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Development of Multiple Emulsions Containing Corn Husk Extracts for Cosmetic Applications

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Abstract

Corn (*Zea mays*) is a widely used agricultural crop in various industries such as food, animal feed, biofuels, and cosmetics. However, corn processing often generates a large amount of agricultural waste, especially corn husks. This research aims to prepare a corn husk extract using ultrasonic extraction techniques to develop a multiple emulsion formulation. A high-energy method was employed to enhance the stability and efficacy of active compounds in the corn husk extract. Experiments showed that the extraction of corn husks using distilled water as a solvent for 1 h yielded an extraction rate of 2.43%. For antioxidant activity, the extract exhibited a FRAP value of 1.62 ± 0.01 mM Fe^{2+}/mg, comparable to ascorbic acid. The ABTS free radical scavenging percentage was 99.24%, comparable to that of ascorbic acid, and the chelating activity was 25.61%. The extract was then incorporated into water-in-oil nanoemulsions. The most stable formulation consisted of a 20% (2.3 *w*/*w*%) corn husk extract solution, a surfactant mixture of Tween 80 and Span80 (24.9 *w*/*w*%), and olive oil (72.8 *w*/*w*%). The physical properties of the formulation showed a droplet size of 21.63 ± 1.04 nm, a polydispersity index of 0.31 ± 0.02, and an encapsulation efficiency of 94.20 ± 0.50%, confirming good encapsulation performance. The extract was then mixed into multiple emulsions. The most stable formulation consisted of a nanoemulsion containing a 20% (20 *w*/*w*%) solution of corn husk extract, a mixed surfactant of Tween 80 (1.25 *w*/*w*%), and water (78.75 *w*/*w*%). The physical properties of the formulation showed a droplet size of 169.97 ± 0.31 nm, a polydispersity index of 0.22 ± 0.01, and a zeta potential of −81.17 ± 0.25 mV; the release study showed a cumulative release of 72% after 12 h. This research highlights the potential of preparing corn husk extract for development into complex emulsion formulations for cosmetic applications.

Keywords: corn husk extract; green extraction; nanoemulsions; multiple emulsions

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1. Introduction

Corn (*Zea mays*) is one of the most widely cultivated cereal crops worldwide and serves as an important source of food, animal feed, and industrial raw materials. The increasing global production and consumption of corn have consequently generated substantial quantities of agricultural residues, including husks, cobs, leaves, and stalks. Among these by-products, corn husk is commonly discarded or utilized as low-value livestock feed, despite its potential as a valuable source of bioactive phytochemicals [1,2].

Previous studies have reported that corn husk contains phenolic acids, particularly ferulic acid, as well as other phenolic compounds and flavonoids [3,4]. A comparative investigation of 23 corn cultivars demonstrated variations in the phenolic and carotenoid compositions of corn husks, with ferulic acid identified as a major phenolic constituent. The extracts also exhibited antioxidant activity, suggesting that genetic variation may influence the phytochemical composition and biological potential of corn husk [5]. These findings support the potential use of corn husk as a source of naturally derived bioactive ingredients for further investigation. In addition, other studies have demonstrated that the phytochemical composition and biological activities of corn husk extracts are strongly dependent on the extraction solvent and processing conditions. Aqueous extraction was reported to produce the highest total phenolic content, whereas extraction with 95% ethanol resulted in superior DPPH radical-scavenging activity and inhibition of lipid oxidation. Notably, ultrasound-assisted water extraction produced an extract with promising skin-related biological activities, including substantial inhibition of nitric oxide production, tyrosinase activity, and collagenase activity. Chemical characterization further revealed the presence of gallic acid and quercetin, respectively, indicating that corn husk is a potential source of phenolic acids and flavonoids relevant to cosmetic applications [6]. In addition, the ultrasound-assisted aqueous extract exhibited no irritation in the HET-CAM assay at a concentration of 10 mg/mL. However, cytotoxicity was observed at the same concentration, emphasizing the importance of optimizing both the formulation and concentration of corn husk extract prior to its incorporation into topical products [6]. The bioactive potential of corn by-products has also been investigated in several studies. Corn silk extracts obtained using different extraction solvents and techniques have demonstrated variations in extraction yield, total phenolic and flavonoid contents, and antioxidant activities, indicating that extraction conditions can substantially influence the resulting phytochemical profile [7]. In particular, phenolic and flavonoid constituents identified in corn silk have been associated with antioxidant and tyrosinase inhibitory activities, suggesting potential applications in skincare and cosmeceutical formulations. Recent investigations have also reported that corn silk extracts can be incorporated into topical formulations while retaining relevant biological activities, supporting its potential as a natural source of cosmetic active ingredients [7].

Phenolic compounds are a diverse group of plant secondary metabolites that exhibit a broad spectrum of biological activities, primarily attributable to their ability to scavenge reactive oxygen species (ROS) through hydrogen atom transfer and electron-transfer mechanisms. Excessive ROS generation can disrupt cellular redox homeostasis and induce oxidative stress, resulting in oxidative damage to cellular components and contributing to various skin-related processes, including premature aging, hyperpigmentation, inflammation, and impaired tissue homeostasis. Accordingly, naturally derived phenolic compounds have received considerable attention as potential active ingredients for cosmetic and cosmeceutical formulations. Beyond their antioxidant properties, numerous phenolic compounds have also been reported to possess anti-inflammatory, antimicrobial, and photoprotective activities, highlighting their potential as multifunctional agents for skin-care applications [8,9]. Despite their considerable biological potential, the practical application of phenolic compounds in topical formulations is frequently constrained by their physicochemical instability and susceptibility to degradation. Exposure to oxygen, light, elevated temperature, and fluctuations in pH can accelerate the degradation of phenolic constituents, potentially reducing their biological efficacy. Moreover, inadequate aqueous solubility, limited skin permeation, and poor compatibility with conventional formulation matrices may further restrict their effective delivery to the skin. Therefore, the development

of suitable delivery systems capable of protecting bioactive compounds while facilitating their incorporation and delivery in topical formulations is of considerable interest.

Nanoemulsion delivery systems have been extensively investigated as potential carriers for poorly soluble and chemically labile bioactive compounds. Nanoemulsions are kinetically stable colloidal dispersions consisting of two immiscible liquid phases, with droplet dimensions typically within the nanometer range. Their small droplet size provides a high interfacial area, which can enhance the dispersion and delivery of active compounds while contributing to favorable sensory and physicochemical properties [10,11]. In topical applications, nanoemulsion systems have been reported to enhance the solubilization and skin permeation of active compounds, improve their apparent bioavailability, and provide a degree of protection against environmental degradation [12–14]. These characteristics make nanoemulsion systems attractive platforms for the incorporation and delivery of plant-derived bioactive compounds in cosmetic formulations.

Among advanced emulsion-based delivery systems, water-in-oil-in-water (W/O/W) multiple emulsions have attracted increasing interest because of their compartmentalized structure and ability to accommodate hydrophilic compounds within internal aqueous droplets. In a W/O/W system, an inner aqueous phase is dispersed within an oil phase, which is subsequently dispersed in a continuous external aqueous phase. This hierarchical structure provides distinct microenvironments that can physically separate active compounds from potentially incompatible formulation components and may enhance the protection of encapsulated compounds. In addition, W/O/W systems have been investigated for their potential to provide controlled and sustained release, improve retention of hydrophilic active ingredients, and enhance the moisturizing characteristics of topical formulations [15–17]. The ability to accommodate multiple phases also provides greater formulation flexibility for incorporating compounds with different physicochemical properties.

However, research on using corn husks, an agricultural byproduct, to develop value-added healthcare products remains limited. Therefore, this study aimed to prepare a crude aqueous extract of corn husk and subsequently incorporate it into a water-in-oil-in-water (W/O/W) nanoemulsion system. The developed formulations were systematically characterized for physicochemical properties, morphological characteristics, rheological behavior, physical stability, antioxidant activity, and in vitro release profiles. The findings were intended to provide insight into the feasibility of W/O/W nanoemulsions as delivery systems for corn husk-derived bioactive compounds and to assess their potential for further development as cosmetic formulations.

