior to extraction, the raw material was characterized by proximate composition analysis. CS was composed (% dry basis) as follows: crude protein 16.04 ± 0.21, ash 7.09 ± 0.005, fat 1.94 ± 0.006, total carbohydrates 75.71 ± 0.13, and crude fiber 55.6 ± 0.32. The results highlighted the significant nutritional potential of corn silk, as evidenced by its high content of bioactive and nutritive compounds (Gulati et al., 2023; Jindal et al., 2022; Mendoza-López et al., 2018). Accordingly, their valorization represents a promising approach for their effective use.

The extraction yields of bioactive compounds from corn silks (CS) using F–G μEs and 80 % (v/v) ethanol are presented in Table 1. Extracts obtained with F–G μEs showed significantly higher ($P < 0.05$) recoveries of both total protein and total polyphenols than those obtained by conventional ethanolic extraction. Moreover, the extraction of bioactive

compounds from CS varied significantly depending on the extraction system used. F–G μEs markedly enhanced the recovery of specific biomolecules compared with conventional ethanolic extraction. In particular, the non-acidic microemulsion (LiTw) recovered significantly more proteins from the CS than the acidic microemulsion (AcLiTw) or the ethanol-water system (Table 1). Regarding the extraction of phenolic compounds, the acidic μE surpassed the non-acidic μE and the ethanol-water system in the recovery of polyphenols (Table 1). These results indicate that the presence of acetic acid enhanced the recovery of phenolic compounds, likely by increasing μEs penetration into the vegetal matrix or by improving the solubilization of phenolic compounds (Naczk & Shahidi, 2004; Zhu et al., 1997). Importantly, the enhanced recovery with F–G μEs was achieved in a substantially shorter extraction period than the conventional ethanol-water system, highlighting the potential of the microemulsion to intensify the recovery step. This behavior is consistent with the well-established understanding that extraction yield depends on several factors, including solvent type, polarity, and interactions with the sample matrix (Dai & Mumper, 2010). This behavior suggests that the microemulsion's interfacial environment can modify compound accessibility and partitioning from the plant matrix. In contrast, total sugar content did not differ significantly among extraction methods ($P > 0.05$) (Table 1).

Table 1 Characterization of ethanolic and non–acidic (LiTw) food–grade micro emulsions (F–G μEs) and acidic F–G μEs (AcLiTw) corn silk extracts

	Extracts			RSME	Significance
	Ethanolic (80 % v/v)	F–G μEs[#]			
		LiTw	AcLiTw		
Total protein[1]	15.83[c]	83.50[a]	48.65[b]	6.11	***
Total sugars[2]	804.30	798.10	757.40	100.41	NS
Total polyphenols[3]	33.13[c]	47.83[b]	61.68[a]	3.39	***
Antioxidant activity IC_{50}					
DPPH	126.66[b]	324.9[a]	92.05[b]	19.06	***
ABTS	1911.88[c]	3089.42[a]	4291.78[b]	175.01	***

Mean (n=3); RMSE = root mean square error
F–G μEs = food-grade microemulsions
[1] = mg eq bovine serum albumin/g dry weight, [2] = mg eq glucose/g dry weight, [3] = mg eq gallic acid/g dry weight,
[#] LiTw = 2 % D-limonene, 1 % Tween 80 (v/v); AcLiTw = 2 % D-limonene, 1 % Tween 80, 40 % acetic acid (v/v).
Significance: NS = not significant, + = P <0.1, * = P <0.05, ** = P <0.01, *** = P <0.001
[abc] = different superscripts in the same row indicate significant differences by the Tukey-Kramer test (P < 0.05)

To complement the spectrophotometric determination of total polyphenols, the chromatographic profiles of corn silk extracts were evaluated by HPLC at 280nm (Fig. 2). Although none of the major chromatographic signals could be unequivocally assigned to the phenolic reference standards available (gallic, p-coumaric, chlorogenic, caffeic, vanillic, and trans-ferulic acids, epicatechin, catechin, rutin, and phloridzin), clear differences in the intensity and distribution of UV-absorbing compounds were observed among the extraction systems tested. Several of these reference compounds have previously been identified in corn silk extracts, although their occurrence and relative abundance are strongly influenced by corn variety, silk maturity, and extraction conditions (Sarepoua et al., 2015).

