

Carbopol-Based Clove Oil (*Syzygium aromaticum*) Emulgel: Formulation, Physical Characterization, and Thermodynamic Stability

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ABSTRACT

Clove oil from *Syzygium aromaticum* contains eugenol with well-documented anti-inflammatory potential, but its use as a topical liquid is limited by volatility, low physical stability, and short skin contact time. This study aimed to formulate and characterize Carbopol-based clove oil emulgels as candidate topical delivery systems. Emulgels containing 10% (w/w) clove oil were prepared with Carbopol 940 at 1%, 2%, and 3% (w/w) and evaluated for organoleptic properties, spreadability, adhesion, pH, and thermodynamic stability using freeze–thaw cycling and centrifugation. All formulations showed acceptable appearance and homogeneity, with pH values of approximately 5.3–5.5, within the physiological skin range. Increasing Carbopol concentration decreased spreadability but significantly increased adhesion, reflecting higher viscosity and a denser gel network. After six freeze–thaw cycles, the 3% Carbopol formulation (F3) showed the smallest change in spreadability and adhesion and no evidence of phase separation, while maintaining skin-compatible pH. These findings indicate that the 3% Carbopol emulgel provides the most favourable balance between handling properties and thermodynamic stability and represents the most promising formulation among those tested for further development as a topical clove oil delivery system.



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Keywords:

Clove oil; *Syzygium aromaticum*; Emulgel; Carbopol 940; Topical drug delivery; Physical stability

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1. Introduction

Clove (*Syzygium aromaticum*) has long been used in food, cosmetic, and traditional medicinal preparations. Its essential oil contains a complex mixture of hydrocarbons, monoterpenes, phenolic compounds, and sesquiterpenes, with eugenol (4-allyl-2-methoxyphenol) as the predominant constituent, accompanied mainly by eugenyl acetate and β -caryophyllene [1]. These constituents contribute to antimicrobial,

antioxidant, and anti-inflammatory activities, which underlie the broad therapeutic potential of clove oil in various pharmaceutical and cosmetic products [2],[3].

Inflammation is a local protective response to tissue injury or infection that aims to remove harmful stimuli and initiate repair. Eugenol has been reported to exert anti-inflammatory effects through inhibition of prostaglandin synthesis and neutrophil chemotaxis, as well as modulation of other inflammatory mediators [4]. Experimental studies have shown that clove oil or eugenol-rich fractions can reduce inflammatory responses in animal models, supporting their potential role as topical anti-inflammatory agents [4],[5].

Despite these benefits, the direct topical use of clove oil in its liquid form is limited by volatility, poor physical stability, and a relatively short residence time on the skin, which may result in suboptimal local exposure and possible irritation [4],[5]. These drawbacks highlight the need for a more suitable semisolid delivery system that can improve stability, enhance retention at the application site, and provide better patient acceptability. Emulgel systems, which combine the structural advantages of gels with the solubilization capacity of emulsions, have been increasingly explored as vehicles for hydrophobic drugs and essential oils [6],[7],[8]. By incorporating the oil phase as droplets dispersed in a gelled aqueous phase, emulgels can improve spreadability, ease of application, and physical stability compared with conventional ointments or creams [6],[9].

In an emulgel, the gel network is commonly formed by polymers such as Carbopol 940, a cross-linked polyacrylic acid that produces clear, thixotropic gels upon neutralization with triethanolamine. The concentration of Carbopol strongly influences the rheological behaviour, spreadability, adhesion, and overall stability of the formulation [6],[9]. However, systematic data on clove oil emulgels based on Carbopol 940, particularly regarding the impact of Carbopol concentration on physical properties and thermodynamic stability, are still limited.

Therefore, this study aimed to develop clove oil emulgels using Carbopol 940 at concentrations of 1%, 2%, and 3% (w/w) and to evaluate their physical characteristics and thermodynamic stability. The objective was to identify a formulation that provides an optimal balance between spreadability, adhesion, skin-compatible pH, and stability, thereby serving as a promising candidate for further development as a topical clove oil delivery system.

