Formulation and Evaluation of Clove Oil Effervescent Mouthwash

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***Abstract*—This study aims to formulate and evaluate an effervescent mouthwash containing clove oil as a natural antimicrobial agent. Clove oil is widely recognized for its antiseptic and analgesic properties, making it effective for oral care. Effervescent granules were prepared using sodium bicarbonate and organic acids to enhance dispersion and improve patient compliance. Three formulations (F1, F2, and F3) were developed and evaluated for pre formulation parameters such as flow properties, bulk density, and Carr's index. Post formulation studies included effervescence time, pH, drug content, foam formation, taste, and mouthfeel. Among the formulations, the optimized batch exhibited rapid effervescence, acceptable pH, and good stability. The results indicate that the prepared effervescent mouthwash is a promising alternative to conventional formulations, offering improved convenience and effectiveness in maintaining oral hygiene.**

***Index Terms*—Clove Oil, Effervescent Mouthwash, Antimicrobial Activity, Oral Hygiene, Formulation and Evaluation**

I. INTRODUCTION

1. Background

Maintaining oral hygiene is crucial for systemic health, given the mouth's role as a key microbial gateway. Inadequate care promotes issues like plaque buildup, gingivitis, periodontal disease, and bad breath. Mouth rinses complement brushing and flossing by lowering bacterial counts and supporting cleanliness. Traditional liquid rinses, though common, struggle with instability, contamination risks, oversized containers, and shorter stability. [1,2]

In contrast, effervescent formats have surged in popularity for their quick water dispersion, CO_2 generation, and even dispersion. They boost adherence, dosing precision, and sensory appeal. Such mouthwash tablets improve portability, shelf life, and on-demand prep, positioning them as superior to liquids. [3,4]

Formulation Table Table no. 1

Ingredient(g)	F1	F2	F3	F4
Clove oil	0.25	0.25	0.25	0.25
Light $MgCO_3$	0.55	0.65	0.75	0.60
Citric acid	1.40	1.35	1.25	1.30
Tartaric acid	1.40	1.35	1.25	1.30
$NaHCO_3$	3.10	3.30	3.50	3.40
Mannitol	3.00	2.90	2.80	2.85
Sodium saccharin	0.12	0.12	0.12	0.12
Talc	0.10	0.08	0.08	0.08
Total	10	10	10	10

2. Literature Study

Essential oils and botanicals show strong promise in oral products. Clove oil from Syzygium aromaticum, abundant in eugenol, delivers robust antibacterial, pain-relieving, and anti-inflammatory effects. Long used in dentistry for pain relief and infection control, it targets diverse oral microbes effectively. [5,6]

Peppermint oil (Mentha piperita) adds a cooling refresh and subtle antimicrobial action, while improving taste appeal in meds. Effervescent blends with herbs enhance dissolution, durability, and user satisfaction. Yet, integrating volatile oils like clove and peppermint into dry forms is tricky due to evaporation and liquidity. Adsorption onto agents like magnesium carbonate transforms them into stable, dispersible powders for consistent dosing. [3]

3. Research Gap

Clove oil's volatility, powder incompatibility, and sensitivity to conditions hinder its use in solid effervescent systems. Liquid rinses also face spoilage and handling drawbacks. Thus, innovative, stable formats are needed to harness clove oil's benefits without compromising efficacy or convenience. [7,8,3]

4. Study Aim and Goals

This work aims to develop and assess an effervescent mouthwash tablet loaded with clove oil via adsorption. Key goals: adsorb clove oil onto magnesium carbonate for solid integration, boost flavor with peppermint oil, and test metrics like fizz duration, pH, and powder flow for viability, potency, and user-friendliness. [4,7,9]

II. MATERIALS AND METHODOLOGY

A) Materials

Analytical-grade reagents were used throughout. Clove oil provided antimicrobial and analgesic effects as the key active. [10] Light magnesium carbonate acted as the adsorbent to solidify the volatile oil into a pourable powder. [11] Anhydrous citric acid and tartaric acid served as acid sources, paired with sodium bicarbonate for CO_2 generation. Mannitol functioned as a filler and mild sweetener for a better sensory profile, supplemented by sodium saccharin for intense sweetness. [12] Talc improved powder flow as a lubricant. All components were precisely measured and kept in sealed, dry storage before processing. [13,14]

Formulation Development (Formulations F1–F4)

Four trial formulations (F1–F4) were developed, adjusting effervescent pair ratios (citric/tartaric acids to sodium bicarbonate) and adsorbent levels, with each batch total 10 g for performance optimization. [7,8]

B) Methodology

The adsorption method was employed to incorporate volatile clove oil into a solid form using magnesium carbonate [11]. All hygroscopic ingredients were dried at 40–50°C, cooled in a desiccator, and passed through a sieve no. 60 to ensure uniform particle size.

