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Comparative Evaluation of Fluoride Release from Glass Ionomer Cement Modified with Different Concentrations of Chitosan: An *in vitro* Study

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Abstract

Background: Restorative materials releasing fluoride are essential for caries prevention. Among the restoratives, glass ionomer cement (GIC) has a remarkable fluoride-releasing ability.
Aim: To evaluate and compare the effect of three different concentrations of Chitosan-modified GIC on its fluoride-releasing properties.
Materials and methods: Thirty-two disk-shaped pellets having dimensions of 10 x 2 mm were made with Restorative GIC (RX Ease, Shofu, Japan) using silicone molds. Specimens were divided into four groups based on the chitosan concentration in GIC liquid, with eight (n=8) in each. Group 1: Control (Unmodified), Group 2: 10 % Chitosan, Group 3: 20 % Chitosan, and Group 4: 30 % Chitosan. The specimens of each group were immersed in deionized water at various time intervals. Electrodes selective for fluoride ions were employed to analyze the amount of fluoride release at 1, 7, and 14 days. Analysis of variance (ANOVA) followed by Post hoc Tukey's tests were performed for statistical analysis of the obtained data. Significance levels were set at p less than 0.05.
Results: Unmodified GICs exhibited higher fluoride release compared to chitosan-modified GICs at all time points. All samples initially released a high amount of fluoride, which decreased over time.
Conclusion: The addition of chitosan to GIC decreased its fluoride-releasing ability. Unmodified GIC showed better fluoride release than chitosan-modified GICs.
Keywords: Chitosan, Electrodes, Fluoride release, GIC.

1. Introduction

The most common noncommunicable disease in the world is dental caries [1]. Fluoride is a potent anticariogenic agent that can lower the incidence of dental caries [2]. There are many fluoride-releasing materials available today, such as glass ionomers, polyacid modified composite resins, and fluoridated composites [3]. The most popular restorative cement among these materials is glass ionomer cement (GIC) which has a sustained release of fluoride ions. GICs have been modified over the years by altering their composition to improve their properties [4]. Chitosan-modified GICs are the most recent in this vast number of altered GICs [5].

Chitosan (CH) is a deacetylated derivative from bio-polysaccharide chitin, making up the exoskeletons of arthropods such as shrimps, lobsters, and the cell walls of fungi [6]. It is a weak base that is insoluble in water but soluble in dilute aqueous acid solutions such as acetic acid [7]. It is biocompatible, biodegradable and recognized as safe by the US FDA [8]. It possesses antibiotic, antimycotic, and anticarcinogenic properties [9].

The RX Ease glass ionomer (Shofu, Japan) comprises unique dual technology, which contributes to its excellent mechanical and optical properties. The high-functional polycarboxylic acid copolymer technology produces high-density ionic crosslinks between polycarboxylic acid and multivalent metal ions thus enhancing its mechanical properties. The high trans fluoroaluminosilicate Glass (HT Glass) technology enhances its optical properties.

The incorporation of chitosan into GIC was discovered to enhance its flexural strength [10]. Since there is limited existing literature on chitosan-altered GICs, particularly in terms of their fluoride release properties, it is not clear how adding chitosan to GIC can impact its ability to release fluoride. Therefore, this study was designed to assess and compare the influence of three different concentrations of chitosan on the fluoride-releasing effectiveness of GIC.

2. Materials and methods

The sample size was estimated using the G Power software v. 3.1.9.4 (Franz Faul, Universität Kiel, Germany). Considering the effective size to be measured (f) at 55%, the power of the study at 80% and the alpha error at 5%, the total sample size needed was 28. An additional 10% (Higher the power of the study to get more accurate results) to the raw sample size of 28, the total sample size is increased to 32 (8 samples x 4 groups = 32 samples). Ethical clearance was obtained from the institutional ethical committee (Ref. No: SHDCH/IEC/2023-24/11), Sri Hasanamba Dental College and Hospital, Hassan.

2.1 Preparation of chitosan solution

20 mg of CH (Bangalore Fine chem, Bangalore) were weighed and dissolved in 0.3 N acetic acid (Prepared by dissolving 1.8 mL glacial acetic acid (Nice chemicals, Kochi) in 100 mL distilled water) and made up to 100 ml with the same acetic acid in a 100 ml standard flask to get 0.2 mg/mL CH solution [11].

2.2 Preparation of glass ionomer liquid modified with chitosan

To get a strength of 10 v/v% of glass ionomer liquid incorporated with chitosan, 0.1 mL of the produced chitosan solution (0.2 mg/mL) was added to 0.9 mL of the glass ionomer liquid. Similarly, 20% and 30% liquid were prepared (Figure 1).

Figure 1. Glass ionomer cement liquid modified with different concentrations of Chitosan.