2. Methods

Tools and Materials

The equipment used in this study included a set of standard glassware, porcelain dishes, a mortar and pestle, a UV-Vis spectrophotometer, a gas chromatography-mass spectrometry (GC-MS) system, a stopwatch, a thermometer, a pH meter, a water bath, and a mechanical stirrer.

The materials used were clove oil, Carbopol 940, triethanolamine (TEA), liquid paraffin, Tween 80, Span 80, propylene glycol, methyl paraben, propyl paraben, 96% ethanol, and distilled water.

Work Procedure

Formulation

The qualitative and quantitative composition of the clove oil emulgels is presented in Table 1. Three formulations (F1-F3) were prepared with varying Carbopol 940 concentrations (1%, 2%, and 3% w/w), while the clove oil content was kept constant at 10% w/w.

Table 1. Clove oil emulgel formulation

Formula component	Function	F1	F2	F3
Clove oil	Active ingredient	10%	10%	10%
Carbopol 940	Gel base / gelling agent	1%	2%	3%
TEA	Neutralizing agent / pH adjuster	0.1%	0.1%	0.1%
Tween 80	Emulsifier (hydrophilic)	1%	1%	1%
Span 80	Emulsifier (lipophilic)	1.5%	1.5%	1.5%
Propylene glycol	Humectant / co-solvent	5%	5%	5%
Methyl paraben	Preservative	0.01%	0.01%	0.01%
Propyl paraben	Preservative	0.03%	0.03%	0.03%
Distilled water	Vehicle / solvent	ad 100	ad 100	ad 100
		g	g	g

Gel base preparation

The gel base was prepared separately by gradually dispersing Carbopol 940 into hot distilled water (70 ± 2 °C) under continuous stirring at 800 rpm for 20 minutes until fully hydrated. Once a uniform dispersion was obtained, methyl paraben and propyl paraben were added and mixed for an additional 10 minutes until completely dissolved. Triethanolamine was then added dropwise under gentle stirring to neutralize the dispersion and adjust the pH to approximately 6.0–6.5, resulting in a clear and homogeneous gel base [7].

Emulsion preparation

The emulsion was prepared by heating the oil and aqueous phases to 70 °C. The oil phase consisted of clove oil and Span 80, while the aqueous phase was prepared separately by mixing Tween 80, propylene glycol, and the required amount of distilled water until homogeneous. The aqueous phase was added gradually to the oil phase under continuous stirring at 200 rpm for 20 minutes until a uniform oil-in-water emulsion was formed [8].

Emulgel preparation

The emulgel was obtained by gradually incorporating the emulsion into the prepared Carbopol gel base while stirring at 300 rpm for 15–20 minutes until a homogeneous emulgel mass was formed. The resulting formulations were then subjected to characterization and thermodynamic stability testing [9].

Characterization Tests

Organoleptic evaluation

Organoleptic evaluation was carried out to ensure that the emulgel met aesthetic and acceptability criteria. The main parameters assessed were colour, odour, texture, and homogeneity. Colour was evaluated visually under normal lighting. Odour was assessed to confirm the characteristic clove scent and the absence of unpleasant odours. Texture was examined by gently rubbing a small amount of emulgel between the fingertips to evaluate smoothness and the absence of coarse particles. Homogeneity was assessed by visual inspection of the emulgel mass and a thin layer of the preparation spread on a glass plate [10].

Spreadability test

Spreadability was determined by weighing 1.0 g of emulgel and placing it at the centre of a glass plate. The sample was covered with a second glass plate and a 125 g weight was placed on top for 1 minute. The resulting diameter of the spread emulgel

was measured in at least two perpendicular directions using a ruler, and the mean value was recorded as the spreadability (cm) [11].

Adhesion test

For the adhesion test, 0.5 g of emulgel was placed on a glass slide marked with an area of 4 × 2 cm and spread evenly to cover the marked area. A second glass slide was placed on top of the preparation. The two slides were pressed together using a 1 kg weight for 5 minutes. The adhered glass slides were then mounted on an adhesion test apparatus with an attached load of 80 g. The time required for the two glass slides to separate was recorded as the adhesion time (s) and used as a measure of the adhesion strength of the emulgel [12].

pH test

The pH of the emulgel was measured by dispersing 1.0 g of sample in 9.0 g of purified water at 25 °C. The dispersion was stirred until homogeneous and allowed to stand for 30 minutes to equilibrate. The pH meter was calibrated using standard buffer solutions (pH 4.0 and 7.0) before measurement. The electrode was then immersed in the dispersion, and the pH value was recorded once the reading stabilized. Measurements were performed in triplicate for each formulation, and the results were expressed as mean ± standard deviation (SD) [13],[14].