2. Materials and Methods

2.1. Materials

Folin–Ciocâlteu was obtained from Merck (Darmstadt, Germany). Analytical chemicals such as gallic acid, 2,4,6-tris(2-pyridyl)-s-triazine, 3-(2-pyridyl)-5,6-diphenyl-1,2,4-triazine-p,p′-disulfonic acid monosodium salt hydrate, 2,2′-azino-bis(3-ethylbenzothiazoline-6-sulfonic acid) diammonium salt, iron(II) chloride tetrahydrate, and sodium acetate trihydrate were supplied by Sigma (St. Louis, MO, USA). Other analytical chemicals, such as potassium persulfate and ethylenediaminetetraacetic acid, disodium salt dihydrate, were supplied by Labscan Asia Co., Ltd. (Bangkok, Thailand). Other analytical chemicals, such as ferrous sulfate, ascorbic acid, ferric chloride hexahydrate, and sodium carbonate anhydrous, were supplied by Loba Chemie Pvt., Ltd. (Mumbai, India). Solvents and additional reagents, such as ethanol and DMSO, were supplied by Labscan Asia Co., Ltd. (Bangkok, Thailand). Span 80, Tween 80, and olive oil were purchased from Namsiang Co., Ltd. (Bangkok, Thailand). Centrifugal filter units were supplied by Merck Millipore Ltd. (Tullagreen,

Ireland). The dialysis membrane was supplied by Beijing Labgic Technology Co., Ltd. (Beijing, China).

2.2. Plant Material

The corn husks, aged 110–120 days with a yellow color, were collected from Mae Tha district, Lampang province, Thailand. The corn husks were cleaned, dried, and then cut into smaller pieces (about 1 cm) using scissors. The cut corn husks were placed in a blender, about a handful at a time, and blended until coarse for about 2 min [7].

2.3. Extraction of Corn Husk

The corn husk extraction procedure was performed based on a previously reported method [7]. 30 g of dried corn husk powder was soaked in 300 mL of DI water, then subjected to an ultrasonic bath for 1 h at 37 ± 2 °C. After that, it was filtered with a filter cloth, followed by filtration with Whatman No. 1 filter paper using a suction filtration kit. The solvent was removed using a rotary evaporator. The percentage yield is calculated using the following formula:

$$\text{Yield } (\%) = \frac{\text{weight of extract}}{\text{weight of plant used for extraction}} \times 100$$

2.4. Antioxidant Activity

2.4.1. Ferric Reducing Antioxidant Power (FRAP)

The FRAP testing solution was mixed with five different concentrations of corn husk extract and incubated in the dark for 30 min. Ferrous sulfate ($FeSO_4$) served as the reference standard, while ascorbic acid was used as the positive control. The absorbance of each concentration was measured at 593 nm using a microplate reader. The percentage of FRAP activity was calculated using the equation: FRAP activity (%) = [(A − B)/A] × 100, where A represents the absorbance of the control, and B represents the absorbance of the sample. A calibration curve of $FeSO_4$ was generated to determine the FRAP value of each sample, expressed as mg $FeSO_4$ equivalent per mg of sample [18].

2.4.2. ABTS Scavenging Activity

The ABTS testing solution was combined with five concentrations of corn husk extract and incubated in the dark for 6 min. Ascorbic acid was used as the positive control. The absorbance of each sample was measured at 734 nm using a microplate reader. The percentage of ABTS scavenging activity was calculated using the following equation: ABTS activity (%) = [(A − B)/A] × 100, where A was the absorbance of the control, and B was the absorbance of the sample [19].

2.4.3. Metal-Chelating Activity

The metal-chelating testing solution was mixed with five concentrations of corn husk extract and incubated in the dark for 15 min. EDTA served as the positive control. The absorbance of each concentration was measured at 570 nm using a microplate reader. The percentage of metal-chelating activity was calculated as follows: metal-chelating activity (%) = [(A − B)/A] × 100, where A was the absorbance of the control, and B was the absorbance of the sample [20].

2.5. Development of W/O Nanoemulsion Base

2.5.1. Preparation of W/O Nanoemulsion Base

The W/O nanoemulsion base was prepared using a high-energy method. All formulations were prepared as described in Table 1. The oil phase consisted of olive oil mixed

with a surfactant mixture (Tween 80 and Span 80) at a ratio of 1:3, and the aqueous phase consisted of DI water [21]. In the high-energy method, the oil and aqueous phases were homogenized using a high-shear homogenizer (T18 digital ULTRA-TURRAX, IKA-Werke GmbH & Co. KG, Staufen, Germany) for 10 min at 5000 rpm to produce a coarse emulsion. Then, nanoemulsions were produced using an ultrasonicator (Vibra-Cell, Sonics & Materials, Inc., Newtown, CT, USA) at 50% amplitude for 10 min [22].

Table 1. The composition of the W/O nanoemulsion base.

Ingredient	Concentration (% *w*/*w*)		
	F1	**F2**	**F3**
Olive oil	q.s. 100	q.s. 100	q.s. 100
Span 80	7.8	9	18.68
Tween 80	2.2	2.7	6.22
DI water	3	3	2.3

2.5.2. Characterization of the W/O Nanoemulsion Base

The W/O nanoemulsion base was evaluated for external appearance through visual inspection. The droplet size and polydispersity index (PDI) were determined using a Zetasizer (SZ-100, HORIBA, Ltd., Kyoto, Japan). Measurements were conducted at room temperature (25 °C) with a scattering angle of 90° [23].

2.6. Development of W/O Nanoemulsions Containing Corn Husk Extract

2.6.1. Preparation of W/O Nanoemulsions Containing Corn Husk Extract

The W/O nanoemulsion base with optimal physicochemical characteristics, such as small droplet size and a narrow PDI range (between 0.1 and 0.3), was selected for the incorporation of the highest concentration of corn husk extract. All formulations were prepared as described in Table 2. Nanoemulsion formulations containing corn husk extract were prepared using the high-energy method [22]. The oil phase, where olive oil was mixed with a surfactant mixture (Tween 80 and Span 80) at a ratio of 1:3, and the aqueous phase, where corn husk extract solution with concentrations between 10, 15, 20, and 25% was used as the aqueous phase.

Table 2. The composition of the W/O nanoemulsions containing corn husk extract.

Ingredient	Concentration (%*w*/*w*)
Olive oil	q.s. 100
Span 80	18.68
Tween 80	6.22
Corn husk extract solution	2.3

2.6.2. Characterization of W/O Nanoemulsions Containing Corn Husk Extract

The prepared W/O nanoemulsions containing corn husk extract were assessed for external appearance via visual inspection. The droplet size and PDI were measured using a Zetasizer (SZ-100, HORIBA, Ltd., Kyoto, Japan) at room temperature (25 °C) with a scattering angle of 90°. Nanoemulsions containing 10% and 15% corn husk extract were diluted 2-fold with olive oil before measurement, while nanoemulsions containing 20% and 25% corn husk extract were diluted 5-fold with olive oil before measurement [23].

2.6.3. The Encapsulation Efficiency of Nanoemulsions Containing Corn Husk Extract

The encapsulation efficiency (EE) of the nanoemulsions containing corn husk extract was determined by using a membrane filtration method. The nanoemulsions were passed

through a centrifugal filter with a 100 kDa molecular weight cut-off and centrifuged at 12,000 rpm for 30 min. The filtrate was collected to determine the amount of encapsulated corn husk extract. Total phenolic content (TPC) in both the original nanoemulsions and the encapsulated liquid was measured using the Folin-Ciocâlteu method [24]. Each sample was mixed with 15% sodium carbonate (Na_2CO_3) and 50% v/v Folin–Ciocâlteu reagent. After incubation for 120 min in the dark at room temperature, the absorbance was measured at 655 nm using a microplate reader (SpectraMax M3, Molecular Devices, San Jose, CA, USA). Results were expressed as mg gallic acid equivalents per liter (mg GA/L) [25]. The percentage of EE was calculated using the following formula:

$$\text{Encapsulation Efficiency } (\%) = \frac{\text{TPC in entrapped}}{\text{TPC in nanoemulsions}} \times 100$$

2.6.4. Stability Test of Nanoemulsions Containing Corn Husk Extract

The stability of nanoemulsions containing corn husk extract was determined by centrifugation and heating-cooling tests for 6 cycles. From the centrifugation test, nanoemulsions containing corn husk extract were centrifuged at 4000 rpm for 15 min and observed for external appearance and phase separation [26]. In the heating-cooling test, nanoemulsions containing corn husk extract were placed in a 20 mL vial and kept at 4 °C for 24 h, followed by 45 °C for 24 h as 1 cycle. After 6 cycles, the samples were measured for phase separation, droplet size, PDI, and total phenolic content [27].