The chromatographic profiles revealed marked differences in the distribution of UV-absorbing compounds according to the extraction conditions. LiTw was characterized predominantly by intense early-eluting signals, particularly within the first 10 min (red square in Fig. 2b), whereas relatively few signals were observed at longer retention times. In contrast, AcLiTw exhibited a broader and more complex chromatographic profile, with numerous signals distributed throughout the intermediate and late retention-time regions (green square in Fig. 2c). The ethanolic extract also displayed a broad distribution of signals, with a profile similar to that observed for AcLiTw, particularly in the intermediate-to-late portion of the chromatogram. These results indicate that the composition of the extraction medium affected not only the overall amount of extracted compounds but also the chromatographic distribution of the recovered constituents.

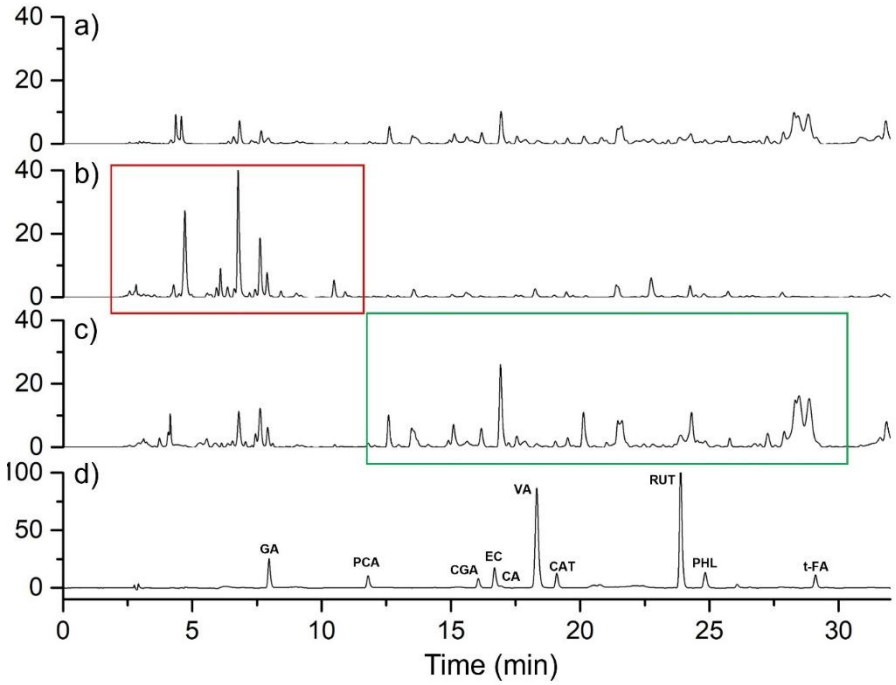


Fig. 2 HPLC chromatographic fingerprints at 280 nm of corn silk extracts obtained with (a) 80% ethanol, (b) LiTw, and (c) AcLiTw, and (d) phenolic reference standards: GA = gallic; PCA = p-coumaric; CGA = chlorogenic acid; CA = caffeic; VA = vanillic; t-FA = trans-ferulic acids; EC = epicatechin; CAT = catechin; RUT = rutin; PHL = phloridzin.

Interestingly, AcLiTw, which showed the highest total polyphenol content, also exhibited a more pronounced and broadly distributed chromatographic response at 280 nm than LiTw. The increased Folin–Ciocalteu response of AcLiTw was therefore accompanied by changes in the chromatographic fingerprint, suggesting that acidification of the microemulsion altered the extraction selectivity rather than simply increasing the recovery of the same constituents extracted by LiTw. Solvent polarity, pH, the chemical characteristics of the target compounds, and their interactions with the plant matrix strongly influence the efficiency and selectivity of phenolic extraction (Dai & Mumper, 2010; Naczk & Shahidi, 2004). In addition, F-G μEs provide heterogeneous microenvironments that may facilitate the solubilization of compounds with different polarities (Carballo et al., 2025). In this context, combining an acidic aqueous phase with D-limonene and Tween-80 may have contributed to the broader chromatographic distribution observed for AcLiTw.