Thermodynamic Stability Test

Thermodynamic stability was evaluated using a freeze-thaw cycling test followed by centrifugation. For the cycling test, samples of each formulation (F1, F2, and F3) were stored at 4 °C for 24 hours and then transferred to 40 °C for 24 hours. This two-step cycle was repeated six times. After completion of the cycles, the samples were examined for any signs of phase separation, creaming, colour change, or the presence of coarse particles.

Following the freeze-thaw cycling, the formulations were subjected to centrifugation at 3000 rpm for 30 minutes. The samples were inspected visually after centrifugation to confirm the absence of phase separation or other signs of physical instability.

Data Analysis

All quantitative data obtained from the evaluation tests, including spreadability, adhesion, and pH, were expressed as mean ± SD from triplicate measurements (n = 3). Statistical analysis was performed using IBM SPSS Statistics software (version 26). A one-way analysis of variance (ANOVA) was applied to determine significant differences among formulations, followed by Tukey's post hoc test for pairwise comparisons. A p-value of less than 0.05 (p < 0.05) was considered statistically significant.

3. Results and Discussion

Organoleptic Testing

Organoleptic testing

Organoleptic evaluation is an important parameter for assessing the quality and acceptability of emulgel preparations, including observations of colour, odour, texture, and homogeneity, which are closely related to aesthetics, user preferences, and early indications of physical stability [2]. All clove oil emulgel formulations (F1, F2, and F3) exhibited a white colour, a characteristic clove odour, smooth texture, and homogeneous appearance (**Figure 1, Table 2**).



Figure 1. Organoleptic Test of Clove Oil Emulgel

No phase separation or coarse particles were observed. These findings indicate that the emulsification and gelling processes were successful and that both excipients and the active ingredient were uniformly distributed within the system. The smooth texture and homogeneous appearance are in line with the ideal criteria for topical preparations and are expected to enhance patient comfort during application.

Table 2. Physical evaluation results

No	Test	F1	F2	F3
1	Organoleptic			
	Colour	White	White	White
	Odour	Characteristic of cloves	Characteristic of cloves	Characteristic of cloves
	Texture	Smooth	Smooth	Smooth
2	Homogeneity	Homogeneous	Homogeneous	Homogeneous
	Spreadability (cm)	6.50 ± 0.10	5.70 ± 0.06	5.50 ± 0.10
3	Adhesion (s)	0.82 ± 0.04	2.03 ± 0.05	2.43 ± 0.07
4	pH	5.50 ± 0.10	5.50 ± 0.15	5.50 ± 0.12

All formulations showed suitable organoleptic characteristics for topical use, suggesting that clove oil can be successfully incorporated into a Carbopol-based emulgel system without compromising visual and sensory properties.

Adhesion test

Adhesion reflects the ability of an emulgel to remain in contact with the skin surface for a sufficient duration, which is crucial for maximizing local drug availability and therapeutic effect. In this study, increasing Carbopol 940 concentration from 1% (F1) to 3% (F3) resulted in a marked increase in adhesion time from 0.82 ± 0.04 s to 2.43 ± 0.07 s (Figure 2). This trend can be attributed to the formation of a denser and more cohesive gel network at higher polymer concentrations, which enhances intermolecular interactions between the formulation and the skin surface [2],[12].