2.7. Development of Multiple Emulsions Containing Corn Husk Extract

2.7.1. Preparation of Multiple Emulsions Containing Corn Husk Extract

The multiple emulsion formulation was prepared using the high-energy method. The oil phase, which contained corn husk extract, and the aqueous phase, where DI water was mixed with Tween 80, were used [28]. All formulations were prepared as described in Table 3. The oil and aqueous phases were homogenized using a high-shear homogenizer (T18 digital ULTRA-TURRAX, IKA-Werke GmbH & Co. KG, Staufen, Germany) for 10 min at 5000 rpm to produce a coarse emulsion. Then, using an ultrasonicator (Vibra-Cell, Sonics & Materials, Inc., Newtown, CT, USA) at 50% amplitude for 10 min [22].

Table 3. Composition of multiple emulsions containing corn husk extract.

Ingredient	Concentration (% *w*/*w*)			
	MEs-Blank1	**MEs-Blank2**	**MEs-Extract1**	**MEs-Ectract2**
DI water	q.s. 100	q.s. 100	q.s. 100	q.s. 100
Tween80	1.25	1.5	1.25	1.5
W/O nanoemulsion base	20	20	-	-
W/O nanoemulsions containing corn husk extract	-	-	20	20

Note: MEs = multiple emulsions.

2.7.2. Characterization of Multiple Emulsions Containing Corn Husk Extract

Droplet Size, PDI, and Zeta Potential

The droplet size, PDI, and zeta potential were determined using a Zetasizer (Horiba nanoparticle analyzer SZ-100, Kyoto, Japan). Measurements were conducted at room temperature (25 °C) with a scattering angle of 173° [29]. Samples were diluted with DI water (1:100) before being measured [30].

Morphology Study

The morphology of the multiple emulsions was examined using a low-voltage transmission electron microscope (LVEM 25E, Delong Instruments, Brno, Czech Republic).

Drops of the sample were placed on a carbon-coated copper grid and allowed to form a thin film, and the grid was air-dried at room temperature. Droplet shape, size, and structural characteristics were analyzed [30].

The Rheological Study

The rheological properties of the multiple emulsions were characterized using a rotational rheometer (Thermo Scientific MARS 60, Thermo Fisher Scientific, Bremen, Germany) equipped with a concentric cylinder geometry. Measurements were performed at a constant frequency of 1.0 Hz over a shear rate range of 1–10 s^{-1}. All measurements were carried out at 25 °C [31]. Rheological measurements were performed solely on Day 0 to assess the flow behavior of the freshly prepared formulations.

The Release Study

The release study of multiple emulsions was conducted using the dialysis method. Corn husk solution was used as the control solution. 10 mL of each sample was placed in a dialysis membrane and immersed in 100 mL of the release medium (PBS, 0.01 M, pH 6.8: DI water, 7:3) at 37 °C with a stirring speed of 100 rpm. At the intervals, 1 mL of each sample was withdrawn from the medium, and 1 mL of fresh medium was added to maintain equilibrium. The total phenolic content released was measured using the Folin–Ciocâlteu method absorbance at 655 nm, and cumulative release was calculated over 12 h [31]. The percentage of cumulative release was calculated using the following formula:

$$\text{Cumulative release } (\%) = \frac{\text{Amount of TPC released}}{\text{Intitial TPC amount}} \times 100$$

2.7.3. Stability Test of Multiple Emulsions Containing Corn Husk Extract

The multiple emulsions were kept at different temperatures for 30 days. Different temperature conditions were applied: room temperature (25 °C ± 1 °C), low temperature (4 °C ± 1 °C), and high temperature (45 °C ± 1 °C). At the determined intervals (24 h, 48 h, 7 d, 15 d, and 30 d), samples were removed and allowed to reach room temperature to evaluate droplet size, PDI, zeta potential [32], and total phenolic content, which was slightly modified and determined as previously described [33].

2.8. Statistical Analysis

All reported results were expressed as means ± SD (n = 3). Statistical significance was assessed by analysis of variance (ANOVA) and t-test, and the value of $p < 0.05$ was considered significant.

3. Results and Discussion

3.1. Corn Husk Extraction

In the study of corn husk extraction by ultrasound-assisted extraction, the appearance of the crude extract was as shown in Figure 1. The crude extract was brownish-yellow, sticky, and semi-solid. The percentage yield obtained from the extraction was 2.43%.



Figure 1. The appearance of the corn husk crude extract.

3.2. Antioxidant Activity

3.2.1. FRAP

The FRAP assay was performed to evaluate and compare the antioxidant activity of corn husk extract at different concentrations (between 100 and 0.16 mg/mL). The results are shown in Table 4. Ascorbic acid, used as a positive control, showed a FRAP value of 2.13 ± 0.01 mM Fe^{2+}/mg sample, while the corn husk extract showed the highest FRAP value of 1.62 ± 0.01 mM Fe^{2+}/mg sample. Overall, the corn husk extract exhibited concentration-dependent antioxidant activity with higher ferric-scavenging capacity.

Table 4. FRAP values for ascorbic acid and corn husk extract.

Working Conc. (mg/mL)	FRAP Value (mM Fe^{2+}/mg Sample)	
	Ascorbic Acid	Corn Husk Extract
100	2.13 ± 0.01	1.62 ± 0.01
20	1.63 ± 0.01	1.54 ± 0.01
4	1.58 ± 0.00	0.78 ± 0.01
0.8	1.29 ± 0.01	Below the limit of quantification
0.16	0.37 ± 0.01	Below the limit of quantification

3.2.2. ABTS Scavenging Activity

The ABTS assay was performed to evaluate the antioxidant activity of ascorbic acid and corn husk extract at various concentrations ranging from 0.16 to 100 mg/mL, as shown in Table 5. Ascorbic acid, used as a positive control, showed the highest ABTS scavenging activity with a percentage of 99.78 ± 0.67%. The corn husk extract showed the highest ABTS scavenging activity with a percentage of 99.24 ± 0.37%. These research results demonstrated that the corn husk extract possessed antioxidant properties.

Table 5. Percentage of ABTS scavenging and percentage of metal-chelating activity between ascorbic acid and corn husk extract.

Working Conc. (mg/mL)	% ABTS Scavenging Activity		% Metal-Chelating Activity	
	Ascorbic Acid	Corn Husk Extract	EDTA	Corn Husk Extract
100	99.78 ± 0.67	99.24 ± 0.37	99.38 ± 0.11	25.19 ± 0.63
20	96.87 ± 0.06	84.01 ± 0.03	55.65 ± 0.45	Below the limit of quantification
4	77.78 ± 0.33	21.62 ± 0.16	29.37 ± 0.30	Below the limit of quantification
0.8	20.28 ± 0.64	5.10 ± 0.52	20.29 ± 0.39	Below the limit of quantification
0.16	1.70 ± 0.18	0.94 ± 0.36	8.60 ± 0.56	Below the limit of quantification

3.2.3. Metal-Chelating Activity

The metal-chelating activity of corn husk extract was evaluated at concentrations ranging from 0.16 to 100 mg/mL, and the results are shown in Table 5. EDTA, used as a positive control, exhibited strong metal-chelating activity, with the highest percentage of chelating activity at 99.38 ± 0.11%. In contrast, the corn husk extract showed relatively low chelating activity at 25.19 ± 0.63%. The high metal-chelating activity found in EDTA was expected in comparison to the relatively weak chelating activity of the corn husk extract. This result indicated that the bioactive compounds present in the extract have limited metal-chelating potential. Although corn husk contained phenolic and flavonoid compounds that might contribute to antioxidant activity, these compounds might not possess sufficient functional groups to effectively chelate iron ions. The slight increase in chelating activity at higher concentrations suggested that the extract's metal-chelating ability was concentration-dependent. The results indicated that the corn husk extract exhibited weak metal-chelating activity compared to EDTA. However, the detected metal-chelating effect at high concentrations suggested that the extract might still contribute to

antioxidant defense mechanisms through limited metal-ion chelation, in addition to its scavenging and free-radical scavenging activities.

