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Comparison of Fluoride Release and Re-release Between Two Bioactive Restorative Materials and a Giomer: An In-vitro Study

Abstract
Aim: To investigate the fluoride release and re-release potential of two bioactive restorative materials and a Giomer. Methodology: Fifteen disc-shaped samples (n = 5 per group) were primed using three restorative materials: Group A – Cention N, Group B – Activa™ Bioactive Restorative™, and Group C– Beautifil II. Each sample was dipped in 20 mL of artificial saliva. Fluoride ion emission was assessed on days 1, 7, and 14 with a fluoride ion-selective electrode. Following initial measurements, samples were recharged, and fluoride re-release was similarly assessed. The data obtained were interpreted applying one-way ANOVA and Bonferroni post-hoc test ($p < 0.05$). Results: Activa Bioactive Restorative showed the maximum initial fluoride release at every time period, followed by Cention N and Beautifil II. Statistically significant differences were documented among the materials on all measured days ($p < 0.05$). In terms of fluoride re-release, Cention N exhibited the highest values, likely due to its alkasite content and ion-exchange mechanism. Activa showed limited re-release, and Beautifil II demonstrated the lowest fluoride recharge potential. Conclusion: Cention N exhibited superior fluoride re-release capacity, while Activa Bioactive Restorative showed the greatest initial fluoride release. Beautifil II (Giomer) performed the least in both aspects

INTRODUCTION

Globally, dental caries is an etiologically diverse disease affecting the tooth with a high rate of prevalence. In clinical practice, fluoride-based treatments and sealant applications have consistently demonstrated their effectiveness in shielding the teeth from the cariogenic process. Fluoride-containing dental materials demonstrate variable patterns of fluoride release and uptake, functioning as reservoirs that elevate salivary fluoride levels and help prevent secondary caries.[1,2]

To augment the fluoride release, restorative materials incorporated with fluoride are available. They comprise a range of materials like glass ionomers, compomers (polyacid-modified composites), resin modified glass ionomers and composite cements with diverse setting mechanisms and varying fluoride release profiles. Evidence indicates that the cariostatic and antibacterial effects of these materials are closely linked to the quantity of fluoride they release.[3]

Fluoride-releasing restorative material brings about cariostatic action through the prolonged release of fluoride. The fluoride release takes place through the removal of ions adhered to the outer layer or entrapped within the voids of the restorative material. Additionally, free fluoride ions trapped in the matrix may subsequently diffuse out. The fluoride ions that leach out of these materials gradually decline due to unavailability. To ensure a consistent level of fluoride release, it is essential to renew the fluoride content in these materials. The capacity of a material to be recharged with fluoride is influenced by its composition, permeability, rate of fluoride exposure, and the fluoridating agent concentration.[3]

Cention N belongs to a novel category of restorative materials termed alkasites, which are considered a subgroup of composites. This material is characterized by the inclusion of alkaline fillers capable of releasing ions that neutralize acids.[4] In its prepared state, Cention N consists of 78.4% inorganic fillers, with alkaline glass making up 24.6%. This component is chiefly responsible for its notable fluoride (F^-) ion release, resembling the release pattern observed in traditional glass ionomer cements. Along with fluoride, alkaline glass also liberates calcium and hydroxide ions, thereby enhancing the material's capacity to minimize tooth demineralization. Cention N comes in a powder–liquid presentation and can set through self-curing. It can also be optionally light-cured for reinforcement.[5] As a resin-based system, it is convenient for clinical use. It provides an economical esthetic option that delivers reliable, durable, and high-quality restorations efficiently.[6]

Activa Bioactive restorative material, introduced in the year 2013 by Pulpdent Company is similar to composite resin, offering high durability, abrasion resistance, and the ability to promote remineralization and apatite formation.[4] This material possess good marginal seal, effectively minimizing microleakage and the risk of secondary cavities. Activa does not contain Bis-phenol A or Bis-GMA, resulting in enhanced biocompatibility and it adapts to ongoing pH variations in the oral environment, thereby supporting the restoration and replenishment of ionic nature of saliva, dental tissues, and the material per se.[4]

