Comparative evaluation of fluoride release and re-release and recharge potential of Zirconomer Improved and Cention

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Abstract

Background: The cariostatic action associated with fluoride-releasing restorative materials is mainly attributed to a sustained release of fluoride. This research aims on comparing Cention N and Zirconomer Improved in vitro fluoride release and re-release.

Methods: Test materials were grouped into two categories containing 15 samples each. The cumulative fluoride release and re-release measurements was made during 1st, 7th and 15th day. The independent sample t-test and paired t-test was used to check mean differences. The level of significance was kept at p<0.05.

Results: At day 1, 7 and 15 the initial fluoride release of zirconomer group was significantly higher (p<0.05) than the cention group. It was found that from day 1 to day 7 the mean initial fluoride release has significantly reduced (p<0.05) for both the groups. Similar results were also seen when comparisons were done between day 7 to day 15 (p<0.05) and day 1 to day 15 (p<0.05) for both the groups.

Conclusion: Zirconomer was more efficient in initial and fluoride re-release than the Cention N restorative material. Further in vivo studies with more parameters are recommended to evaluate fluoride release and cariostatic performance of Cention N and Zirconomer in real environmental circumstances.

Keywords: Cention N; fluoride release; fluoride re-release; Zirconomer

INTRODUCTION

Fluoride is a well-acknowledged anticariogenic agent extensively used in dentistry. A wide array of mechanisms provides fluorides and its anticariogenic effects which include the reduction of demineralization, the enhancement of remineralization, the interference of the pellicle and plaque formation, and the inhibition of microbial growth and metabolism.[1-4] Fluoride released from restorative dental materials is assumed to suppress caries formation by all these mechanisms and therefore reducing or preventing demineralization and promoting remineralization of dental hard tissue structures.[5]

There are many fluoride-containing dental restorative materials available in the market including amalgams, glass ionomers, resin-modified glass ionomer, compomers – polyacid-modified composites, and composite cement. These products have diverse matrices and setting mechanisms because of which they vary in their fluoride release capabilities. It has been proved that the antibacterial and cariostatic properties of restorative materials are often associated with the amount of fluoride released.[5]
The cariostatic action associated with fluoride-releasing restorative materials is mainly attributed to a sustained release of fluoride. Fluoride leaches from these restorative materials decreased over time (more significantly for glass ionomers and resin-modified glass ionomers). To maintain a continuously increased level of fluoride release, the “recharging” of restoratives with fluoride is necessary. The ability of a restorative material to act as a fluoride reservoir mainly depends on the type and permeability of the material. It also depends on the frequency of fluoride exposure and on the kind and concentration of the fluoridating agent. A review of in vitro and in situ published literature shows that various methods of aging (distilled water, deionized water, and human or artificial saliva), aging periods (days to months), and methods of fluoride recharge may also act as contributing factors.

After the application of fluoridated agents, fluoride releases partly by washout of fluoride ions that are retained on the surface or in the pores of the restorative material. Fluoride ions bound to the surface of restorative materials might be more easily detached during an acidic attack (e.g., erosions). The free fluoride ions incorporated into the matrix might also be washed out. The present research is the first of its kind on comparing Cention N and Zirconomer Improved where an in vitro fluoride release and re-release was evaluated.

Cention N is an “alkasite” restorative material. Alkasite refers to a new category of restorative material, which like ormocer or compomer materials is fundamentally a subgroup of the composite material class. This new category utilizes an alkaline filler which is capable of releasing acid-neutralizing ions. In its mixed state (liquid + powder), Cention N contains 78.4% by weight inorganic filler. The alkaline glass accounts for 24.6% in weight of the final material and this releases substantial levels of fluoride (F-) ions - comparable to those released by traditional glass ionomers. The acidic glass also releases hydroxide and calcium ions which further help prevent demineralization of the tooth substrate. The release of F- ions also depends on the pH value in the oral cavity. When the pH value is low (acidic), for example, an active plaque biofilm with cariogenic bacteria, Cention N releases a significantly higher number of ions than compared to neutral pH.