Despite the use of several phenolic standards previously reported in corn silk, the major chromatographic signals observed in the extracts could not be unequivocally identified under the conditions used. This finding is not unexpected, considering the phytochemical complexity reported for corn silk. Advanced LC–MS analyses have revealed numerous phenolic constituents, particularly C- and O-glycosylated flavonoids and structurally related derivatives, including maysin, apimaysin, schaftoside, and other luteolin- and apigenin-derived glycosides (Fougère et al., 2020, 2023; Lapčík et al., 2023). Consequently, the absence of retention-time matches with the available standards does not necessarily indicate the absence of phenolic compounds but rather suggests that the predominant constituents may not be adequately represented by the reference set used in this study. Therefore, the HPLC results were interpreted as comparative chromatographic fingerprints of UV-absorbing constituents rather than definitive identification or quantification of individual phenolic compounds.

From a practical perspective, the markedly shorter extraction time required by AcLiTw represents an additional advantage of this system. While conventional extraction with 80%

ethanol required 24 h, AcLiTw achieved a comparable chromatographic fingerprint and higher total phenolic recovery within only 30 min. Thus, the extraction time was reduced by approximately 98%, suggesting that the acidic microemulsion promoted substantially faster recovery of UV-absorbing constituents from the corn silk matrix. This improved extraction efficiency, along with the use of food-grade components, highlights the potential of AcLiTw as a rapid alternative to conventional ethanolic extraction for recovering bioactive compounds from corn silks.

Antioxidant capacity of corn silk extracts

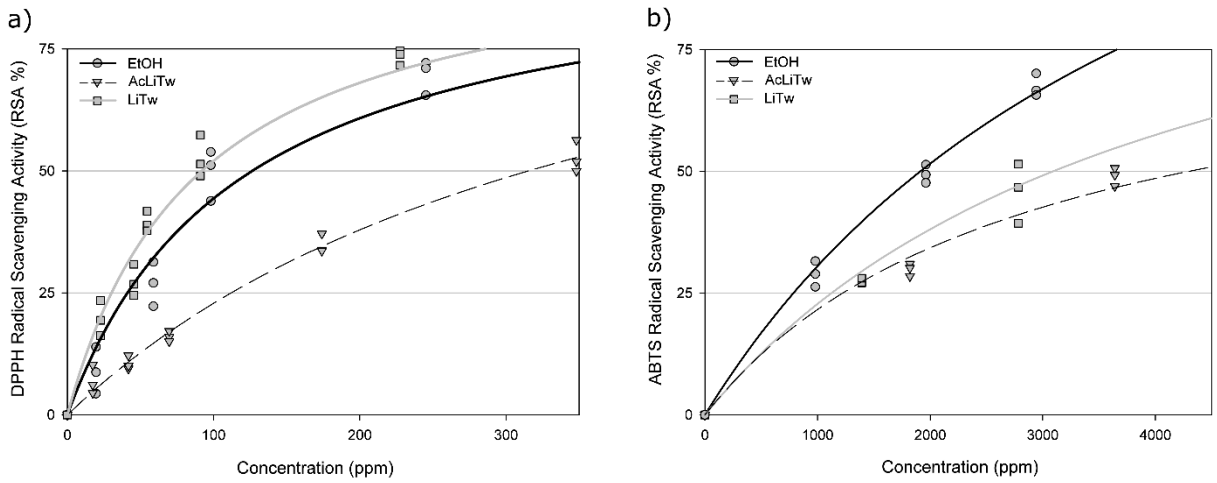
The antioxidant capacity of the extracts was evaluated through radical scavenging activity profiles and corresponding IC_{50} values for both DPPH and ABTS* assays, as shown in Fig. 2 and Table 1. The extraction solvent type significantly affected the antioxidant activity of corn silk (CS) extracts ($P < 0.05$). In the DPPH assay, AcLiTw and ethanolic CS extracts showed the lowest IC_{50}, while the LiTw extract showed the highest IC_{50} value (Table 1). This behavior was consistent with the polyphenol content of each extract, suggesting that phenolic compounds contribute the most to DPPH-scavenging activity. Corn silks contain phenolic compounds and flavonoids with antioxidant properties, and previous studies have shown that extraction conditions can markedly influence both the recovery of these compounds and the resulting antioxidant activity (Liu et al., 2011).