The next restorative material is Giomer which is a tooth-colored, resin based, tooth-colored material that incorporates pre-reacted glass ionomer (PRG) technology. Fluoride-consisting acid-reactive glass is combined with polyalkenoic acid in an aqueous solution to create the PRG filler, and then it is blended it into the resin matrix. The PRG filler technology is of two types: Surface-reacted PRG (S-PRG) filler and Fully-reacted PRG (F-PRG) filler. The S-PRG fillers are employed in second-generation Giomers, addressing the problems of moisture sorption and expansion that were prevalent in first-generation Giomers. They share similar properties with those of resin composites, offering outstanding aesthetics, ease of polishing, biocompatibility, and an improved surface finish. These agents exhibit an antiplaque effect by creating a protective film layer through interaction with saliva, which is believed to decrease the plaque adhesion onto the surface and thus inhibits bacterial colonization.[7]

The present study was aimed to compare Cention N, ACTIVA™ and BEAUTIFIL II by assessing their in vitro fluoride emission and re-emission properties.

MATERIALS AND METHODOLOGY

Ethical clearance: Since this was an invitro study involving Teflon moulds, ethical approval is not applicable. However, ethical exemption was obtained from Institutional Ethics Committee
(No: YEC-1/2023/067)

Study location: the study was conducted at Yenepoya Dental College, Yenepoya (Deemed to Be University).

Patient Consent: Since this was an invitro study involving Teflon moulds, patient consent is not applicable.

This in-vitro study was carried in a controlled laboratory environment. The sample size was estimated for a within-factor of two way repeated- measures ANOVA using G* Power software. The minimum number of sample size required was 15 to detect an effect size of 1.0677 as statistically significant with 99% power and 1% level of significance, within standard deviation to be 1.44 and correlation between repeated measures to be 0.5. In total, 15 samples of restorative materials were prepared and allocated into three groups.

- Group A (Cention N): 5 samples of light cured Cention N restorative material.
- Group B (Activa Bioactive): 5 samples of dual cured Activa Bioactive restorative material.
- Group C (Beautifil II): 5 samples of light cured Beautifil II restorative material.

Each sample in every group was evaluated for fluoride ion emission on the 1st, 7th, and 14 days. The same samples were re-evaluated again for fluoride ion re-emission on the 1st, 7th, and 14 days after treatment with a fluoridated dentifrice.

SAMPLE PREPERATION:

Preparation of the specimen:

A 5 mm diameter and 3 mm deep stainless-steel moulds were used for preparing the test specimens. The restorative materials were manipulated as per the instructions given by the manufacturer and placed in the

moulds. They were kept at room temperature for 10 seconds to allow for the setting. Polymerization was carried out for 40 seconds for light-activated materials from the upper and lower surfaces using a curing unit.
Following this the specimens were placed in a controlled environment for 24 hours at 37°C with 95% relative humidity. Following this, each specimen was dipped in separate containers having 20 ml of artificial saliva with a pH of 5. The containers were made of plastic and were allowed to rest in an incubator set at 37°C.

Fluoride release analysis:
Following a 24-hour period, the containers were agitated, and the specimens were collected, allowed to dry, and relocated into new vials with 20 ml of artificial saliva. The fluoride emission was analyzed cumulatively on the 1st, 7th, and 14th days.