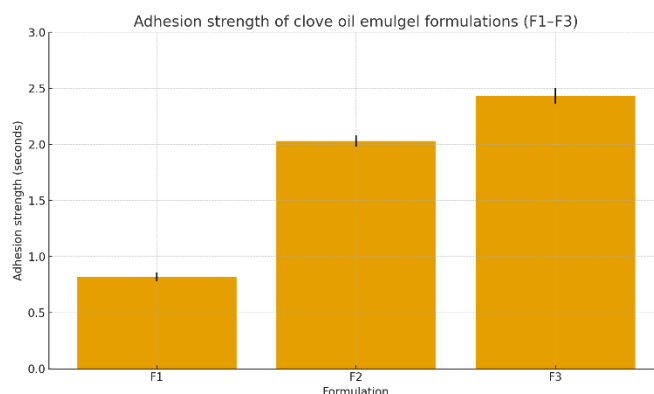


Figure 2. Adhesion strength of clove oil emulgel formulations F1–F3 before stability testing (mean ± SD, n = 3)

Formulations F2 and F3 demonstrated higher adhesion than F1 while still remaining within an acceptable range for topical use. Excessive adhesion may cause discomfort and stickiness, whereas low adhesion may lead to rapid removal of the formulation before sufficient drug release occurs. The adhesion values observed for F2 and especially F3 can therefore be considered favourable, providing adequate contact time without compromising user comfort [2],[12].

Spreadability test

Spreadability is a key parameter for topical semisolid preparations because it determines the ease of application and the ability of the product to form a uniform film on the skin surface. The spreadability values of the clove oil emulgels ranged from 5.50 ± 0.10 cm to 6.50 ± 0.10 cm (**Figure 3**), which fall within the commonly recommended range of 5–7 cm for acceptable topical preparations [15]. Formulation F1, containing 1% Carbopol 940, exhibited the highest spreadability (6.50 ± 0.10 cm), whereas F3, containing 3% Carbopol 940, showed the lowest spreadability (5.50 ± 0.10 cm), with F2 in between these two values.

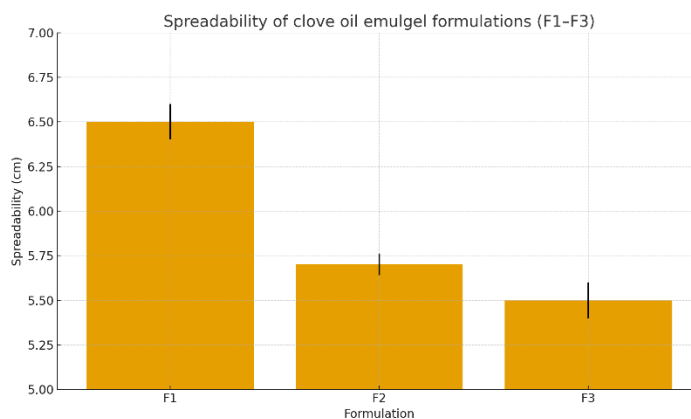


Figure 3. Spreadability of clove oil emulgel formulations F1–F3 before stability testing (mean ± SD, n = 3).

The decrease in spreadability with increasing Carbopol concentration reflects the rise in viscosity and rigidity of the gel network at higher polymer levels [6],[9],[15]. In practical terms, F1 is easier to spread but shows lower adhesion and stability, while F3, although slightly less spreadable, still lies within the desirable range and provides higher adhesion and better resistance to thermodynamic stress. These findings indicate that the 3% Carbopol emulgel offers a favourable compromise between adequate spreadability for patient convenience and sufficient consistency to maintain prolonged contact with the skin, supporting its selection as the most promising formulation for topical delivery of clove oil.

pH test

The pH of topical semisolid preparations must be compatible with the physiological pH of the skin, generally reported in the range of 4.5–6.5, in order to minimize the risk of irritation and preserve barrier function [13],[14],[16]. In the present study, the initial pH values of all clove oil emulgel formulations were approximately 5.5, namely 5.50 ± 0.10 for F1, 5.50 ± 0.15 for F2, and 5.50 ± 0.12 for F3 (**Figure 4**). These values lie within the physiological skin pH range, indicating good compatibility and suggesting that the formulations are unlikely to cause irritation under normal conditions of topical use. Maintaining the pH near 5.5 is particularly advantageous because it supports the integrity of the stratum corneum and the skin's natural antimicrobial defence.