In contrast, the ABTS antioxidant assay showed a different trend. The ethanolic extract had the lowest IC_{50}, followed by LiTw, whereas AcLiTw had the highest IC_{50} value (Table 1). Thus, the extract with the highest polyphenol content did not show the highest ABTS* radical scavenging activity. This difference may relate to extract composition and the different reaction mechanisms of the DPPH and ABTS* assays. These methods can respond differently depending on the polarity and chemical structure of the antioxidant compounds present in the extracts. DPPH is more sensitive to the presence and reactivity of compounds in organic media, whereas ABTS* can react with both hydrophilic and lipophilic

antioxidants. Therefore, compounds with different structural characteristics may respond differently in these assays (Platzer et al., 2021).

Overall, AcLiTw extracts showed the highest DPPH activity, while the ethanolic extract showed the highest ABTS* activity. This difference indicates that the extracts' antioxidant capacity was not determined solely by their total polyphenol content (Platzer et al., 2021). The extraction procedure can modify the profile of compounds recovered from corn silks and, consequently, their response in different antioxidant assays. This has also been reported in corn silk extracts, where the extraction procedure significantly affected antioxidant activity measured by DPPH and ABTS* (Lapčík et al., 2023).

Compared with BHA, used as a synthetic antioxidant reference, all CS extracts showed higher IC_{50} values in both assays, indicating lower radical-scavenging capacity under the conditions evaluated. BHA showed IC_{50} values of 17.19 ± 1.02 ppm for DPPH and 3.95 ± 0.42 ppm for ABTS, whereas the CS extracts required higher concentrations to achieve 50 % radical inhibition. The antioxidant activity observed for BHA in both assays is consistent with previous reports (Huyut et al., 2017), supporting its use as a reference antioxidant in the present study. BHA's higher activity is consistent with previous reports describing its strong radical-scavenging capacity in both DPPH and ABTS assays (Dawidowicz et al., 2015). Nevertheless, the antioxidant activity observed for the CS extracts, particularly AcLiTw in the DPPH assay, is relevant because it was obtained from a natural waste source using a food-grade extraction system. Therefore, although the extracts did not reach the antioxidant power of BHA, their activity supports their potential as natural sources of antioxidant compounds for food applications.



485

486 **Fig. 3** Percentage of a) DPPH and b) ABTS radical-scavenging activity (% RSA) of
487 ethanolic and food-grade microemulsions (F–G μEs) corn silk extracts

488

489 **Application of corn silk extract in mayonnaise**

490

491 To evaluate the effect of incorporating microemulsion extracts into a food matrix,
492 mayonnaise formulations containing different additives were prepared and characterized.
493 The formulations included a regular mayonnaise, consisting of 4% vinegar (MV); a regular
494 mayonnaise with vinegar (4%) and BHA (100 ppm) as antioxidant (MVBHA), and a
495 mayonnaise added with 4% of acidic μE corn silk extract (MAcCS). Notably, direct
496 incorporation of the AcLiTw extract made the extraction medium part of the food
497 formulation, eliminating the need to isolate and then redisperse the recovered bioactives.

498

499 *Physicochemical properties of mayonnaise*

500 As shown in **Table 2**, incorporating AcCS into the mayonnaise formulation significantly
501 affected the *physicochemical*, *color*, and *rheological properties* compared with the control
502 mayonnaise (MV) and the formulation containing BHA (MVBHA). Total polyphenols
503 were detected only in MAcCS (52.70 ±1.5 mg Eq GA/kg), confirming the incorporation of
504 corn silk phenolic compounds into the mayonnaise matrix. Similar studies show that

incorporating plant extracts can increase the phenolic content of mayonnaise while modifying some of its physicochemical and quality properties (Turker et al., 2024).

Table 2 Physicochemical characterization of mayonnaise formulations during six months of storage time

	Mayonnaise formulation (treat)[#]			Storage time (time)		RSME	Significance		
	MV	MVBHA	MAcCS	0 (Freshly prepared)	Six months		Treat	time	Treat*time
Total polyphenols[1]	0	0	52.7	nd	nd	-	-	-	-
pH	4.78[a]	4.71[b]	3.96[c]	4.53	4.43	0.01	***	***	***
Color									
*L**	91.09[a]	91.29[a]	89.9[b]	90.04	91.48	0.22	***	***	***
*a**	0.13[b]	0.14[b]	0.56[a]	0.43	0.122	0.06	***	***	***
*b**	15.72[c]	16.28[b]	18.13[a]	16.53	16.90	0.33	***	**	NS
Hue angle	88.18[b]	89.54[b]	88.18[a]	88.52	89.37	0.37	***	***	***
Rheological parameters									
Apparent viscosity	3.21[a]	3.20[a]	2.36[b]	2.89	2.95	0.11	***	NS	**
G´	303.33[a]	290.67[a]	177.02[b]	193.22	320.78	28.59	***	***	**
G´´	76.35[a]	83.03[a]	41.88[b]	53.70	80.47	6.96	***	***	*
Tan δ	0.27[b]	0.29[a]	0.24[c]	0.28	0.26	0.03	***	**	*