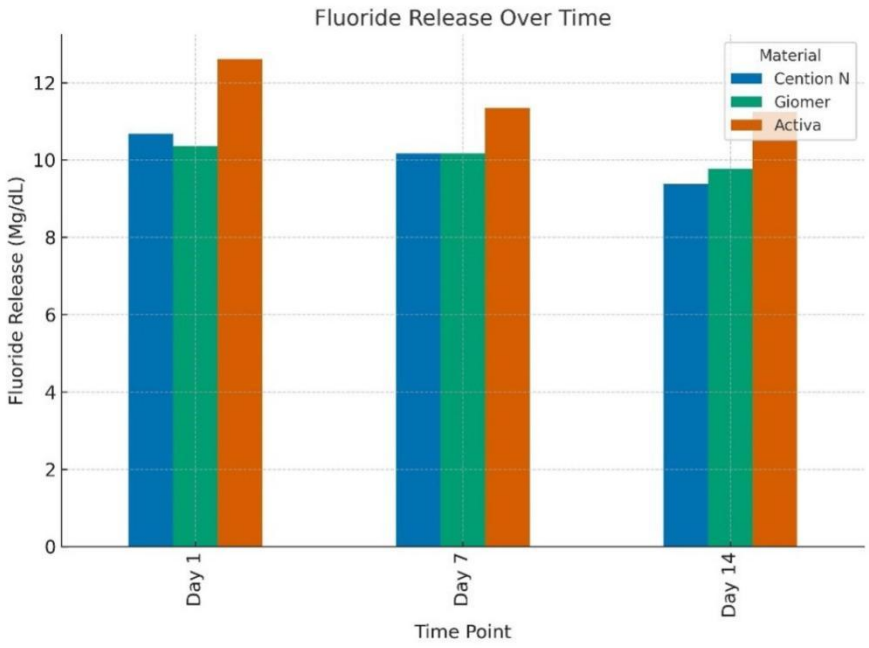
Fluoride re-release analysis:
A fluoridated toothpaste containing over 900 ppm fluoride was manually brushed over the specimens for 4 minutes, after which each sample was placed in plastic containers holding 20 ml of artificial saliva at 37°C and was left undisturbed for 24 hours.
The samples were then taken out of the plastic containers, rinsed with 1 ml of double-distilled water using a syringe, let to dry on absorbent paper, and transferred to new vials with 20 ml of artificial saliva. The released fluoride from artificial saliva was analyzed on the 1st, 7th, and 14th days with an ion-specific electrode.

STATISTICAL ANALYSIS
The data was organized in MS Excel, and all statistical analyses were performed using IBM SPSS 27 software. The significant difference between the three groups was assessed through one-way ANOVA. The significant difference between three-time intervals (Day 1, Day 7 and Day14) within a group of continuous variables was assessed through one-way repeated ANOVA. In both cases (ANOVA and repeated ANOVA), post hoc test (Bonferroni correction) was carried out for dyadic comparisons. If the obtained p value was < 0.05, it was evaluated to be statistically significant.
The values was uploaded in MS Excel, and was interpreted with statistical analysis using IBM SPSS version 27. The normal distribution of all variables ($p > 0.05$) was affirmed using the Shapiro-Wilk test, allowing for parametric testing.

RESULTS
Fluoride release:
One-way ANOVA displayed a statistically significant difference in the release of fluoride among the three materials (Cention N, Giomer, and Activa Bioactive Restorative) at Day 1, Day 7, and Day 14 ($p < 0.001$).
Post hoc Bonferroni analysis presented that:
Day 1: Significant differences between all groups ($p < 0.001$).
Day 7: Significant differences between Activa Bioactive Restorative and both Cention N and Giomer ($p < 0.001$); no significant difference between Giomer and Cention N ($p = 1.000$).
Day 14: Significant differences between all groups ($p < 0.001$).
Intragroup comparisons using ANOVA with repeated observations demonstrated statistically significant reductions in fluoride release over time within each group ($p < 0.001$), and the post hoc analysis validated significant differences between all-time points for each material ($p < 0.05$). (Table 1) (Figure 1).