2.3 Sample preparation and grouping

The silicone molds with an inner diameter of 10 mm and a depth of 2 mm were used to prepare the samples (Figure 2). A total of 32 specimens were fabricated by mixing the GIC powder and the modified liquid as per the manufacturer's recommendations. The GIC mix was condensed into the silicone mold, and the excess was removed by pressing a glass plate against the mold. All the samples were allowed to set at room temperature for 10 minutes. Samples were divided into four groups based on the concentration of CH in the GIC (Figure 3). Among the four groups, one group was unmodified GIC and was considered the control group (Group 1). The remaining three groups were chitosan-modified, 10% (Group 2), 20% (Group 3) and 30% (Group 4). Then, the discs extracted from the silicon molds and were immersed in deionized (DI) water (4 mL) in plastic vials and incubated at 37 °C for 14 days.

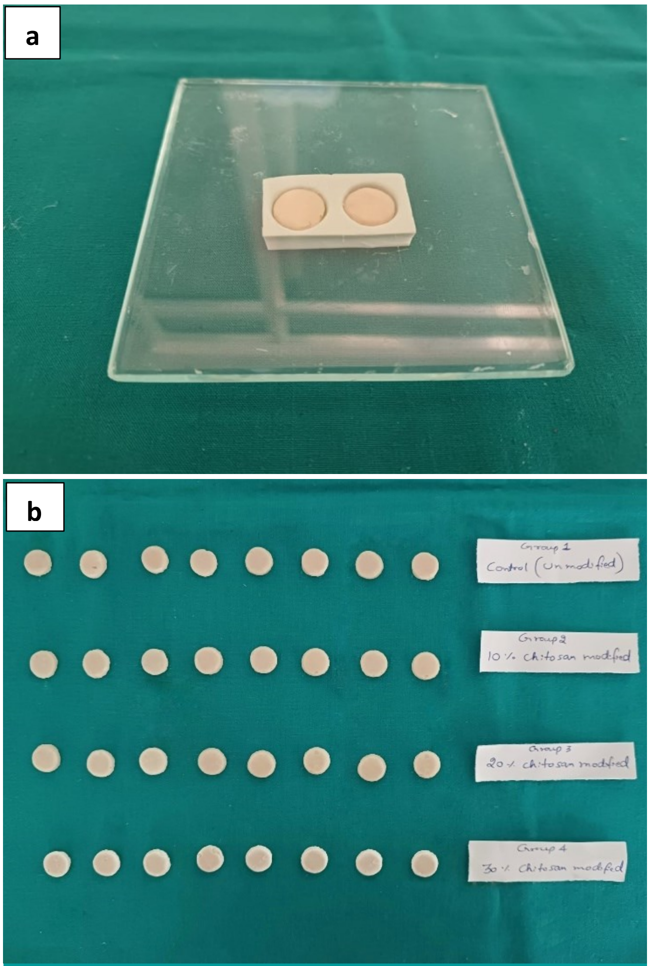

Figure 2. a. Sample preparation using silicone mould, and b. Glass ionomer samples.

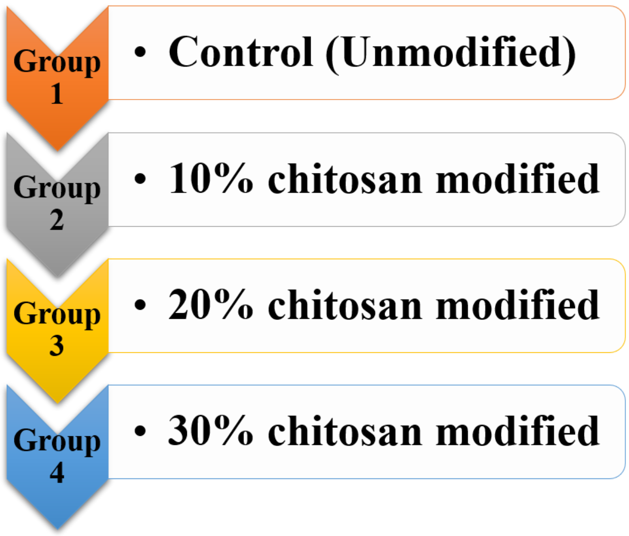

Figure 3. Grouping of samples.

2.4 Fluoride release analysis

Each vial's DI water was changed after the 1st, 7th, and 14th Days. Equal volumes of total ionic strength acid buffer were added to control pH and avoid the formation of fluoride complexes. A precalibrated fluoride ion-selective electrode (Mettler Toledo, Switzerland) was used (Figure 4) to assess the release of fluoride. The total amount of fluoride released was recorded in parts per million (ppm) at every interval. Fluoride release analysis was carried out at Nanowatts Technologies Ltd, Bangalore.