Mean (n=3); RMSE = root mean square error
[1] = mg eq gallic acid/kg of mayonnaise, nd = non detected.
[#] MV = mayonnaise base with vinegar (4%); MVBHA = mayonnaise base with vinegar (4%) and BHA (100 ppm); MAcCS = mayonnaise base with acidic microemulsion corn silk extract (4%).
Significance: NS = not significant, + = P <0.1, * = P <0.05, ** = P <0.01, *** = P <0.001
[abc] = different superscripts in the same row indicate significant differences by the Tukey-Kramer test (P <0.05)

Formulation and storage time significantly affected mayonnaise *pH* and *color* ($P < 0.001$). MAcCS showed the lowest pH (3.96), compared with MV (4.78) and MVBHA (4.71), which can be attributed to the incorporation of 4% acidic microemulsion (AcLiTw = pH 0.71). Nevertheless, the final pH of the MAcCS remained within the range commonly

reported for mayonnaise, approximately 3.3–4.2 (Chandrapala et al., 2025). Storage caused a small decrease in pH with a significant treatment x time interaction ($P < 0.001$). A similar decrease has been reported in mayonnaise during storage and has been associated with the formation of free fatty acids from lipid hydrolysis and oxidation, which increase acidity (Wang et al., 2022). The interaction between treatment and time ($P < 0.05$) can be explained by differences in formulation behavior during storage. Although pH decreased slightly over time, the MAcCS formulation showed a smaller decrease compared with MV and MVBHA (Data not shown in the table for brevity). This suggests that incorporating the acidic corn silk microemulsion extract resulted in more stable pH during storage. Regarding *color*, MAcCS exhibited a 1.01-fold reduction ($P < 0.001$) in L^* and approximately 4.3-fold and 1.15-fold increases ($P < 0.001$) in a^* and b^*, respectively, compared with MV (Table 2), indicating a slightly darker and more reddish-yellow appearance (Fig. 4). These differences likely reflect the pigmented phenolic compounds introduced by the corn silk extract, since corn silks contain flavonoids, anthocyanins, and chlorophylls whose proportions vary with silk maturity and extraction method (Khushe et al., 2024). These changes are consistent with previous reports on mayonnaise enriched with plant-derived antioxidant extracts, where pigment- or phenolic-rich additions altered CIELab values (De Bruno et al., 2021).

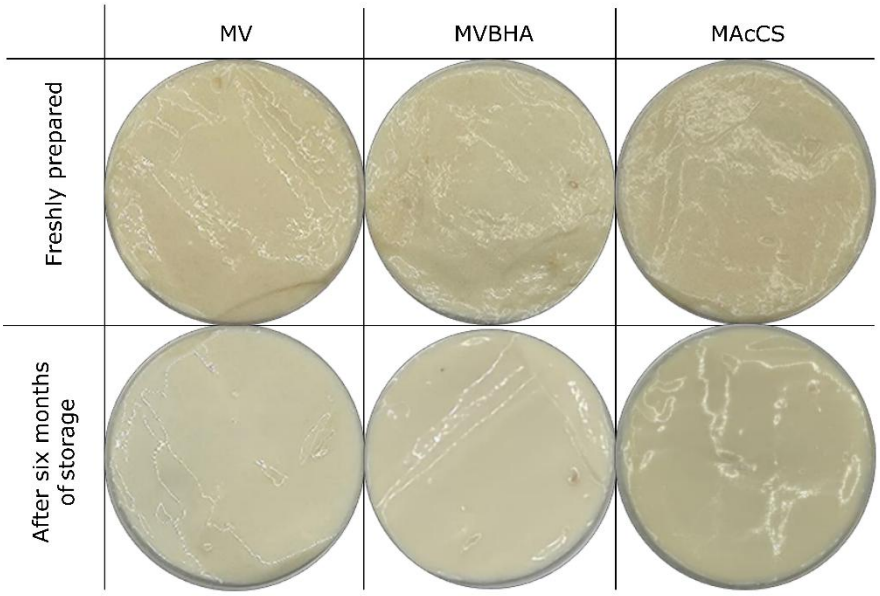


Fig. 4 Visual appearance of mayonnaise formulations during six months of storage (4 C). MV = mayonnaise base with vinegar (4%); MVBHA = mayonnaise base with vinegar (4%)

and BHA (100 ppm); MAcCS = mayonnaise base with acidic microemulsion corn silk extract (4%).