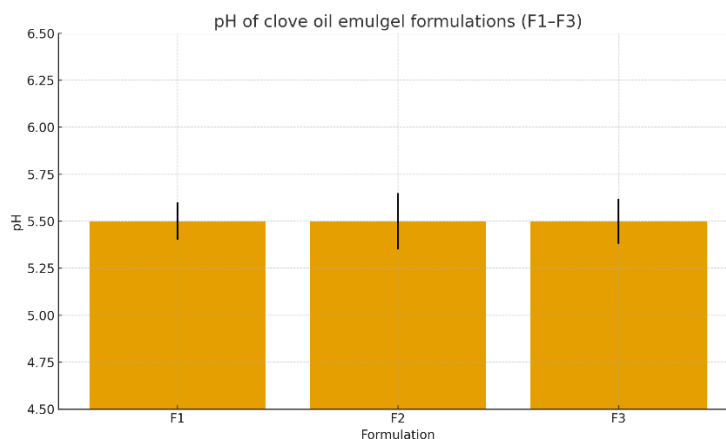


Figure 4. pH of clove oil emulgel formulations F1–F3 before stability testing (mean \pm SD, n = 3)

After the freeze-thaw stability test, the pH of all formulations showed a slight decrease to around 5.30 ± 0.10 – 0.15 (Table 4), but still remained within the acceptable physiological range. One-way ANOVA confirmed that there were no statistically significant differences in pH among the formulations ($p > 0.05$), either before or after cycling, indicating that variations in Carbopol concentration primarily affected the mechanical properties of the emulgel rather than its acid–base balance. Taken together, these findings demonstrate that the clove oil emulgels possess skin-compatible pH values and maintain satisfactory pH stability under thermodynamic stress, supporting their suitability for topical application.

Stability test (freeze-thaw cycling and centrifugation)

The freeze-thaw cycling test was conducted to evaluate the physical stability of the emulgels under temperature stress that mimics long-term storage conditions and accelerated handling during distribution [6],[9]. After six cycles between 4 °C and 40 °C, none of the formulations exhibited phase separation, creaming, discolouration, or formation of coarse particles, either before or after centrifugation at 3000 rpm for 30 minutes (Table 3). These macroscopic observations indicate good overall physical integrity and are consistent with previous reports on Carbopol-based emulgels, which generally show good physical stability when properly neutralized and emulsified [6],[9],[15].

However, quantitative analysis revealed formulation-dependent changes in spreadability and adhesion. The spreadability of F1 increased markedly from 6.50 ± 0.10 cm to 7.80 ± 0.10 cm, suggesting a reduction in viscosity and partial disruption of the gel structure during thermal cycling. In contrast, F2 and F3 showed smaller changes in spreadability (5.70 ± 0.06 to 6.40 cm and 5.50 ± 0.10 to 5.70 ± 0.10 cm, respectively), indicating greater structural robustness at higher Carbopol concentrations, in line with the role of crosslinked polyacrylic acid polymers in forming more cohesive gel networks at higher levels [6],[9],[15].

Table 3. Physical stability of clove oil emulgel formulations before and after cycling

Test		Before cycling			After cycling		
		F1	F2	F3	F1	F2	F3
Organoleptic	Colour	White	White	White	White	White	White
	Odour	Characteristic of cloves	Characteristic of cloves	Characteristic of cloves	Characteristic of cloves	Characteristic of cloves	Characteristic of cloves
	Texture Homogeneity	Smooth Homogeneous	Smooth Homogeneous	Smooth Homogeneous	Smooth Homogeneous	Smooth Homogeneous	Smooth Homogeneous
Spreadability (cm)		6.50 ± 0.10	5.70 ± 0.06	5.50 ± 0.10	7.80 ± 0.10	6.40	5.70 ± 0.10
Adhesion (s)		0.82 ± 0.04	2.03 ± 0.05	2.43 ± 0.07	0.32 ± 0.04	1.32 ± 0.05	2.26 ± 0.07
pH		5.50 ± 0.10	5.50 ± 0.15	5.50 ± 0.12	5.30 ± 0.10	5.30 ± 0.15	5.30 ± 0.12