Figure 4. Fluoride Release Analysis.

2.5 Statistical analysis

Statistical Package for Social Science (SPSS) Software, Version 25.0 (IBM Corporation, USA) was used for analyzing the obtained data. Analysis of variance (ANOVA) followed by Post hoc Tukey's tests were performed for statistical analysis. Significance levels were set at p value less than 0.05.

3. Results

The fluoride ion release profiles in DI water from four different groups were recorded for 14 days at three specific intervals. Table 1 shows the comparative evaluation of fluoride release among the groups. Unmodified GIC released a significantly greater quantity of fluoride compared to chitosan-modified GICs. The four groups reported the highest fluoride release after the first day, followed by a progressive decline until the 14th day. ANOVA test displayed significant differences in the fluoride release among the different time intervals in the groups (Group 1: $p=0.0006$; Group 2: $p < 0.001$; Group 3: $p=0.00112$; and Group 4: 0.0008) among the groups at different time intervals. Post Hoc Tukey's test displayed significant difference ($p < 0.01$) between day 1 & day 7; and day 7 & day 14, in all 4 groups.

Table 1. Comparison of fluoride release at different time intervals.

Groups	Fluoride release (Mean ± SD) ppm			p-Value
	Day 1	Day 7	Day 14	
Control (Unmodified)	3.646 ±0.614	1.994 ±0.106	0.338 ±0.032	0.0006
10% chitosan-modified	2.783 ±0.122	1.224 ±0.133	0.191 ±0.075	<0.001
20% chitosan-modified	2.002 ±0.594	1.183 ±0.010	0.338 ±0.027	0.00112
30% chitosan-modified	1.734 ±0.454	1.144 ±0.016	0.114 ±0.011	0.0008

4. Discussion

Glass ionomer cement (GIC) is one of the biomaterials used in dentistry with better properties such as biocompatibility, antibacterial effect, ion leachability and its capacity to bond to bone, enamel, dentin and metals. GIC and its modified dental cements are associated with a lower incidence of secondary caries due to its fluoride-releasing properties [12]. The goal is to achieve the highest possible fluoride content in restorative materials without compromising their physical and mechanical properties. Additionally, the fluoride release should be maximized without affecting the integrity of the filling [13].

The fluoride release is a complex process influenced by various intrinsic and environmental factors. These factors include the composition of the organic matrix and filler, the method of manipulation, the solubility and porosity of the material, its surface area, and the pH [14]. The release of fluoride from GICs occurs through three different mechanisms: surface loss, diffusion through pores and cracks, and bulk diffusion [15]. It has been suggested that the significant fluoride release observed on the first day is linked to the initial surface erosion. In contrast, the subsequent lower and relatively consistent fluoride release is thought to be attributed to fluoride's ability to diffuse through pores and cracks within the cement over time [16]. This study observed the highest amount of fluoride release in the first day and tapering off over weeks. The results of this study were in accordance with the studies by Harhash *et al.*, Tay *et al.*, Hasan *et al.*, Pellizari *et al.*, and Nagi *et al.* confirms this pattern of fluoride release [15-19].

In the present study the unmodified GIC demonstrated better fluoride releasing ability compared to the chitosan-modified groups. This is in contradictory to the previous research by Nishanthine *et al.* [20], Petri *et al.* [10], who found that 10% CH-modified FUJI II GIC had greater fluoride release compared to the conventional GIC.

According to Wiegand *et al.*, the potential to release fluoride from fluoride-containing dental materials varies between different materials and different brands. The optimal fluoride release from restoration is related to their matrices, setting mechanism, fluoride content and several other environmental conditions [21]. In the present study, the GIC with high functional polycarboxylic acid copolymer technology was used, and it might be the reason for decreased fluoride release after modification with chitosan. Earlier studies indicated that the quantity of fluoride released was lower in artificial saliva compared to deionized water. This discrepancy was attributed to the fact that in deionized water, the fluoride-releasing property is more accurately reflected due to the absence of any influence from organic or mineral components present in the solution [22].

In the current research, an ion-selective electrode-based potentiometer device was selected to ensure that the fluoride analysis methods meet universal standards. Additionally, this method offers easy accessibility and a lower detection threshold [23].

Limitation of this study was testing fluoride release in a neutral environment. Fluoride release tends to increase in an acidic medium, which could occur in the oral cavity. The oral cavity presents a pH varying environment that cannot be accurately replicated in experimental conditions.

6. Conclusion

Within the limitations of the present study, the fluoride release in unmodified GIC was found to be higher than the chitosan modified GIC. Further Structural analysis of the cement matrix is needed to confirm and validate these findings.

Conflicts of interest: Authors declared no conflicts of interest.

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