3.3. Characterization of W/O Nanoemulsion Bases

3.3.1. Appearance

In the study of the external appearance of W/O nanoemulsions prepared from different components, as shown in Figure 2, it was revealed that both formulas F1 and F2 exhibited turbidity and no phase separation, while formula F3 was clear and showed no phase separation. When the formulas were left for 24 h, all nanoemulsions showed no phase separation.



(**a**) (**b**) (**c**)

Figure 2. The external appearance of the W/O nanoemulsion base. (**a**) Formulation 1 (F1); (**b**) Formulation 2 (F2); (**c**) Formulation 3 (F3).

3.3.2. Droplet Size and PDI

The study of the droplet size and PDI values of water-in-oil nanoemulsions formulations F1, F2, and F3 is shown in Table 6. It was found that the droplet size of formulation F3 was 21.03 ± 0.379 nm, which was the smallest droplet size, and the droplet size was in the appropriate range for nanoemulsion formulations (20–200) nm [10]. Formulation F3 had a PDI of the formulation of 0.247 ± 0.051, which was considered to have good distribution [11]. The smaller droplet size observed for F3 compared with F1 and F2 suggested that the composition of F3 provided more favorable conditions for droplet disruption and stabilization during the emulsification process. In particular, the surfactant system played an important role in reducing interfacial tension between the oil and aqueous phases and stabilizing the newly generated droplets against coalescence. An appropriate surfactant concentration could therefore facilitate the formation of smaller droplets during high-energy emulsification [34]. Moreover, droplet size was also influenced by the viscosity ratio between the dispersed and continuous phases and by the energy density applied during homogenization [34]. Therefore, formulation F3 was selected for development to apply corn husk extract to this formulation.

Table 6. Characterization of the nanoemulsions base.

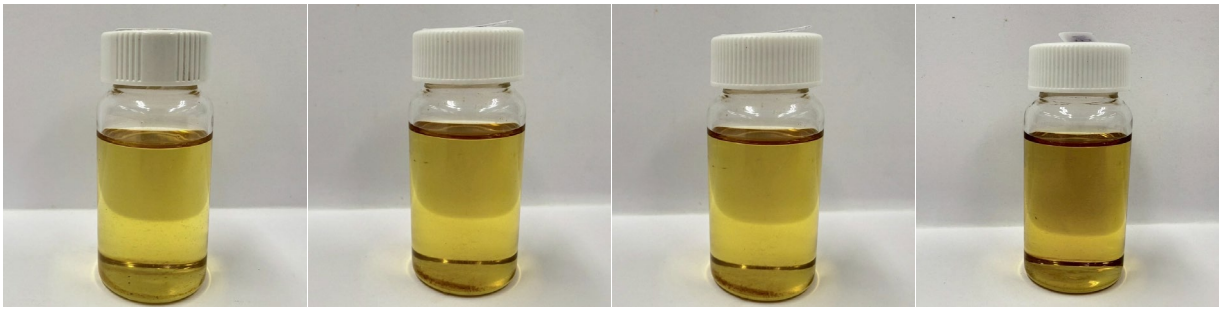
Physical Characteristic	Formulation		
	F1	F2	F3
Droplet size (nm)	295.07 ± 1.12 [a]	269.70 ± 1.61 [a]	20.23 ± 0.12 [b]
PDI	0.697 ± 0.11 [a]	0.647 ± 0.14 [a]	0.235 ± 0.02 [b]

The data were presented as mean ± SD (n = 3). Statistical significance at $p < 0.05$ among groups was denoted by superscript letters, with comparisons evaluated by Tukey's HSD test in one-way ANOVA.

3.4. Characterization of W/O Nanoemulsions Containing Corn Husk Extract

3.4.1. Appearance

The study of the external appearance of W/O nanoemulsions containing corn husk extract at different concentrations (ranging from 10 to 25%*w*/*w*), as shown in Figure 3, revealed that the formula with 10% corn husk solution was the clearest and showed no phase separation, while the formulas with 15%, 20%, and 25% corn husk solutions were slightly cloudy and also showed no phase separation. After 24 h, none of the nanoemulsion formulations showed increased cloudiness or phase separation.



(**a**) (**b**) (**c**) (**d**)

Figure 3. The external appearance of W/O nanoemulsions containing corn husk extract. (**a**) Nanoemulsions with 10% corn husk extract; (**b**) nanoemulsions with 15% corn husk extract; (**c**) nanoemulsions with 20% corn husk extract; (**d**) nanoemulsions with 25% corn husk extract.

3.4.2. Droplet Size and PDI

The study of droplet size and PDI of nanoemulsions containing corn husk extract, as shown in Table 7, revealed the droplet size and PDI values. Nanoemulsions with 10% extract and 15% extract showed a decrease in droplet size compared to the nanoemulsion base, decreasing from 21.03 ± 0.379 nm to 17.13 ± 1.002 nm with 10% extract and to 19.07 ± 1.563 nm with 15% extract. Nanoemulsions with 20% extract and 25% extract showed an increase in droplet size compared with the nanoemulsion base, increasing from 21.03 ± 0.379 nm to 21.63 ± 1.041 nm with 20% extract and to 64.97 ± 2.312 nm with 25% extract. Despite this increase in droplet size, it remained within the optimal range for the nanoemulsion formulation. This reduction in droplet size may be associated with changes in the physicochemical properties of the dispersed phase and the interfacial environment following incorporation of the extract. The composition of the dispersed phase could influence interfacial tension and the efficiency of droplet disruption during homogenization, thereby affecting the final droplet size of nanoemulsions [34]. Similar findings have been reported in nanoemulsion systems in which changes in formulation composition and surfactant-to-oil ratios resulted in substantial changes in droplet size and PDI [35]. This table showed an increase in PDI from 0.247 ± 0.051 to 0.253 ± 0.02 when 10% extract was added, increased to 0.288 ± 0.028 when 15% extract was added, increased to 0.314 ± 0.018 when 20% extract was added, and increased to 0.625 ± 0.069 when 25% extract was added. It was found that nanoemulsions with 25% extract showed more dispersion, which might lead to long-term instability of the formula and might cause precipitation or separation. Both droplet size and PDI increased significantly when 25% extract was added compared to the formulas with 10%, 15%, and 20% extracts ($p < 0.05$).

Table 7. Characterization of W/O nanoemulsions containing corn husk extract.

Physical Characteristic	Formulation			
	NEs-Extract10	**NEs-Extract15**	**NEs-Extract20**	**NEs-Extract25**
Droplet size (nm)	17.13 ± 1.00 [a]	19.07 ± 1.56 [a]	21.63 ± 1.04 [a]	64.97 ± 1.31 [b]
PDI	0.253 ± 0.02 [a]	0.288 ± 0.02 [a]	0.314 ± 0.02 [a]	0.625 ± 0.06 [b]

Note: NEs = nanoemulsions. The data were presented as mean ± SD (n = 3). Statistical significance at $p < 0.05$ among groups was denoted by superscript letters, with comparisons evaluated by Tukey's HSD test in one-way ANOVA.

3.4.3. Encapsulation Efficiency (EE)

The encapsulation efficiency of the nanoemulsions containing corn husk extract was evaluated, as shown in Table 8. The results indicated that encapsulation efficiency increased with higher concentrations of corn husk extract. This trend suggested that a greater amount of extract was successfully entrapped within the oil droplets when a higher extract loading was applied. No detectable unencapsulated corn husk extract was observed in the supernatant after centrifugation under the experimental conditions used. The increase in EE with increasing extract concentration might be related to the partitioning behavior of phytochemical constituents among the oil phase, interfacial region, and aqueous phase. During nanoemulsion formation, the encapsulation or retention of bioactive compounds was governed by their physicochemical properties, including polarity, solubility, and affinity for the different phases of the emulsion, as well as by the composition and concentration of the emulsifier system [34]. Overall, the high encapsulation efficiency values obtained across all formulations (>82%) indicated that the nanoemulsion system was effective for encapsulating corn husk extract.

