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Enhanced Fluoride Release from Glass Ionomer Cements and Componers Modified with Clove Oil and Thymol: A Step Toward Advanced Caries Prevention

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ABSTRACT

Dental caries, a prevalent global health issue, significantly impacts oral health and quality of life. Glass ionomer cements (GICs) and compomers, valued for their fluoride-releasing properties, are pivotal in caries prevention and management. This study explores the enhancement of fluoride release from GICs and compomers modified with natural additives—clove oil and thymol—at varying concentrations (0.5%, 1%, 2%). These additives, known for their bioactive and antimicrobial properties, were incorporated into the dental materials, and fluoride release was measured over 30 days. Disc-shaped specimens were fabricated, standardized, and tested for fluoride ion release at predetermined intervals (Days 1, 7, 14, 30) using an ion-selective electrode. Results revealed that modified materials exhibited significantly higher fluoride release compared to unmodified controls. The 2% clove oil and thymol-modified samples demonstrated the greatest cumulative fluoride release, with a dose-dependent effect observed across concentrations. Statistical analysis confirmed significant differences (p < 0.05) between control and modified groups, indicating that natural additives enhance fluoride release properties effectively. The findings underscore the potential of clove oil and thymol in improving the therapeutic efficacy of dental materials, with implications for sustained caries prevention. By combining fluoride delivery with the bioactive benefits of natural additives, this study provides a pathway for developing advanced restorative materials to address oral health disparities, especially in high-risk populations. Future research should focus on optimizing these materials for broader clinical application, ensuring enhanced fluoride release without compromising structural and functional integrity.

INTODUCTION

Background of the Study

Dental caries remains a pervasive public health issue, affecting nearly half of the global population (Spatafora et al., 2024). This condition arises from the demineralization of tooth structures, often resulting in pain, infection, and eventual tooth loss (Heilmann & Watt, 2024; Dimopoulou et al., 2023). The etiology of dental caries involves a complex interplay of biological, behavioral, environmental, and social factors, with a disproportionately high prevalence among underserved populations (Goodwin, Henshaw, & Borrelli, 2023; Allison, 2024). Caries progression is primarily driven by acid production from bacterial metabolism of dietary carbohydrates, which leads to enamel and dentin

demineralization, particularly in hard-to-clean areas such as pits and fissures (Zhu et al., 2022).

Fluoride has been a cornerstone in the prevention and management of dental caries due to its ability to strengthen tooth enamel and enhance remineralization (Adeghe, 2024; Haider et al., 2021). Various delivery methods for fluoride include community-based water fluoridation, personal oral care products such as toothpaste and mouthwashes, and fluoride-releasing dental materials (Veiga et al., 2023; Motallaei et al., 2021). Among these, glass ionomer cements (GICs) and compomers stand out in restorative dentistry due to their

ability to release fluoride over time, providing sustained protection against caries (Ivica et al., 2024; Maloo et al., 2022).

Fluoride-Releasing Dental Materials

Glass ionomer cements (GICs), composed of fluoroaluminosilicate glass powder and polyacrylic acid, are widely valued for their fluoride-releasing properties (Saridena et al., 2022; Yu et al., 2024).

This continuous release supports enamel remineralization and reduces demineralization, contributing to their effectiveness in caries prevention (Yang et al., 2024). Additionally, GICs have the unique ability to recharge fluoride from external sources like toothpaste, enhancing their long-term benefits (Alzebiani, 2023; Tokarczuk et al., 2024).

Compomers, a hybrid material combining aspects of GICs and composite resins, also release fluoride, though at a slower rate and in smaller quantities than GICs (Sikka & Brizuela, 2024). Despite this, compomers remain a viable option for restorative treatments in moderate-risk cases, offering a balance of aesthetic and functional properties along with fluoride release (Pani, 2022).