Zirconomer and Zirconomer Improved are novel restorative materials that are composed of ceramic and zirconia-reinforced glass-ionomer cement (GIC). These materials can overcome the drawbacks of previously used amalgam. Zirconomer is developed following a rigorous manufacturing technique that exhibits a strength equivalent to amalgam. The glass component of this high-strength restorative material undergoes finely controlled micronization to achieve optimum particle size and characteristics. The uniform incorporation of zirconia particles in the glass component further reinforces the material for durability and higher tolerance to occlusal load. The polyalkenoic acid and the glass components of this high-strength GI stand for glass ionomer have been specially processed to impart superior mechanical and handling qualities. It contains zirconium oxide, glass powder, polyacrylic acid (20%–50%), tartaric acid (1%–10%), and deionized water as its liquid. Zirconium oxide, the main powder component of Zirconomer, results from baddeleyite (ZrO2) that contains high levels of zirconia ranging from 96.5% to 98.5%. Zirconomer Improved was developed as a reliable and durable self-adhesive tooth-colored zirconia-reinforced posterior bulk-fill restorative material. It comprises nano-sized zirconia fillers for superior handling characteristics and also to enhance esthetic properties.

**MATERIALS AND METHODS**

The present in vitro study was conducted in the Department of Conservative Dentistry and Endodontics, Faculty of Dental Sciences, Rama University, Kanpur. The ethical clearance to carry out this study was obtained from the Institutional Ethical Committee. The study was conducted in accordance with the guidelines laid down by the World Medical Association and the Helsinki Declaration. Two materials, Cention (Ivoclar Vivadent AG, Liechtenstein) and Zirconomer (Shofu Inc., Japan), were tested in the present study and grouped into two categories containing 15 samples each.

**Preparation of the specimen**

Round stainless steel molds (5 mm in diameter and 3 mm in depth) were used to prepare the required samples. The materials were mixed according to the manufacturer’s instructions and packed into the molds. A thin layer of petroleum jelly was used to coat lateral surfaces of the mold to prevent material adhesion. The unwaxed dental floss was held in the center of the mold. A cover of Mylar strip and glass slides and allowed to set at room temperature for 10 s. The light-curing materials were cured from the top and bottom using a light source (QHL75 curing unit Dentply, Germany) for 40 s. The specimens in each group were incubated in a 95% relative humidity environment at 37°C for 24 h before setting. Then, specimens of each group were immersed in 20 ml deionized water in plastic bottles and stored in the incubator at 37°C (Figure 1).

**Fluoride release**

After 24 h, the containers were thoroughly shaken, and then, the samples were removed, dried, and returned into a new vial containing 20 ml of deionized water. The cumulative fluoride release measurement was made during the 1st, 7th, and 15th days.
Exposure protocol
After 15 days of initial fluoride release, the samples of each group were divided into three subgroups of five samples each. The samples were hand brushed with a fluoridated dentifrice (1000 ppm) for 4 min and then wiped clean with a tissue and rinsed for 10 s using copious deionized water and dried. Each sample after fluoride application was suspended in plastic bottles containing 20 ml of deionized water and incubated at 37°C for 24 h.

Fluoride re-releases
Deionized water was then analyzed for fluoride release on the 1st, 7th, and 15th days using ion-specific electrode. After 24 h, samples were removed from the bottle, washed with 1 ml of double-distilled water using a syringe, dried on absorbent paper, and then restored in 20 ml of fresh deionized water.

Measurement of fluoride release
For the analysis of fluoride released from materials into aqueous solutions, an ion-selective electrode attached to an ion meter is most commonly used. A total ionic strength adjustment buffer (TISAB) solution is normally added to the solution in order to control pH and prevent the formation of fluoride complexes. The use of TISAB II frees fluoride ions bound to hydrogen and eliminates hydroxyl ion interference, so enabling an accurate measurement of the total fluoride content.

Statistical analysis
Descriptive and analytical statistics were done. The independent sample t-test and paired t-test were used to check the mean differences between the groups wherever appropriate. The level of significance was kept at \( P < 0.05 \). The percent reduction (%) was calculated by the formula

\[
\text{Percent Reduction} = \left( \frac{N_2 - N_1}{N_1} \right) \times 100
\]

RESULTS
The initial fluoride release and re-release (in ppm) at days 1, 7, and 15 between the Zirconomer and Cention groups were compared [Table 1]. It was found that statistically significant differences existed between the two groups. At day 1, the initial fluoride release of the Zirconomer group (35.07 ± 4.80) was significantly higher (\( P < 0.001 \)) than the Cention group (20.13 ± 3.54). Similar results were also seen at days 7 and 15. The fluoride re-release of the Zirconomer group (24.17 ± 4.47) was significantly higher (\( P < 0.001 \)) than the Cention group (14.89 ± 4.20) at day 1. Similar results were also seen at days 7 and 15.