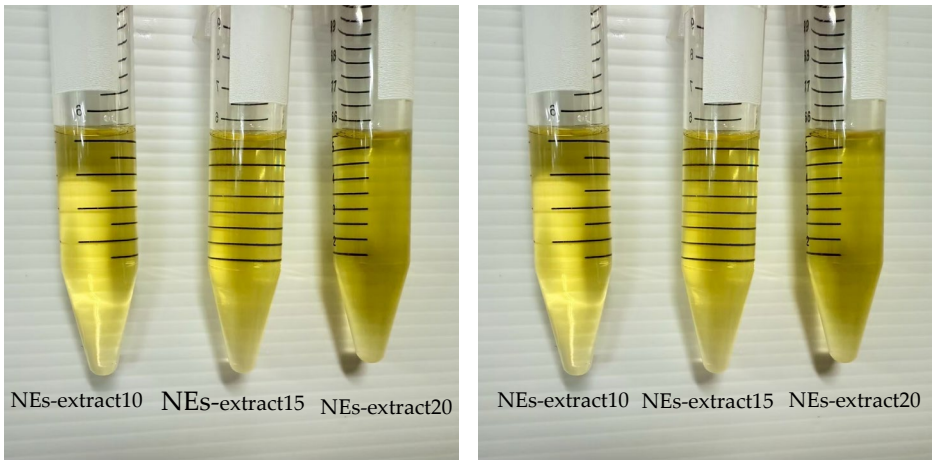
Table 8. The percentage encapsulation efficiency of W/O nanoemulsions containing corn husk extract.

Sample	EE (%)
NEs-extract20	94.20 ± 0.50 [a]
NEs-extract15	91.46 ± 0.60 [b]
NES-extract10	82.07 ± 1.00 [c]

Note: NEs = nanoemulsions. The data were presented as mean ± SD (n = 3). Statistical significance at $p < 0.05$ among groups was denoted by superscript letters, with comparisons evaluated by Tukey's HSD test in one-way ANOVA.

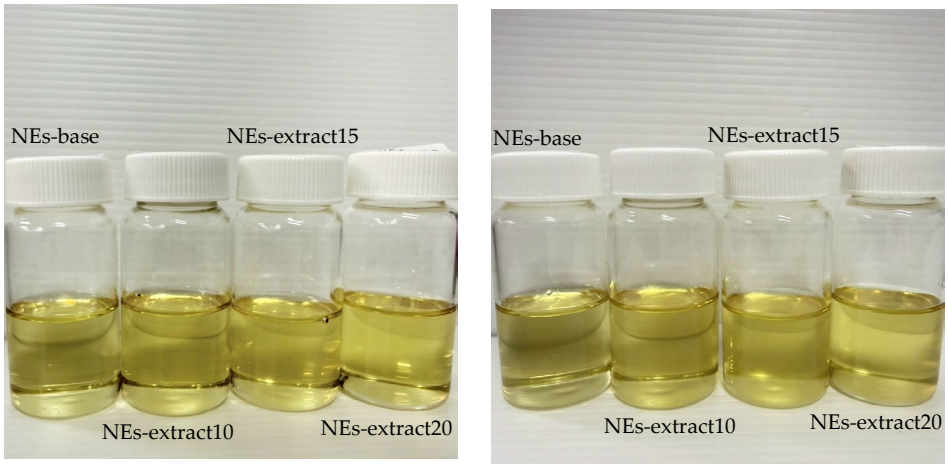
3.4.4. Stability Test of W/O Nanoemulsions Containing Corn Husk Extract

The stability of the nanoemulsions containing corn husk extract was assessed by a centrifugation test. Before testing, the nanoemulsions containing corn husk extract were clear and showed no phase separation (Figure 4a). After testing, their external appearance (Figure 4b) showed no precipitation or separation. Before the heating-cooling test, the external appearance of the formulations remained clear and did not separate, as shown in Figure 5a. After the heating-cooling test, the external appearance of the formulations remained clear and did not separate, as shown in Figure 5b. Table 9 illustrates the physical characteristics of droplet size, PDI, and TPC. F3 and NEs-base, which were the same formulation, were investigated for droplet size and PDI before the stability test. After the stability test, there was a statistically significant increase in both droplet size and PDI, while TPC decreased significantly.



(**a**) (**b**)

Figure 4. The external appearance of W/O nanoemulsions containing corn husk extract (**a**) before the centrifugation test and (**b**) after the centrifugation test.



(**a**) (**b**)

Figure 5. NEs = nanoemulsions. The external appearance of W/O nanoemulsions base and nanoemulsions containing corn husk extract (**a**) before the heating-cooling test and (**b**) after 6 cycles of the heating-cooling test.

Table 9. Characterization of W/O nanoemulsions containing corn husk extract before and after the heating-cooling test.

Sample	Before Heating-Cooling Test			After Heating-Cooling Test		
	Droplet Size (nm)	PDI	TPC (mg GA/L)	Droplet Size (nm)	PDI	TPC (mg GA/L)
NEs-base	20.23 ± 0.12	0.235 ± 0.02	ND	32.87 ± 1.32 *	0.479 ± 0.04 *	ND
NEs-extract10	14.87 ± 1.25	0.217 ± 0.20	224.26 ± 1.72	20.80 ± 0.40 *	0.299 ± 0.01 *	99.26 ± 1.54 *
NEs-extract15	18.53 ± 0.80	0.270 ± 0.01	261.76 ± 0.36	24.07 ± 0.35 *	0.347 ± 0.03 *	199.47 ± 0.32 *
NEs-extract20	21.00 ± 0.66	0.323 ± 0.01	389.99 ± 0.65	27.17 ± 1.50 *	0.336 ± 0.01 *	327.49 ± 0.58 *

Note: NEs = nanoemulsions; ND = not detected. Data are presented as mean ± SD (n = 3). * = a statistically significant difference at $p < 0.05$, as determined using a paired-sample t-test.

3.5. Characterization of Multiple Emulsions Containing Corn Husk Extract

3.5.1. Appearance

The study of the external appearance of the multiple emulsions, both with and without corn husk extract, as shown in Figure 6, revealed that the formulation without corn husk extract was cloudy white and no phase separation. Similarly, the formulation with corn

husk extract was also cloudy white and no phase separation. After 24 h, all formulations still showed no phase separation.



(**a**) (**b**) (**c**) (**d**)

Figure 6. The external appearance of multiple emulsions: (**a**) multiple emulsions blank1, (**b**) multiple emulsions blank2, (**c**) multiple emulsions extract1, and (**d**) multiple emulsions extract2.

3.5.2. Droplet Size, PDI, and Zeta Potential

The physical characteristics of the multiple emulsions, including droplet size, polydispersity index (PDI), and zeta potential, were summarized in Table 10. The blank formulations, MEs-blank1 and MEs-blank2, exhibited mean droplet sizes of 161.43 ± 0.61 and 162.63 ± 0.87 nm, respectively. Following incorporation of corn husk extract, the droplet size increased significantly ($p < 0.05$) to 169.97 ± 0.31 nm for MEs-extract1 and 170.67 ± 0.68 nm for MEs-extract2. An increase in droplet size following incorporation of an active compound could be associated with changes in the composition and interfacial properties of the multiple emulsion system. In W/O/W multiple emulsions, droplet size was influenced by factors including the composition and volume fraction of the phases, emulsifier type and concentration, viscosity of the phases, and emulsification conditions [36,37]. Therefore, the observed increase in droplet size after addition of corn husk extract might reflect changes in the physicochemical environment of the emulsion droplets caused by incorporation of the extract. Despite the significant increase in droplet size, the PDI values remained highly consistent among all formulations, ranging from 0.223 ± 0.02 to 0.225 ± 0.01, with no statistically significant difference among formulations ($p > 0.05$). This narrow range indicated that incorporation of corn husk extract did not substantially alter the breadth of the droplet-size distribution under the conditions investigated. A relatively low and consistent PDI suggested that the emulsification process produced a comparatively homogeneous population of droplets. In multiple-emulsion systems, droplet size distribution was affected by emulsification conditions, phase composition, surfactant concentration, and the mechanical forces applied during preparation [38]. The maintenance of a similar PDI after extract incorporation therefore suggested that the addition of corn husk extract did not markedly disrupt the overall droplet-formation process. The zeta potential values were strongly negative for all formulations. The blank formulations exhibited zeta potentials of −89.07 ± 0.15 mV for MEs-blank1 and −88.93 ± 0.15 mV for MEs-blank2. Following incorporation of corn husk extract, the zeta potential became significantly less negative ($p < 0.05$), reaching −81.17 ± 0.25 mV for MEs-extract1 and −80.83 ± 0.50 mV for MEs-extract2. The decrease in the magnitude of the negative zeta potential might be related to changes in the interfacial composition or surface environment following incorporation of the extract. Bioactive compounds presented in plant extracts might be distributed within different regions of an emulsion, including the aqueous phase and interfacial region, and could consequently alter the surface charge measured by electrophoretic mobility [38]. However, the zeta potential change observed here should not be interpreted as direct evidence of a specific molecular interaction between corn husk phytochemicals and the emulsifiers because such interactions

were not directly characterized in the present study. Despite the statistically significant reduction in the magnitude of the zeta potential after extract incorporation, the measured values remained strongly negative, ranging from approximately −81 to −89 mV. High-magnitude zeta potential values were generally associated with stronger electrostatic repulsive forces between charged droplets, which could reduce the tendency of droplets to approach and aggregate. Nevertheless, zeta potential should not be considered an independent measure of long-term multiple emulsion stability because steric stabilization, interfacial film properties, surfactant composition, droplet size, and the complex internal structure of multiple emulsions could also contribute substantially to physical stability [36,37].