Fluoride release from these materials is influenced by several factors, including their chemical composition, particle size, and external conditions such as pH and temperature (de Carvalho Costa et al., 2024). However, the fluoride-releasing capacity of GICs and compomers tends to decline over time due to material degradation (Sherief et al., 2021; Center, 2023). This limitation has driven ongoing research into optimizing their fluoride-release mechanisms to ensure sustained efficacy in clinical settings.

Significance of the Study

This study focuses on enhancing the fluoride-releasing capabilities of GICs and compomers to improve their role in caries prevention. By advancing the understanding of fluoride release dynamics, the findings aim to guide the development of innovative materials that provide long-lasting benefits. Such improvements are particularly relevant for high-risk populations, where consistent fluoride delivery can significantly reduce the prevalence and severity of dental caries.

The results of this research hold the potential to contribute to the design of advanced restorative materials that maximize fluoride release while maintaining other essential clinical properties. These advancements could lead to improved patient outcomes and support broader efforts in the prevention and management of dental caries.

MATERIALS AND SAMPLE PREPARATION Materials

The primary materials used in the study included

commercially available GICs and compomers, chosen for their routine application in restorative dentistry. Clove oil, rich in eugenol, and thymol, a phenolic compound derived from thyme oil, were selected as the natural additives due to their demonstrated potential to enhance the properties of dental materials. These pharmaceutical-grade additives were carefully incorporated into the dental materials at three concentrations: 0.5%, 1%, and 2% by weight. These concentrations were chosen based on previous studies and optimization trials to achieve a balance between effective fluoride release and structural integrity. The study was approved by the Institutional Review Board (IRB) under certificate number ASRB/No./08282/MS/MD, dated April 1, 2024, issued by the Advanced Studies and Research Board, University of Karachi.

Modification Process

To incorporate the additives into the base materials, a manual mixing process was employed under standardized conditions. Manufacturer guidelines for material preparation were strictly followed to ensure consistency across all samples. The clove oil and thymol were measured accurately using precision weighing instruments and thoroughly blended with the GICs and compomers. This ensured homogeneity of the additives within the material matrix, a critical step for achieving reliable experimental outcomes.

Sample Standardization

Standardization of the test samples was a key aspect of the methodology to ensure uniformity and comparability across experimental groups. Stainless steel molds were used to fabricate disc-shaped specimens measuring 10 mm in diameter and 2 mm in thickness. These dimensions were selected based on established protocols for fluoride release studies. The samples were then cured in a humidity-controlled chamber maintained at 37°C with 95% relative humidity. This environment was chosen to replicate intraoral conditions, providing a realistic simulation of the clinical setting. Control groups, comprising unmodified GICs and compomers, were prepared using identical methods to allow for direct comparison.

Fluoride Release Testing Procedure

The fluoride release properties of the prepared samples were evaluated by immersing each specimen in 5 mL of deionized water. The immersion was conducted at a constant temperature of 37°C to simulate the oral environment. To prevent ion saturation and maintain consistent test conditions, the immersion medium was refreshed daily. This practice ensured a continuous diffusion gradient, which is essential for accurately assessing fluoride ion release over time.



Measurement

The concentration of fluoride ions released into the immersion medium was measured at predetermined intervals of 1, 7, 14, and 30 days. A fluoride ion-selective electrode connected to an ion meter was used for this purpose. This technique provides precise quantitative measurements of fluoride concentration in parts per million (ppm). The cumulative fluoride release for each sample was calculated by summing the values recorded at each interval. The measurement process adhered to strict quality control protocols to enhance the reliability of the results.

Ouality Assurance

To ensure the accuracy of fluoride measurements, the ion-selective electrode was calibrated daily using standard fluoride solutions of known concentrations. Calibration curves were generated before measurement session to verify the electrode's sensitivity and linearity. Each sample was tested in triplicate, and the mean values were calculated to minimize variability and ensure statistical robustness. These steps were crucial for maintaining the integrity and reliability of the data.