The mean percent reduction (%) in initial fluoride release and re-release between the Zirconomer and Cention groups was compared. It was found that NO significant difference existed in mean percent reduction (%) in initial fluoride release and re-release between the Zirconomer and Cention groups (\( P > 0.05 \)) [Table 2].

The intragroup comparison of mean initial fluoride release (ppm) of the Zirconomer group was done. It was found that from day 1 (35.0 ± 4.80) to day 7 (35.0 ± 4.80), the mean initial fluoride release has significantly reduced (\( P < 0.001 \)). Similar results were also seen when comparisons were done between day 7 to day 15 (\( P < 0.001 \)) and day 1 to day 15 (\( P < 0.001 \)). For the Cention group, the mean initial fluoride release has also significantly reduced (\( P < 0.001 \)) from day 1 (20.13 ± 3.54) to day 7 (14.73 ± 3.19). Similar results were also seen when comparisons were done between day 7 to day 15 (\( P < 0.001 \)) and day 1 to day 15 (\( P < 0.001 \)) [Table 3].

The intragroup comparison of mean fluoride re-release (ppm) of the Zirconomer group was done [Table 4]. It was found that from day 1 (24.17 ± 4.47) to day 7 (21.20 ± 4.23), the mean fluoride re-release has significantly reduced (\( P < 0.001 \)). Similar results were also seen when comparisons were done between day 7 to day 15 (\( P < 0.001 \)) and day 1 to day 15 (\( P < 0.001 \)). For the Cention group, the mean fluoride re-release has significantly reduced (\( P < 0.001 \)) from day 1 (14.89 ± 4.20) to day 7 (13.18 ± 3.77). Similar results were also seen when comparisons were done between day 7 to day 15 (\( P = 0.010 \)) and day 1 to day 15 (\( P = 0.001 \)).

DISCUSSION
The amount of fluoride released from zirconia-reinforced glass ionomer and Cention N is not well documented in the literature. Till date, this is the first study to compare fluoride release and re-release of Zirconomer Improved and

![Figure 1: Specimens of each group immersed in 20 ml deionized water in plastic bottles](image-url)
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Table 1: Comparison of initial fluoride release and re‑release (in ppm) at days 1, 7, and 15 between Zirconomer and Cention groups

| Timeline         | Zirconomer | Cention | P*   |
|------------------|------------|---------|------|
| Initial          |            |         |      |
| Day 1            | 35.07±4.80 | 20.13±3.54 | <0.001* |
| Day 7            | 24.23±4.64 | 14.73±3.19 | <0.001* |
| Day 15           | 17.90±4.60 | 10.17±3.59 | <0.001* |
| Re‑release       |            |         |      |
| Day 1            | 24.17±4.47 | 14.89±4.20 | <0.001* |
| Day 7            | 21.20±4.23 | 13.18±3.77 | <0.001* |
| Day 15           | 17.67±4.22 | 11.26±3.68 | <0.001* |

*P value derived from independent sample t‑test, †Significant at P<0.05

Table 2: Comparison of mean percent reduction (%) in initial fluoride release and re‑release between Zirconomer and Cention groups

| Timeline         | Zirconomer | Cention | P*   |
|------------------|------------|---------|------|
| Initial          |            |         |      |
| Day 1‑7          | 30.17±13.21 | 26.16±13.92 | 0.426 |
| Day 7‑15         | 25.82±15.60 | 31.96±16.31 | 0.302 |
| Day 1‑15         | 48.51±12.82 | 49.78±15.36 | 0.807 |
| Re‑release       |            |         |      |
| Day 1‑7          | 12.24±6.97  | 10.74±9.79  | 0.675 |
| Day 7‑15         | 16.30±11.63 | 13.68±18.24 | 0.642 |
| Day 1‑15         | 26.68±12.48 | 22.37±21.29 | 0.504 |

*P value derived from independent sample t‑test

Centron N. Zirconomer was found to be more efficient in initial and fluoride re‑release than the Centron N restorative material.