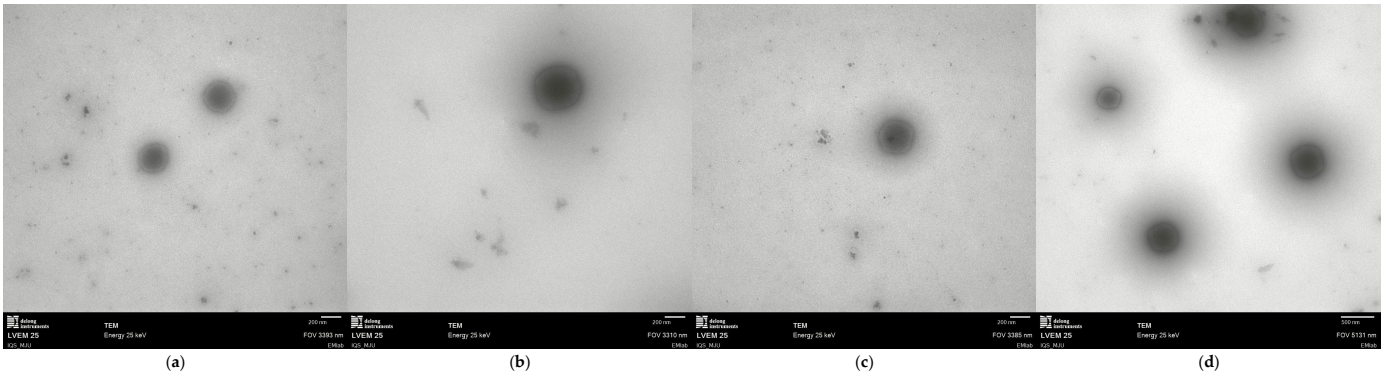
Table 10. Physical characterization of multiple emulsions blanks and multiple emulsions containing corn husk extract.

Physical Characteristic	Formulation			
	MEs-Blank1	**MEs-Blank2**	**MEs-Extract1**	**MEs-Extract2**
Droplet size (nm)	161.43 ± 0.61 ^{a}	163.07 ± 0.56 ^{a}	169.97 ± 0.31 ^{a}	170.67 ± 0.68 ^{b}
PDI	0.223 ± 0.02 ^{a}	0.228 ± 0.02 ^{b}	0.224 ± 0.01 ^{a}	0.226 ± 0.01 ^{b}
Zeta potential (mV)	−89.07 ± 0.15	−88.93 ± 0.15	−81.17 ± 0.25	−80.83 ± 0.50

Note: MEs = Multiple emulsions. The data were presented as mean ± SD (n = 3). Statistical significance at $p < 0.05$ among groups was denoted by superscript letters, with comparisons evaluated by Tukey's HSD test in one-way ANOVA.

3.5.3. Morphology Study

Micromorphological studies of multiple emulsions were performed using TEM to examine the shape and surface morphology of both the multiple emulsion base and corn husk extract-containing multiple emulsions. Microscopic images showed that most of the formulations exhibited spherical droplets with a relatively uniform size distribution and no evidence of extensive aggregation or fusion, as shown in Figure 7. The droplets in the evaluated samples showed good, uniform distribution, a spherical shape, and an average particle size below 200 nm, consistent with droplet size and PDI measurements. Furthermore, the images revealed nanoparticles separated into clearly defined layers, indicating successful formation of a stable multiple emulsion system.



(**a**) (**b**) (**c**) (**d**)

Figure 7. The microscopic morphology of multiple emulsions by TEM: (**a**) multiple emulsions blank 1, (**b**) multiple emulsions 2, (**c**) multiple emulsions extract 1, and (**d**) multiple emulsions extract 2.

3.5.4. The Release Study

We investigated and compared the release of total phenolic compounds from free corn husk extract and corn husk extract incorporated into multiple emulsions. The cumulative release of total phenolic compounds from each sample was assessed over time to assess the

effect of combining the extracts with multiple emulsion systems on the release of phenolic compounds, as shown in Figure 8. The results showed that the free corn husk extract released faster than the multiple emulsions. After 12 h, the cumulative release of the free corn husk extract was approximately 92%, while the multiple emulsion released only 72%. The slower release observed from the multiple emulsion may be associated with the more complex structural organization of the multiple-emulsion system. In a W/O/W multiple emulsion, the encapsulated aqueous phase was surrounded by an oil phase and subsequently dispersed within an external aqueous phase. Consequently, incorporated compounds must undergo a more complex transport pathway before reaching the external release medium compared with the free extract, which is directly exposed to the release medium. The oil phase and interfacial layers could therefore act as diffusion barriers that retard the transfer of encapsulated compounds from the internal aqueous phase to the external aqueous phase [36,37]. The release behavior of W/O/W multiple emulsions was not governed solely by passive diffusion through the oil phase. Previous studies have reported that release from multiple emulsions might involve diffusion through the oil membrane, transport facilitated by surfactant-associated micellar structures, and swelling or breakdown of the oil globules. The relative contribution of these mechanisms depended on the composition and structural characteristics of the multiple-emulsion system [39,40]. Therefore, the slower release observed in the present study might reflect the combined effects of the oil phase, interfacial films, surfactants, and the internal structure of the multiple emulsions rather than the oil phase acting as an absolute barrier. The difference in cumulative release between the free extract and the multiple-emulsion formulation also suggested that encapsulation could provide a more prolonged release profile for corn husk extract. Similar controlled-release characteristics have been reported for multiple-emulsion systems, which have been investigated as carrier systems for sustained delivery of active compounds [36,37].

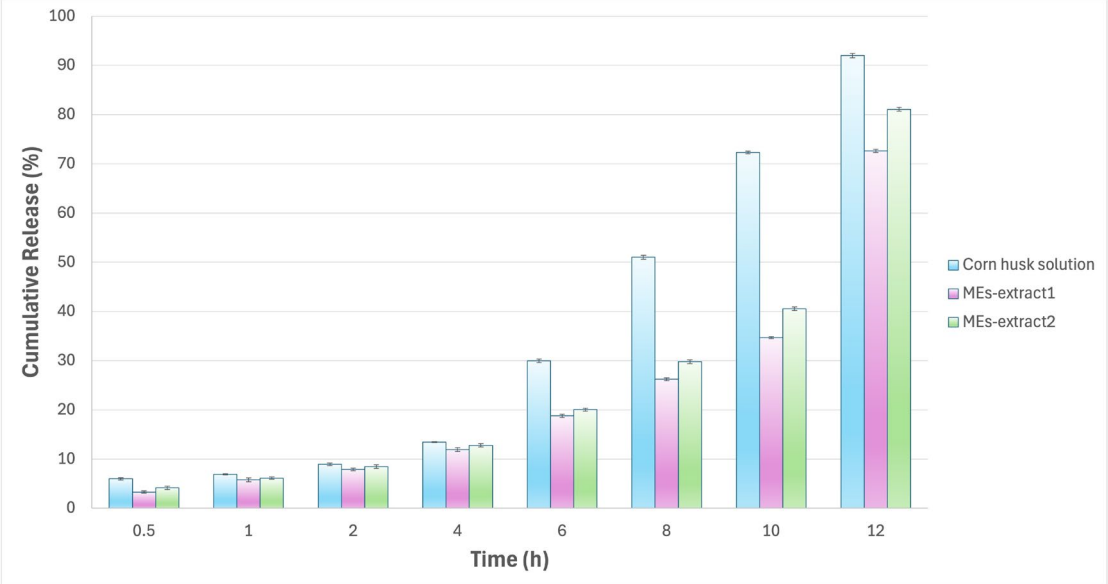


Figure 8. The cumulative release of phenolic compounds from corn husk solution, MEs-extract1, and MEs-extract2 over a period of 12 h.