MAcCS markedly affected rheological properties. The apparent viscosity of MAcCS was approximately 26% lower than that of MV and MVBHA, while G′ and G″ were reduced by approximately 42% and 45%, respectively, compared with MV. These results indicate a reduction in the magnitude of the viscoelastic structure after incorporating the acidic microemulsion. Nevertheless, all formulations exhibited a pseudoplastic (shear-thinning) behavior, which is characteristic of mayonnaise and other concentrated oil-in-water emulsions. Nevertheless, all formulations exhibited pseudoplastic (shear-thinning) behavior, characteristic of mayonnaise and other concentrated oil-in-water emulsions (Katsaros et al., 2020). The lower viscosity and viscoelastic moduli in MAcCS may be related to the combined effect of the microemulsion, its acidic conditions, and the compounds extracted from corn silks; therefore, they cannot be attributed exclusively to the polyphenols. Moreover, Nessabian et al. (2024) reported comparable changes in rheological properties after incorporating phenolic extracts into mayonnaise. During storage, apparent viscosity was not significantly affected ($P = 0.267$), whereas G′, G′′, and tan δ showed significant changes. After six months, storage and loss moduli increased by approximately 66% and 50%, respectively, indicating a more pronounced viscoelastic structure during storage. Additionally, the significant ($P < 0.001$) treatment × time interaction suggests that these changes depended on the formulation. Interestingly, MAcCS showed the lowest tan δ, approximately 11% and 17% lower than MV and MVBHA, respectively, indicating the greater relative contribution of the elastic component despite its lower absolute G′ and G′′ values. Similar storage-dependent changes in the viscoelastic properties of mayonnaise have been reported, with the evolution of G′ and G′′ being associated with changes in the emulsion structure during storage (Katsaros et al., 2020).

Oxidative stability during storage

The oxidative stability experiment represents the functional validation of the extract-and-deliver concept under prolonged refrigerated storage. Thus, TBARS values were determined to evaluate the formation of secondary lipid oxidation products during mayonnaise storage. As shown in Fig. 5, TBARS increased in all formulations as storage progressed, confirming mayonnaise's susceptibility to lipid oxidation. At the start of storage, no clear differences emerged among treatments ($P > 0.05$); however, differences became more evident at six months. At this time, MAcCS showed the lowest ($P < 0.05$) TBARS value, while MV showed the highest MDA accumulation, and MVBHA showed an intermediate response. Thus, incorporating acidic corn silk (CS) extract reduced secondary oxidation products more than the formulation containing BHA, a synthetic antioxidant commonly used to delay lipid oxidation in foods. BHA is widely used because it inhibits oxidative reactions; however, its effectiveness in complex food systems can be influenced by the antioxidant's physicochemical characteristics and distribution within the food matrix. In mayonnaise, a compound's antioxidant performance depends heavily on its interaction with the oil-water interface, where lipid oxidation is initiated and propagated (Ghorbani Gorji et al., 2019). The improved performance of MAcCS may therefore reflect not only the amount of antioxidant compounds recovered but also their localization and interactions within the oil-water interfacial region where lipid oxidation is initiated.