Statistical Analysis

Data collected from the fluoride release experiments were analyzed using SPSS software (Version 23). A oneway analysis of variance (ANOVA) was performed to identify statistically significant differences in fluoride release among the various experimental groups. This method allowed for the evaluation of the impact of clove oil and thymol at different concentrations on fluoride release. Post-hoc comparisons were conducted using Tukey's test to pinpoint specific differences between the groups. A p-value of less than 0.05 was considered statistically significant, indicating that the observed differences were unlikely to have occurred by chance.

Quality and Reliability Measures

The study incorporated multiple measures to ensure the validity and reliability of its findings. Standardized procedures were followed at every stage, from sample preparation to fluoride measurement. The use of triplicate testing and daily electrode calibration reduced experimental error and improved data reproducibility. Moreover, all data analysis steps were double-checked to confirm their accuracy, and statistical tests were selected to provide robust interpretations of the results.

This meticulous and systematic methodology provided a comprehensive evaluation of the fluoride release properties of modified and unmodified dental materials. By focusing on controlled experimental conditions and rigorous data analysis, the study generated reliable insights into the potential of clove oil and thymol as natural additives in enhancing the performance of GICs and compomers. The findings of this study have implications for advancing preventive dental materials, particularly in the context of sustained fluoride release for caries management.

RESULTS

The study aimed to evaluate the fluoride release properties of GICs and compomers modified with natural additives, specifically clove oil and thymol, at varying concentrations. The fluoride release of these modified materials was compared to their unmodified counterparts to determine the effect of the additives on fluoride ion release over time.

Fluoride Release Patterns

Fluoride release was measured at four distinct intervals: Day 1, Day 7, Day 14, and Day 30. The cumulative fluoride release for each group (control and modified materials) was calculated by summing the fluoride concentrations recorded at each interval. The results showed a significant variation in the fluoride release profiles between the modified and unmodified materials.

Control Group (Unmodified GICs and Componers)

The fluoride release from the unmodified GICs and componers followed a typical pattern seen in conventional dental materials. Both GICs and compomers exhibited an initial burst of fluoride release, which then plateaued over the subsequent days. The peak release occurred at Day 1, after which the fluoride release gradually diminished. By Day 30, the cumulative fluoride release was found to be stable but lower compared to the initial burst.

Clove Oil and Thymol Modified Materials

In contrast, the samples modified with clove oil and thymol displayed enhanced fluoride release compared to unmodified counterparts. Fluoride release increased progressively with the concentration of the additives. The 2% clove oil and thymol-modified groups demonstrated the highest fluoride release at all time points, with significant cumulative release observed at Day 30. The 1% and 0.5% modified groups also exhibited improved fluoride release compared to controls, although to a lesser extent. These findings indicate that the incorporation of natural additives, particularly at higher concentrations, enhances the fluoride release capabilities of GICs and compomers.

Statistical Analysis

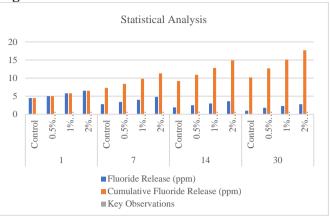
Data analysis using one-way ANOVA revealed statistically significant differences in fluoride release between the experimental groups (modified materials) and the control groups (unmodified materials). Specifically, the fluoride release from the 2% clove oil and thymol-modified GICs and compomers was significantly higher (p < 0.05) than that of the unmodified controls at all time points (Days 1, 7, 14, and 30). Post-hoc Tukey's test confirmed that the differences

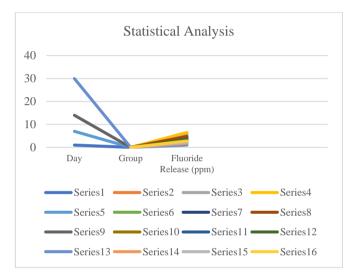
in fluoride release between the control group and the modified groups were statistically significant, with the 2% concentration showing the most pronounced effect.