Adhesion values decreased for all formulations after cycling, but F3 retained the highest adhesion (2.26 ± 0.07 s), followed by F2 (1.32 ± 0.05 s) and F1 (0.32 ± 0.04 s). Compared with their initial values, the adhesion of F1 decreased by more than 60%, whereas the decrease for F3 was relatively small, confirming that the denser Carbopol network in F3 confers improved resistance to mechanical weakening and helps maintain longer contact with the skin surface, even after exposure to temperature fluctuations [6],[15]. The pH values after cycling remained within the physiological range (5.30 ± 0.10 – 0.15) for all formulations, with no significant differences among them ($p > 0.05$), supporting their chemical stability and safety for topical use [13],[14],[16]. Overall, F2 and especially F3 exhibited more stable spreadability and adhesion profiles after the stability test, indicating better thermodynamic stability than F1

Statistical analysis

One-way ANOVA followed by Tukey's post hoc test showed that variations in Carbopol concentration had a statistically significant effect on spreadability and adhesion ($p < 0.05$), whereas pH did not differ significantly among formulations ($p > 0.05$). These findings quantitatively confirm that Carbopol 940 concentration governs the mechanical behaviour of the emulgel without substantially affecting its pH (Table 4).

Table 4. One-way ANOVA results for spreadability, adhesion, and pH

Parameter	Sum of Squares	df	Mean Square	F	Sig. (p)
Spreadability (cm)	5.32	2	2.66	237.2	0.000*
Adhesion (s)	6.51	2	3.26	452.1	0.000*
pH	0.00	2	0.00	0.021	0.979 (ns)

Note: *Significant at $p < 0.05$; ns = not significant

Formulations containing 2% and 3% Carbopol (F2 and F3) exhibited significantly higher adhesion and lower spreadability than F1, which is consistent with their higher viscosity and denser gel structure. Among them, F3 provided the most favourable combination of spreadability, adhesion, and thermodynamic stability, supporting its selection as the optimized formulation for topical clove oil delivery.

Taken together, the physical evaluation, stability testing, and statistical analysis demonstrate that Carbopol 940 concentration is a critical determinant of the overall performance of clove oil emulgels. Increasing the polymer content from 1% to 3% (w/w) significantly reduced spreadability and enhanced adhesion, while maintaining a skin-compatible pH. These trends are in line with previous reports on Carbopol-based emulgels, where higher polymer levels increase the viscosity and cohesiveness of the gel network, thereby improving residence time on the skin but slightly reducing ease of spreading [6],[9],[15]. In the present study, all formulations remained within the recommended spreadability range and showed acceptable adhesion values, indicating that the selected concentration window (1–3% Carbopol 940) is pharmaceutically relevant for topical delivery systems.

Among the three formulations, the emulgel containing 3% Carbopol 940 (F3) offered the most favourable overall profile. It combined adequate spreadability with the highest adhesion and the most stable response to thermodynamic stress, as evidenced by minimal changes in spreadability and adhesion after freeze–thaw cycling. From a formulation perspective, these characteristics suggest that F3 is better able to maintain its structural integrity and interfacial contact with the skin during storage and use, which is desirable for maximizing the local availability of clove oil at the site of application. Although the present work did not evaluate rheology or drug release, the observed trends strongly support the selection of F3 as the base formulation for subsequent pharmacodynamic and permeation studies.

Although the F3 formulation demonstrated the best overall physical and stability profile, this study has several limitations. Rheological properties (flow curves, yield stress), microscopic droplet size distribution, and in vitro or ex vivo release and permeation data were not evaluated. Furthermore, no biological assays were conducted to confirm the anti-inflammatory activity of the emulgel. Future research should therefore include comprehensive rheological characterization, microscopic analysis, and biological activity testing such as protein denaturation inhibition or nitric oxide suppression assays to substantiate the therapeutic potential of the optimized clove oil emulgel as a topical anti-inflammatory product.

4. Conclusion

Clove oil emulgel formulations containing 1–3% Carbopol 940 (w/w) showed acceptable organoleptic properties and homogeneous appearance, with pH values around 5.3–5.5 within the physiological skin range. Increasing Carbopol concentration reduced spreadability but enhanced adhesion and stability, and the 3% Carbopol emulgel (F3) provided the most favourable combination of these properties, indicating its suitability as an optimized base for topical clove oil delivery. Further studies on rheology, droplet size, in vitro release and permeation, as well as anti-inflammatory assays, are still required to confirm the therapeutic potential of this formulation.

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Conflicts of Interest:

The author declares that there are no conflicts of interest in this research.

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