Table 1: Fluoride Release (Mean ± SD) at Different Time Points

Group	Day 1 (Mg/dL)	Day 7 (Mg/dL)	Day 14 (Mg/dL)
Cention N	10.67 ± 0.0546	10.17 ± 0.0255	9.37 ± 0.0642
Giomer	10.36 ± 0.0261	10.17 ± 0.0228	9.77 ± 0.0259
Activa Bioactive Restorative	12.61 ± 0.0224	11.34 ± 0.074	11.25 ± 0.046



Fluoride Re-Release

Similar analysis of fluoride re-release showed statistically significant differences among the groups at all evaluated time periods ($p < 0.001$).

Post hoc Bonferroni analysis presented that:

Day 1, Day 7, and Day 14: The difference between all groups was found to be statistically significant ($p < 0.001$). Cention N was seen to have the highest fluoride re-release at all intervals, followed by Activa Bioactive Restorative and Giomer. (Table 2) (Figure 2).

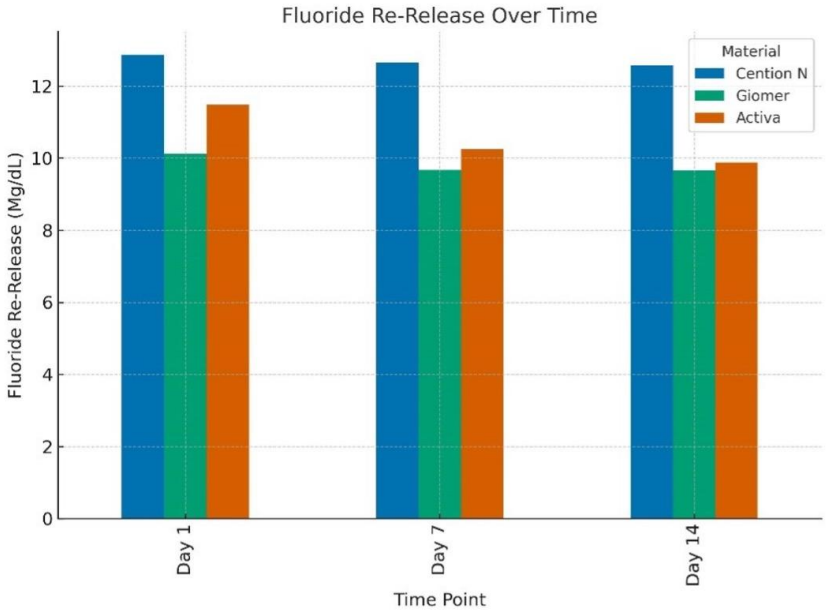


Table 2: Fluoride Re-Release (Mean ± SD) at Different Time Points

Group	Day 1 (Mg/dL)	Day 7 (Mg/dL)	Day 14 (Mg/dL)
Cention N	12.87 ± 0.0415	12.65 ± 0.0303	12.58 ± 0.0249
Giomer	10.12 ± 0.0297	9.67 ± 0.0224	9.66 ± 0.023
Activa-Bioactive Restorative	11.48 ± 0.0288	10.25 ± 0.1665	9.88 ± 0.0451

Intragroup comparisons using repeated ANOVA observations revealed significant differences over time within each group, except for Giomer between Day 7 and Day 14, where the difference was not significant statistically ($p = 0.155$)**.(Table 3)**

Table 3: Intragroup Comparisons (Repeated Measures ANOVA)

Comparison	Cention N (p-value)	Giomer (p-value)	Activa Bioactive Restorative (p-value)
Day 1 vs Day 7	<0.001	<0.001	<0.001
Day 1 vs Day 14	<0.001	<0.001	<0.001
Day 7 vs Day 14	<0.001	0.155	0.019 (Release) / 0.035 (Re-release)

DISCUSSION

Based on the data got from the Global Burden of Disease Study, dental caries affects about 2.5 billion people worldwide, with its incidence increasing by 14.6% over the past decade.[8] The fluoride released from restorative materials acts by reducing demineralization, augmenting remineralization, suppressing bacterial growth, and altering metabolism.[10] The leached-out fluoride from these restorative cements decreases over a period of time; hence recharging of restoratives is necessary to maintain and cater the continuous levels of fluorides. The potential of dental restorative materials to function as a storehouse of fluoride is determined by its composition and permeability, periodicity of fluoride exposure, and the type and the fluoridating agent concentration.[10]

This study was purposed to evaluate the fluoride emission and re-emission among three different materials: Cention N, Activa Bioactive Restorative and a Giomer, over a 14-day period.