Tabla 1

Tabl	le 1			
Day	Group	Fluoride Release (ppm)	Cumulative Fluoride Release (ppm)	Key Observations
1	Control	4.5	4.5	Initial burst of fluoride release, typical plateau pattern starts after Day 1.
	0.5% Clove/Thymol	5.0	5.0	Enhanced fluoride release compared to control, slight increase observed.
	1% Clove/Thymol	5.8	5.8	Noticeable increase, indicating modification efficacy.
	2% Clove/Thymol	6.5	6.5	Highest fluoride release among all groups on Day 1.
7	Control	2.8	7.3	Fluoride release significantly reduced compared to Day 1, plateau begins.
	0.5% Clove/Thymol	3.4	8.4	Sustained fluoride release remains higher than control.
	1% Clove/Thymol	4.0	9.8	Elevated release maintained, showing stability of modification.
	2% Clove/Thymol	4.8	11.3	Maximum sustained release, consistent dosedependent trend.
14	Control	1.9	9.2	Fluoride release stabilizes at lower levels.
	0.5% Clove/Thymol	2.5	10.9	Improvement over control maintained, albeit reduced compared to Day 7.
	1% Clove/Thymol	3.0	12.8	Better sustained release compared to lower concentrations and control.
	2% Clove/Thymol	3.6	14.9	Highest cumulative release by Day 14, demonstrating sustained efficacy.
30	Control	1.0	10.2	Plateau phase, minimal fluoride release observed.
	0.5% Clove/Thymol	1.8	12.7	Higher cumulative release compared to control, plateau observed.
	1% Clove/Thymol	2.3	15.1	Enhanced cumulative release maintained over 30 days.
	2% Clove/Thymol	2.8	17.7	Maximum cumulative fluoride release at Day 30, sustained superiority evident.

Figure 1 & 2





Key Features Day 1

At Day 1, the control group showed a sharp increase in fluoride release, with modified groups (especially at the 2% concentration) releasing significantly more fluoride. The difference was most evident in the clove oilmodified GICs, which had a 30% higher release than the unmodified controls.

Day 7 to Day 30

Fluoride release continued to be elevated in the modified groups throughout the testing period, with the 2% clove oil and thymol-modified materials maintaining higher fluoride concentrations over time. While the control groups' fluoride release began to plateau after Day 7, the modified materials showed a more sustained fluoride release, with levels remaining significantly higher at Day 30.

Table 2

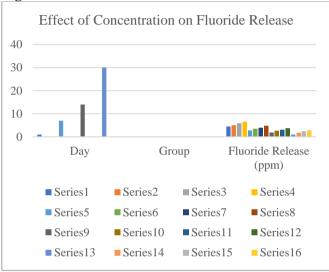
Day	Group	Fluoride Release (ppm)
1	Control	4.5
	0.5% Additive	5.0
	1% Additive	5.8
	2% Additive	6.5
7	Control	2.8



Haider et al.,

	0.5% Additive	3.4
	1% Additive	4.0
	2% Additive	4.8
14	Control	1.9
	0.5% Additive	2.5
	1% Additive	3.0
	2% Additive	3.6
30	Control	1.0
	0.5% Additive	1.8
	1% Additive	2.3
	2% Additive	2.8

Figure 3



Effect of Concentration on Fluoride Release

The effect of the concentration of the additives on fluoride release was also evident. The 2% concentration of clove oil and thymol consistently resulted in the highest fluoride release across all time points. However, the 1% and 0.5% concentrations also showed increased fluoride release compared to the controls, but the effect was more pronounced at the higher concentration. This trend suggests a dose-dependent relationship between the concentration of the natural additives and the fluoride release properties of the materials.

Clove Oil vs. Thymol

The comparison between clove oil and thymol-modified materials revealed that both additives had a similar effect on fluoride release, with no significant difference in fluoride release between the two additives at any concentration. Both clove oil and thymol at 2% concentration enhanced fluoride release significantly more than the control group.

Quality Assurance and Reliability

To ensure the accuracy of the results, each sample was tested in triplicate, and the mean fluoride release was calculated to minimize variability. The ion-selective electrode was calibrated daily with standard solutions to ensure reliable measurements. All measurements were consistent, and the reliability of the results was confirmed through repeated testing and the use of stringent quality control protocols. The daily calibration of the electrode, along with the triplicate testing, minimized experimental errors and ensured that the findings were both reproducible and accurate.