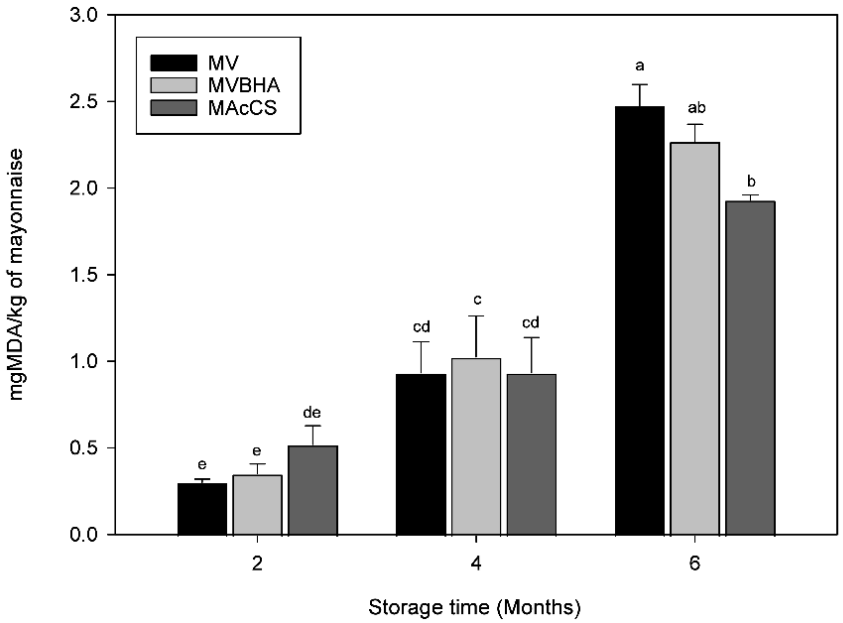


Fig. 5 Changes in TBARS values of mayonnaise formulations during six months of refrigerated storage (4 °C). MV = mayonnaise base with vinegar (4%); MVBHA = mayonnaise base with vinegar (4%) and BHA (100 ppm); MAcCS = mayonnaise base with acidic microemulsion corn silk extract (4%). ^{abc} = different superscripts indicate significant differences among formulations at each storage time by the Tukey-Kramer test ($P < 0.05$).

The lower MDA accumulation in MAcCS may be associated with the polyphenolic compounds present in CS extract. As previously commented, corn silk contains phenolic compounds and flavonoids with antioxidant activity, which can inhibit lipid oxidation by scavenging reactive species and interrupting oxidative chain reactions. The fact that MAcCS showed lower TBARS values than the BHA-containing formulation after prolonged storage is particularly relevant, as it suggests that the CS extract may represent a natural alternative to synthetic antioxidants. Soltan et al. (2023) reported similar protective effects, where Rosa canina extract reduced TBARS formation more effectively than BHT after storage. These findings are particularly noteworthy considering the toxicological concerns reported for tert-butyl-phenolic antioxidants, such as BHA or BHT, including cytotoxic, genotoxic, and other adverse effects under certain exposure conditions, which have encouraged continued research into safer natural alternatives (Hoang & Park, 2024).

Conclusion

Food-grade microemulsions based on D-limonene and Tween 80, with and without acetic acid, showed potential as integrated systems for recovering and delivering corn silk bioactives into a food matrix. AcLiTw enhanced total polyphenol recovery and produced the highest DPPH radical-scavenging activity, while showing a chromatographic fingerprint comparable to the ethanolic extract despite requiring only 30 min of extraction compared with 24 h for ethanol; however, the ethanolic extract showed the highest ABTS activity. Although the extracts showed lower antioxidant capacity than BHA, their incorporation into mayonnaise, particularly the AcLiTw extract, resulted in lower TBARS formation after six months of storage than both the control and BHA-containing formulation. More importantly, the selected system demonstrated a dual technological function by enabling bioactive recovery and subsequent direct incorporation into a complex food emulsion. This integrated approach provides a proof of concept for simplifying the transition from extraction to food formulation and supports food-grade microemulsions as versatile extract-and-deliver platforms for valorizing plant-derived side streams.

Author contributions: Diego E. Carballo: Conceptualization, Formal analysis, Investigation, Methodology, Validation, Writing - original draft, Writing - review & editing. Néstor Gutiérrez-Méndez: Formal analysis, Funding acquisition, Project administration, Resources, Supervision, Validation, Writing - review & editing. Johan Mendoza: Formal analysis, Investigation, Methodology. Diana L. Segovia-Guzmán: Formal analysis, Investigation, Methodology. Martha Yarely Leal-Ramos: Formal analysis. Nubia Ivette Amaya-Olivas: Formal analysis, Methodology. Yamileth Cruz-Trillo: Formal analysis, Methodology.

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Data Availability: No datasets were generated or analyzed during the current study.

Competing Interest Declaration: The authors declare that they have no competing interests as defined by Springer or other interests that might be perceived to influence the results and/or discussion reported in this paper.

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