3.5.5. The Rheology Study

The rheological behavior of the developed multiple emulsions was investigated by measuring the apparent viscosity as a function of shear rate ranging from 1 to 10 s^{-1}. As illustrated in Figure 9, all formulations exhibited a gradual decrease in viscosity with in-

creasing shear rate, demonstrating a typical non-Newtonian pseudoplastic (shear-thinning) flow behavior. This result indicated that flow resistance decreased as the applied shear rate increased. Similar shear-thinning behavior was reported for multiple-emulsion systems and considered a characteristic rheological response of structured emulsion systems. The observed decrease in apparent viscosity with increasing shear rate might be attributed to the rearrangement and alignment of dispersed droplets under applied shear. At low shear rates, droplets in the multiple-emulsion system might maintain a more structured arrangement, resulting in greater resistance to flow and, consequently, higher apparent viscosity. As the shear rate increased, the dispersed droplets progressively rearranged and oriented in the direction of flow, leading to a reduction in internal resistance and, consequently, lower apparent viscosity. The rheological behavior of multiple emulsions could also be influenced by several formulation-related factors, including the concentration of the dispersed phase, droplet size, droplet distribution, and emulsifier composition, which might affect the internal structure and flow characteristics of the system [41]. The shear-thinning behavior observed in this study might be advantageous for topical and cosmetic formulations. Under low-shear conditions, such as during storage, the relatively higher viscosity might contribute to maintaining the physical structure of the formulation, whereas the reduction in viscosity under applied shear might facilitate spreading during application to the skin. Similar rheological characteristics were considered desirable in pharmaceutical and cosmetic formulations intended for topical application because they can contribute to both formulation stability and ease of application [42]. However, this study evaluated the rheological behavior only within a shear-rate range of 1–10 s^{-1}. Therefore, additional parameters, such as the flow behavior index, yield stress, and thixotropic recovery, would be required to provide a more comprehensive understanding of the flow and structural properties of the developed multiple emulsions.

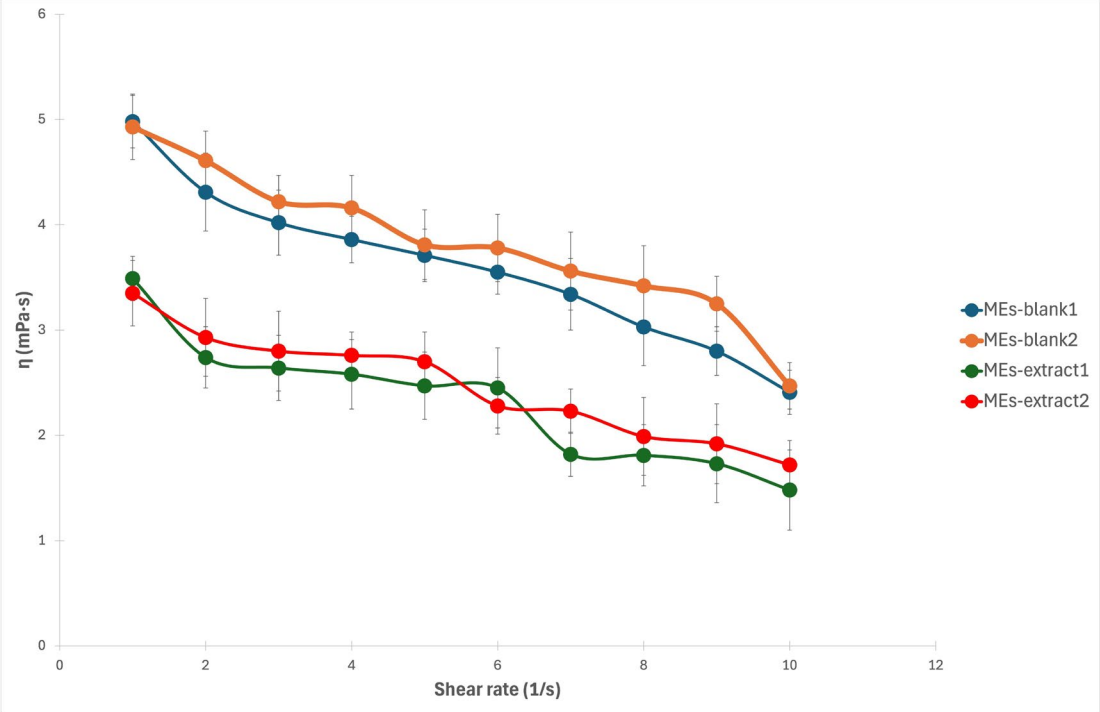


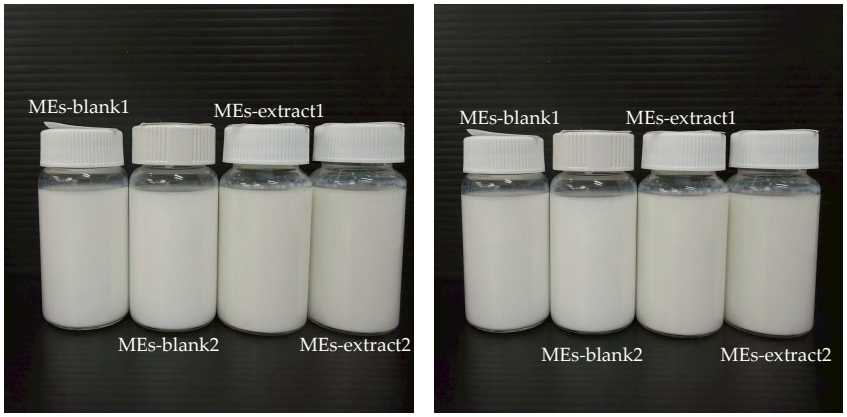
Figure 9. Rheology study of multiple emulsions blank and multiple emulsions containing corn husk extract.

3.5.6. Stability Test of Multiple Emulsions Containing Corn Husk Extract

The stability of multilayer emulsions was investigated at different temperatures. The multiple emulsion and the multiple emulsion containing corn husk extract before testing, as