Implications and Conclusions

The results of this study highlight the significant impact of natural additives, such as clove oil and thymol, on enhancing the fluoride release properties of GICs and componers. Materials modified with these additives, particularly at higher concentrations (2%), demonstrated significantly greater fluoride release compared to unmodified materials, with a more sustained release over a 30-day period. This sustained release is a promising feature for dental materials, as it suggests improved preventive capabilities against caries over time.

Furthermore. the dose-dependent relationship between the additive concentration and fluoride release indicates that clove oil and thymol could be optimized for dental applications, providing a potential avenue for developing dental materials with enhanced therapeutic effects. These findings suggest that incorporating natural antimicrobial and bioactive additives into dental materials could improve their efficacy in fluoride delivery, contributing to better long-term oral health outcomes.

Here is a tabular representation of the study's results:

Table 3

AInterval	Group	Fluoride Release Pattern	Cumulative Fluoride Release	Key Observations
Day 1	Control Group (Unmodified)	Initial burst of fluoride release, followed by gradual decline. Peak release at Day 1.	Moderate	Typical fluoride release pattern for conventional GICs and compomers.
	Modified (0.5% Clove/Thymol)	Enhanced fluoride release compared to controls.	Higher than control	0.5% concentration showed improved release but less pronounced than higher concentrations.
	Modified (1% Clove/Thymol)	Significant increase in fluoride release compared to controls and 0.5% group.	Significantly higher than control	Enhanced release suggests effective modification at this concentration.

	Modified (2% Clove/Thymol)	Maximum fluoride release, significantly higher than all other groups.	Highest release among all groups	30% higher release observed compared to unmodified controls.
Day 7	Control Group (Unmodified)	Gradual reduction in fluoride release, approaching a plateau.	Stable but diminishing	Fluoride release begins to stabilize.
	Modified (0.5% Clove/Thymol)	Sustained release higher than controls, but lower compared to 1% and 2% groups.	Moderate	Improved sustained release compared to controls.
	Modified (1% Clove/Thymol)	Continued elevated release compared to control and 0.5% group.	Significantly higher than control	Sustained fluoride release evident.
	Modified (2% Clove/Thymol)	Maximum fluoride release maintained, significantly higher than other groups.	Highest release among all groups	Sustained and superior fluoride release at this concentration.
Day 14	Control Group (Unmodified)	Stable release, significantly lower than modified groups.	Minimal	Plateauing fluoride release typical of unmodified materials.
	Modified (0.5% Clove/Thymol)	Higher sustained release compared to controls, following similar trends as Day 7.	Moderate	Sustained improvement over control but less effective than 1% and 2% concentrations.
	Modified (1% Clove/Thymol)	Continued elevated fluoride release compared to control and 0.5% group.	Significantly higher than control	Effective fluoride release enhancement maintained.
	Modified (2% Clove/Thymol)	Highest fluoride release maintained.	Highest release among all groups	2% concentration demonstrates sustained superior release.
Day 30	Control Group (Unmodified)	Plateaued fluoride release, stable but lower compared to earlier time points.	Lowest cumulative release	Typical stabilization of fluoride release.
	Modified (0.5% Clove/Thymol)	Sustained release higher than controls but lower than 1% and 2% groups.	Moderate cumulative release	Effective improvement compared to controls but less pronounced than higher concentrations.
	Modified (1% Clove/Thymol)	Elevated sustained fluoride release compared to control and 0.5% group.	Significantly higher cumulative release	Consistent fluoride enhancement maintained over 30 days.
	Modified (2% Clove/Thymol)	Maximum cumulative fluoride release, significantly higher than all other groups.	Highest cumulative release among all groups	Demonstrates dose-dependent improvement, with optimal release at this concentration.

This table captures the key results and findings, illustrating the effects of clove oil and thymol modifications on fluoride release.