Clove oil was gradually added to magnesium carbonate and triturated until a dry, free-flowing powder was obtained [15]. Acid components were mixed separately, and base components were blended independently. The adsorbed mixture was incorporated into the base blend, followed by geometric addition of the acid mixture to prevent premature reaction [15]. Finally, talc was added to improve flow properties, and the formulation was packed in airtight containers.

Evaluation Parameters (F1–F4)

1. Organoleptic Evaluation (F1–F4)

Organoleptic properties, including color, odor, taste, and overall appearance, were systematically evaluated for formulations F1–F4 both in their intact state and post-dissolution. This assessment ensures high patient acceptability and compliance, critical for oral effervescent dosage forms.

2. Effervescence Time

Accurately weighed powder samples (1–2 g) were introduced into 100 mL of purified water maintained at ambient temperature (25 ± 2°C). The duration from addition until complete cessation of effervescence (no visible bubbles) was precisely recorded using a digital stopwatch, providing a direct measure of reaction kinetics and disintegration efficiency.[15]

3. pH Determination

Post-effervescence, the resulting solution was equilibrated, and its pH was measured at 25°C using a calibrated digital pH meter (accuracy ±0.01). This evaluation confirms the formulation's compatibility with the oral mucosa, minimizing the irritation risks during buccal administration.

4. Micromeritic Properties

Flow characteristics were quantified via the fixed funnel method for the angle of repose

[Tan θ = h/r]

whereas (h = height, r = radius).

Bulk density and tapped density were determined using a 100 mL graduated cylinder with 100 taps, followed by Carr's compressibility index computation:

Carr's Index = [Tapped Density-Bulk Density/Tapped Density] ×100

These parameters assess powder flowability, uniformity, and tableting suitability. [13,16]

5. Eugenol Content (Drug Content)
Precisely 1 g of each formulation was dispersed in 100 mL of ethanol, sonicated for 30 min, filtered, and appropriately diluted. Absorbance was recorded at the eugenol
% Drug Content = (Measured Concentration /Label Claim) ×100
ensuring formulation potency and content uniformity. [10]

6. CO_2 Content
The volume of carbon dioxide generated during effervescence was quantified by the water displacement method: 1 g of powder was reacted in a closed setup over a graduated inverted cylinder filled with water, and the displaced volume was measured at STP, reflecting effervescent capacity and gas-generating efficiency.
% CO_2 = [Volume of CO_2 /Theoretical Volume] ×100 [1,2]

7. Dispersion/Solubility Test
Formulation samples (1 g) were added to 100 mL of water at 25°C, gently swirled, and observed for time to complete dispersion, solution clarity, and absence of gritty residue or precipitates. This confirms rapid solubility and grittiness-free mouthfeel, essential for patient-centric effervescent systems. [17,8]

8. Moisture Content (Loss on Drying)
Samples (2 g) were accurately weighed and dried in a hot air oven at 105°C to constant weight. Moisture content was computed as:
Moisture Content = {(Initial weight - Final weight)/Initial weight} × 100],
evaluating hygroscopicity and storage stability.

9. Stability Studies
Accelerated stability testing followed ICH Q1A(R2) guidelines: formulations were packaged in amber glass vials and stored at 40°C ± 2°C/75% ± 5% RH for 1 month. Pre- and post-storage samples were re-evaluated for all parameters (e.g., pH, drug content, effervescence time) to detect physicochemical changes, ensuring shelf-life predictability. [12]

III. RESULTS AND DISCUSSION

Four adsorption-based clove oil effervescent powders (F1–F4) were prepared successfully and subjected to comprehensive physicochemical testing. Key findings appear in Tables 2–5, with comparative analysis highlighting F3 as optimal.
1. Organoleptic Characteristics: Powders displayed uniform off-white color and free-flowing nature. Reconstituted solutions (10–15 mL water) were transparent, emitting a strong eugenol aroma. Taste balanced pungency with sweetness from mannitol/saccharin; F3 and F4 scored highest for acceptability, likely from refined excipient-active ratios.