shown in Figure 10a, were cloudy white and showed no separation. After stability testing, as observed in Figure 10b, no change was observed; i.e., they remained cloudy white, with no precipitation or separation. In Table 11, the physical characteristics of the multiple emulsions were evaluated by examining droplet size, PDI values, and zeta potential during 30 days of storage at 4, 25, and 45 °C. No significant changes in these parameters were observed at 24 and 48 h or after 7 and 15 days of storage. The droplet sizes of ME-blank1, ME-blank2, ME-extract1, and ME-extract2 were 161.50 ± 0.70, 163.13 ± 0.35, 169.93 ± 0.32, and 170.50 ± 0.66 nm, respectively, at 25 °C. After 30 days of storage, ME-blank1 showed only a slight change in droplet size. Similarly, ME-extract1 also had a constant droplet size at 25 and 45 °C. Only the formulation containing ME-extract1 exhibited an increase in droplet size, which rose from 170.00 ± 0.26 nm at 4 °C. In contrast, droplet sizes in ME-blank2 and ME-extract2 at 25 and 45 °C increased significantly. The droplet sizes of MEs-blank2 and MEs-extract2 increased to 169.30 ± 0.40 and 172.30 ± 0.40 nm and to 173.13 ± 0.21 and 181.77 ± 0.42 nm, respectively. These increases in mean droplet size might indicate limited droplet growth during storage, potentially resulting from processes such as droplet coalescence or diffusion-driven growth. In multiple emulsions, instability could occur through several pathways, including coalescence of internal aqueous droplets, coalescence of the outer droplets, and molecular diffusion between phases. Such processes could lead to an increase in the measured average droplet size even when no visible phase separation occurred [37,43]. Therefore, the observed increase in droplet size should be interpreted as evidence of some degree of physical change rather than complete destabilization of the formulations. All formulations showed an initial PDI of approximately 0.22. After 30 days of storage, the PDI remained in a narrow range of 0.22 to 0.24 at all storage temperatures. The consistently low PDI values indicated that the emulsion retained a relatively narrow droplet size distribution, although a slight increase in the average droplet size was observed in some formulations. Importantly, the absence of a significant increase in PDI indicated that droplet growth was not accompanied by the formation of large clumps or a noticeable expansion of the droplet-size distribution range. This finding was important because changes in average droplet size and changes in the breadth of the droplet-size distribution did not necessarily occur to the same extent. A stable PDI together with the absence of visible phase separation suggested that the formulations maintained a relatively homogeneous droplet population during storage, although the observed increase in droplet size indicated that some degree of droplet growth might have occurred. Previous studies have emphasized that droplet size distribution and PDI were useful complementary parameters for monitoring the physical stability of multiple emulsions because destabilization might occur before macroscopic phase separation [37,43]. Zeta potential measurements provided a better understanding of emulsion-interface stability. At 25 °C, the initial zeta potentials of ME-blank1 and ME-blank2 were −89.10 ± 0.20 and −89.27 ± 0.15 mV, respectively, while the initial zeta potentials of ME-extract1 and ME-extract2 were −81.07 ± 0.25 and −81.13 ± 0.21 mV, respectively. After 30 days, all formulations maintained relatively high negative zeta potentials. The zeta potentials of ME-blank1 and ME-blank2 changed significantly from the initial time. ME-extract1 had a constant zeta potential at all temperatures, while ME-extract2 also had a constant value at 4 and 25 °C. Only the formulation containing ME-extract2 exhibited a significant change in zeta potential to −71.53 ± 0.35 at 4 °C. Total phenolic content was also tested, with results shown in Table 12. The initial TPC after 24 h of preparation at 25 °C was 255.82 ± 0.48 mg GAE/L for ME-extract1 and 255.61 ± 0.65 mg GAE/L for ME-extract2. After 30 days, TPC decreased significantly in both formulations at all storage temperatures. At 4 and 25 °C, the TPC values remained relatively higher, at approximately 188 mg GAE/L, whereas substantially greater losses were observed at 45 °C, with TPC decreasing to 125.93 ± 0.48 and 121.97 ± 0.31 mg GAE/L for ME-extract1

and ME-extract2, respectively. Based on the initial values, this temperature-dependent decrease indicated that elevated temperature markedly accelerated the loss of measurable phenolic compounds. The pronounced reduction in TPC at 45 °C was consistent with the known sensitivity of plant phenolic compounds to environmental and storage conditions. Temperature can accelerate oxidation, hydrolysis, and other degradation reactions involving phenolic compounds, resulting in a reduction in the concentration of detectable phenolics over time. Previous reviews have also reported that temperature, oxygen, and light were important factors influencing the stability of polyphenols during storage, with higher temperatures generally promoting degradation [44]. In encapsulated systems, the carrier matrix could provide a degree of protection against environmental stresses; however, encapsulation did not completely prevent chemical degradation, particularly under elevated-temperature conditions. Therefore, the substantial decrease in TPC at 45 °C observed in this study suggested that the multiple-emulsion structure provided only partial protection against thermal degradation of the phenolic constituents.



(**a**) (**b**)

Figure 10. MEs = multiple emulsions. The external appearance of multiple emulsions blank and multiple emulsions containing corn husk extract (**a**) before the stability test and (**b**) after the stability test for 30 days.

Table 11. The droplet size, PDI, and zeta potential of emulsions before and after the stability test for 30 days.

Sample	Temperature (°C)	Droplet Size (nm)		PDI		Zeta Potential (mV)	
		24 h	30 Days	24 h	30 Days	24 h	30 Days
MEs-blank1	4	161.50 ± 0.70	162.27 ± 0.32	0.22 ± 0.01	0.22 ± 0.01	−89.10 ± 0.20	−83.67 ± 0.95 *
	25		162.40 ± 0.50		0.22 ± 0.02		−84.97 ± 0.51 *
	45		163.30 ± 0.20		0.23 ± 0.01		−83.33 ± 1.14 *
MEs-blank2	4	163.13 ± 0.35	171.07 ± 0.42 *	0.22 ± 0.03	0.23 ± 0.01	−89.27 ± 0.15	−82.23 ± 0.31 *
	25		169.30 ± 0.40 *		0.22 ± 0.01		−84.97 ± 0.51 *
	45		172.30 ± 0.40 *		0.23 ± 0.01		−74.67 ± 0.83 *
MEs-extract1	4	169.93 ± 0.32	170.00 ± 0.26 *	0.22 ± 0.01	0.23 ± 0.02	−81.07 ± 0.25	−80.98 ± 0.51
	25		169.93 ± 0.32		0.22 ± 0.01		−80.93 ± 0.15
	45		172.27 ± 0.47 *		0.23 ± 0.01		−79.53 ± 0.49
MEs-extract2	4	170.50 ± 0.66	179.07 ± 0.15 *	0.22 ± 0.01	0.23 ± 0.01	−81.13 ± 0.21	−79.83 ± 0.25
	25		173.13 ± 0.21 *		0.22 ± 0.01		−80.63 ± 0.49
	45		181.77 ± 0.42 *		0.24 ± 0.01		−71.53 ± 0.35 *

Note: MEs = multiple emulsions. The data were presented as mean ± SD (n = 3). The asterisk (*) indicates a significant difference compared with their formulation after being kept for 24 h. Statistical significance at $p < 0.05$, using a paired-sample t-test.

Table 12. The total phenolic content of multiple emulsions before and after the stability test for 30 days.

Sample	Temperature (°C)	TPC (mg GA/L)	
		24 h	30 Days
MEs-extract1	4	255.82 ± 0.48	188.11 ± 0.36 *
	25		188.74 ± 0.48 *
	45		125.93 ± 0.48 *
MEs-extract2	4	255.61 ± 0.65	188.01 ± 0.36 *
	25		188.32 ± 0.36 *
	45		121.97 ± 0.31 *

Note: MEs = Multiple emulsions. The data are presented as mean ± SD (n = 3). The asterisk (*) indicates a significant difference compared with their formulation after being kept for 24 h. Statistical significance at $p < 0.05$, using a paired-sample t-test.

4. Conclusions

This study successfully demonstrated the valorization of agricultural corn husk waste through ultrasound-assisted extraction, yielding an extract of 2.43% rich in natural bioactive compounds. The resulting corn husk extract exhibited potent antioxidant capacity, with a FRAP value of 1.62 ± 0.01 mM Fe^{2+}/mg and an ABTS radical scavenging activity of 99.24%, demonstrating performance comparable to ascorbic acid. The corn husk extract was successfully incorporated into the W/O nanoemulsion system. NEs-extract20 showed an exceptional encapsulation efficiency of 94.20 ± 0.50% with a droplet size of 21.63 ± 1.04 nm and a PDI value of 0.31 ± 0.02. For W/O/W multiple emulsions, MEs-extract1 produced a droplet size of 169.97 ± 0.31 nm, a PDI value of 0.22 ± 0.01, and a zeta potential of −81.17 ± 0.25 mV. Rheological analysis confirmed non-Newtonian pseudoplastic (shear-thinning) behavior, which may facilitate spreadability and contribute to favorable application properties for topical skin applications. Furthermore, in vitro release kinetics verified that the multiple-emulsion barrier efficiently sustained release of the active total phenolic content, with 72% cumulative release over 12 h, compared with the rapid diffusion of the unencapsulated extract of 92%. Stability studies confirmed that MEs-extract1 maintained its physicochemical integrity without visible phase separation, although slight changes in droplet size and total phenolic content were observed. In conclusion, the developed W/O/W multiple emulsion system represents a promising carrier platform for transforming agricultural residues into high-value bioactive ingredients for a potential delivery system for cosmetic applications.

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Abbreviations

The following abbreviations are used in this manuscript:

ROS	Reactive oxygen species
NEs	Nanoemulsions
F	Formulation
h	Hour
DI water	Deionized water
FRAP	Ferric reducing antioxidant power
PDI	Polydispersity index
EE	Encapsulation efficiency
TPC	Total phenolic content
MEs	Multiple emulsions
TEM	Transmission electron microscope
PBS	Phosphate-buffered saline

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