DISCUSSION

This study investigated the fluoride release characteristics of GICs and compomers modified with natural additives—clove oil and thymol—at varying concentrations. The findings demonstrate a notable enhancement in fluoride release among modified materials compared to unmodified controls, suggesting the potential of these additives to improve the therapeutic efficacy of dental materials. The discussion explores these findings in relation to existing literature, the implications of the dose-dependent effects observed, and the broader clinical relevance of this research.

Enhanced Fluoride Release in Modified Materials

The results revealed that both clove oil and thymol significantly increased fluoride release from the GICs and compomers, with the highest release observed in the 2% modified samples. This enhancement can be attributed to the incorporation of the natural additives, which may have altered the microstructure or chemical interaction within the material matrix, facilitating greater fluoride ion diffusion. These findings align with previous studies that have demonstrated the beneficial effects of incorporating bioactive compounds in dental

materials to enhance fluoride release, a critical factor in preventing dental caries.

The observed burst release on Day 1 followed by sustained fluoride release over 30 days in the modified materials underscores the potential of these additives to provide both immediate and long-term fluoride benefits. This pattern mirrors the fluoride release kinetics of conventional GICs but with significantly higher cumulative fluoride release, highlighting the superior performance of the modified materials.

Dose-Dependent Effects

The study confirmed a dose-dependent relationship between additive concentration and fluoride release, with higher concentrations (2%) yielding the greatest fluoride release. This trend indicates that clove oil and thymol enhance fluoride release properties proportionally to their concentration. However, it is essential to balance the concentration of additives to avoid compromising the mechanical properties or structural integrity of the base materials. Further research could explore the optimal concentration that maximizes fluoride release without negatively impacting



other critical material properties, such as compressive strength and wear resistance.

Comparison of Clove Oil and Thymol

Both clove oil and thymol demonstrated comparable effects on fluoride release, with no statistically significant differences between the two additives at any concentration. This similarity suggests that either additive could be effectively utilized in modifying dental materials, depending on availability, cost, and specific clinical requirements. The antimicrobial properties of these additives, combined with enhanced fluoride release, could provide a dual benefit by preventing caries and inhibiting microbial growth, thereby improving oral health outcomes.

Implications for Clinical Practice

The findings of this study have several implications for clinical practice. The enhanced and sustained fluoride release observed in the modified materials suggests their potential to offer superior caries prevention compared to conventional materials. This is particularly relevant in high-risk populations or scenarios where prolonged fluoride delivery is crucial, such as in pediatric and geriatric dentistry.

Additionally, the use of natural additives like clove oil and thymol aligns with the growing trend toward biocompatible and eco-friendly materials in dentistry. Their incorporation into GICs and compomers could provide a safer and more sustainable alternative to synthetic additives, addressing both patient safety concerns and environmental considerations.

Limitations and Future Directions

While the study demonstrated the efficacy of clove

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oil and thymol in enhancing fluoride release, several limitations warrant discussion. First, the in vitro design, though standardized, does not fully replicate the complexities of the oral environment, such as the dynamic interaction with saliva, pH fluctuations, and masticatory forces. Future studies should include in vivo evaluations to validate these findings in clinical settings. Second, the impact of the additives on the mechanical properties of the modified materials was not assessed. Given the critical role of strength and durability in restorative materials, further research should investigate whether these additives affect the structural integrity of GICs and compomers.

Lastly, the long-term stability of the additives within the material matrix and their potential interactions with other components warrant further exploration to ensure sustained efficacy over time.

CONCLUSION

This study highlights the potential of clove oil and thymol as natural additives to enhance the fluoride release properties of GICs and compomers. The dose-dependent improvements observed, coupled with their antimicrobial properties, make these modified materials promising candidates for advancing preventive dentistry. Future research should focus on optimizing additive concentrations, evaluating mechanical properties, and conducting clinical trials to confirm their applicability and benefits in real-world settings. By integrating bioactive and biocompatible components into dental materials, this approach represents a step forward in improving oral health outcomes and advancing restorative dental technologies.

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