When the day wise fluoride release trends were studied, it showed that, on day 1, Activa Bioactive Restorative exhibited the highest fluoride release (12.61 mg/dL), significantly higher than Cention N (10.67 mg/dL) and Giomer (10.36 mg/dL). The rapid initial fluoride burst is likely due to the surface dissolution of fluoride-containing particles in these materials.[13,14]

By Day 7, fluoride released across all materials, but Activa Bioactive Restorative continued to maintain the highest levels (11.34 mg/dL), while Cention N and Giomer released similar amounts (10.17 mg/dL each). By Day 14, fluoride release had further declined, with Activa Bioactive Restorative (11.25 mg/dL) still outperforming Cention N (9.37 mg/dL) and Giomer (9.77 mg/dL).

The fluoride release from Cention N was lower than Activa Bioactive Restorative, but higher than Giomer suggesting that its bioactive glass particles effectively contribute to fluoride diffusion.[17] Giomer displayed the least fluoride release among all the materials in the study. This aligns with findings that Giomer releases less fluoride than RMGIs but more than conventional resin composites.[18] One possible reason for the variation in fluoride release rates between Giomer and glass ionomer could be the level of porosity. Giomer has lower porosity compared to glass ionomer, which may lead to reduced fluoride release.[13]

The findings of this study are coherent with research by Porenczuk A et al. who observed that resin-modified bioactive materials like Activa release significantly more fluoride than Giomer and alkasite restoratives.[19] Similarly, studies by Nigam AG, Mousavinasab SM, Geurtsen W et al., have suggested that Cention N's fluoride release is moderate and not as sustained as glass ionomer-based materials.[19-22]

The recharging of the restoratives was achieved using fluoridated dentifrice. When the fluoride re-release patterns were analyzed, interestingly, Cention N demonstrated maximum fluoride re- release followed by Active Bioactive Restorative and Giomer. A plausible explanation is that Cention N responded more vigorously under acidic conditions, where its surface protective layer may have degraded more rapidly than that of Activa Bioactive Restorative and Giomer.[23] This result is in synchrony with Gupta N et al. who concluded that Cention N released significantly higher fluoride ions in acidic pH.[24] When the day-wise fluoride Re-Release trends were observed they showed that, on day 1, after fluoride recharge, Cention N released the most fluoride (12.87 mg/dL), significantly higher than Activa Bioactive Restorative (11.48 mg/dL) and Giomer (10.12 mg/dL). By Day 7, Cention N continued to show the highest fluoride re-release (12.65 mg/dL), while Activa dropped to 10.25 mg/dL, and Giomer decreased further to 9.67 mg/dL. On Day 14, Cention N still maintained superior re-release (12.58 mg/dL) compared to Activa (9.88 mg/dL) and Giomer (9.66 mg/dL).

However, this study has a few shortcomings. The fluoride release was assessed under in vitro conditions, which may not fully simulate the fluctuating oral environment. The study was also limited to a specific time frame, and long-term fluoride release patterns beyond this period remain unexplored. Long-term in-vivo studies are needed to confirm these in-vitro findings and to further explore the fluoride release and recharge mechanisms of bioactive restoratives and Giomers.

CONCLUSION

From the findings, Cention N possessed the maximum fluoride re-release compared to Activa Bioactive Restorative and Giomer. While Activa Bioactive Restorative demonstrated superior initial fluoride release, its re-release capacity was comparatively lower. Giomer exhibited the least fluoride release and re-release, suggesting that it may not be the most suitable choice for cases requiring long-term fluoride release and recharge benefits.

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