2. Effervescence Time: Time varied from 125 ± 6 s (F1) to 95 ± 4 s (F3) [Table 4]. F3's rapid fizz reflects superior acid-base synergy (higher bicarbonate, balanced acids), accelerating CO_2 output for quick dispersion and user preference. F1's delay signals imbalance, slowing reaction kinetics. [19,20]

3. pH Profile: Values spanned 5.8 ± 0.2 to 6.2 ± 0.2, all oral-safe [Table 4]. F3's near-neutral 6.1 ± 0.1 minimizes enamel erosion risk, ideal for daily use.

4. Powder Flow Metrics:
1) Angle of Repose = [Tan θ = h/r]
was calculated (h = height, r = radius).
2) Carr's Index = [Tapped Density-Bulk Density/Tapped Density] ×100
3)Hausner Ratio = [Tapped Density / Bulk Density

5. Eugenol Assay & CO_2 Yield [Table 4]: All met 85–115% (F3: 99.1 ± 1.2%), confirming adsorption uniformity. F3's peak recovery and highest CO_2 (82 ± 3 mL/g) validate carrier/effervescent optimization. [19,24]

6. Solubility & Stability [Table 3]: Full dissolution (no residues/globules); stable foam; moisture <2% (LOD) across batches proved design robustness. (21)

7. Formulation tweaks—adsorbent dose and acid-base proportions—drove variances. F3 balanced these for fastest effervescence, top flow/drug content, and max CO_2, positioning it for scale-up. Adsorption mitigated

clove oil volatility, yielding stable, patient-friendly tablets rivaling liquids.

All values: mean ± SD (n=3).

Table 2. Micromeritic Properties

Angle of Repose (°)	32.5 ± 1.2	29.8 ± 1.1	26.5 ± 1.0	28.2 ± 1.0
Bulk Density (g/mL)	0.62 ± 0.03	0.65 ± 0.02	0.68 ± 0.02	0.66 ± 0.03
Tapped Density (g/mL)	0.73 ± 0.02	0.74 ± 0.02	0.73 ± 0.01	0.75 ± 0.02
Carr's Index (%)	15.2 ± 1.0	12.1 ± 0.9	7.0 ± 0.8	11.8 ± 0.9
Hausner Ratio	1.18 ± 0.02	1.14 ± 0.02	1.07 ± 0.02	1.14 ± 0.02

Table 3. Solubility, Foam, and Moisture

Batch	Time(s)	pH
F1	125 ± 6	5.8 ± 0.2
F2	110 ± 5	5.9 ± 0.1
F3	95 ± 4	6.1 ± 0.1
F4	102 ± 5	6.2 ± 0.2

Table 4. Effervescence Time and pH

Batch	Eugenol (% label)	CO_2 (mL/g)
F1	92.5 ± 1.5	70 ± 3
F2	95.8 ± 1.3	75 ± 2
F3	99.1 ± 1.2	82 ± 3
F4	97.2 ± 1.4	78 ± 2

Table 5. Eugenol Content and CO_2 Yield

Parameter	F1	F2	F3	F4
Dispersion	Comp lete	Comp lete	Comp lete	Comp lete
Residue/GLO BULES	None	None	None	None
Foam (cm, 1 min)	2.5 ± 0.2	2.8 ± 0.2	3.2 ± 0.2	2.9 ± 0.2
Moisture (LOD %)	1.4 ± 0.1	1.2 ± 0.1	1.1 ± 0.1	1.3 ± 0.1

IV. CONCLUSION

This research successfully developed and characterized a clove oil-loaded effervescent mouthwash powder via adsorption onto light magnesium carbonate, transforming volatile oil into a stable, flowable solid. The citric/tartaric acid–sodium bicarbonate system ensured swift aqueous dispersion, boosting portability and adherence over liquid formats.

Trial batches F1–F4 met pharmacopoeial standards across key metrics: effervescence time, pH, micromeritics, eugenol content, CO_2 yield, solubility, and moisture. Compositional tweaks drove performance differences.

F3 outperformed with rapid fizz(95 ± 4 s), neutral pH(6.1 ± 0.1), superior flow (Carr's index 7.0 ± 0.8%),near-ideal drug assay (99.1 ± 1.2%), and peak CO_2(82 ± 3 mL/g)—reflecting optimal excipient balance.

F3 emerges as the lead candidate for upscale and advanced testing. This solid effervescent platform offers stability, convenience, and efficacy advantages versus traditional rinses, warranting accelerated stability trials and in vivo validation for market readiness.

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