The content of fluoride in restorative materials should, however, be as high as possible without unfavorable effects on mechanical and physical properties of the restorative material. The release of F− ions should also be as high as possible without undue degradation of the filling material. An initial fluoride “burst” effect is beneficial, as it will decrease the viable bacteria that may have been left in the dentin and encourage enamel/dentin remineralization.[14]

The rapid fall of fluoride release during subsequent days results mainly from the initial fluoride burst from the glass particles as they dissolve in polyalkenoate acid during the setting reaction. The initial superficial rinsing effect may also cause a high level of fluoride release on the 1st day, while the constant fluoride release during the subsequent days occurs because of the ability of fluoride to diffuse through cement pores and fractures.[15]

Virmani et al. compared the amount of fluoride released from zirconia-reinforced GIC (Zirconomer, Shofu Inc.), high-density GIC (Ketac™ Molar, 3M™ ESPE™), and packable posterior glass-ionomer restorative material (GC Fuji IX GP). In their research, artificial saliva was used to evaluate fluoride release to better simulate the oral environment. The maximum amount of fluoride release, i.e., 1.584 ppm, was observed by Ketac molar at 24 h. Zirconomer exhibited a maximum amount of release of fluoride at 7 h, 1.026 ppm, followed by Fuji IX at 48 h, 1.088 ppm. Fluoride release by Zirconomer was constant from 14 h up to 10 days with a decline thereafter.[16] The rapid elution pattern of fluoride by Zirconomer may be attributed to the finely controlled micrionization of the glass-ionomer particles as per the manufacturer’s claim. It is in conjunction with results reported by various studies that smaller glass particles provide a larger surface area, which increase the acid-base reactivity, and hence, have increased capacity to release fluoride from the powder more rapidly, thereby increasing the fluoride release of the materials.[16,17,18]

A study by Nicolet Ilié analyzed Centron and described this material as releasing fluoride (F−), hydroxide (OH−), and calcium (Ca2+) ions able to prevent tooth demineralization.[19] Gupta et al. in their study showed higher fluoride ion release from Centron N in acidic pH as compared to neutral pH. This indicates that when conditions become acidic due to cariogenic challenges, GIC and Centron N would release relatively more fluoride ion.[20] In our study, the amount of fluoride release decreased with an increasing period. Similar results have been observed by Kiran and Hegde, [21] Neelakantan et al., [22] Cardoso et al., [23] Dasgupta et al., [24] and Gupta et al.[20] who

Table 3: Intragroup comparison of mean initial fluoride release (ppm) of Zirconomer and Cention groups

| Timeline         | Mean±SD  | P*   |
|------------------|----------|------|
| Zirconomer       |          |      |
| Day 1            | 35.0±4.80 | <0.001* |
| Day 7            | 24.23±4.64 | <0.001* |
| Day 15           | 17.90±4.60 | <0.001* |
| Cention          |          |      |
| Day 1            | 20.13±3.54 | <0.001* |
| Day 7            | 14.73±3.19 | <0.001* |
| Day 15           | 17.90±4.60 | <0.001* |

*P value derived from paired t‑test, †Significant at P<0.05. SD: Standard deviation

Table 4: Intragroup comparison of mean fluoride re‑release (ppm) of Zirconomer and Cention groups

| Timeline         | Mean±SD  | P*   |
|------------------|----------|------|
| Zirconomer       |          |      |
| Day 1            | 24.17±4.47 | <0.001* |
| Day 7            | 21.20±4.23 | <0.001* |
| Day 15           | 17.67±4.22 | <0.001* |
| Cention          |          |      |
| Day 1            | 14.89±4.20 | <0.001* |
| Day 7            | 13.18±3.77 | 0.010* |
| Day 15           | 11.26±3.68 | 0.001* |

*P value derived from paired t‑test, †Significant at P<0.05. SD: Standard deviation
compared GIC with different restorative materials in a different context.

There is one limitation of this study that the novel restorative materials were evaluated in laboratory conditions (in vitro study). Oral environment is dynamic and different from in vitro conditions.

**CONCLUSION**

The present in vitro study compared fluoride release and re-release of novel restorative material Cention N and Zirconomer Improved. It was found that both the materials had a good capability of initial and fluoride re-release. In comparison, Zirconomer was more efficient in initial and fluoride re-release than the Cention N restorative material. Further, in vivo studies with more parameters are recommended to evaluate the fluoride release and cariostatic performance of Cention N and Zirconomer in real environmental circumstances.

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**Conflicts of interest**

There are no conflicts of interest.

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