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Effect of Topical Fluoride Application Temperature on Recharge and Rerelease in Different Restorative Materials

Ji-Eun Kim [a], *Ji-Yeon Hong* [a,b], *Kwang-Mahn Kim* [a], *Jae-Sung Kwon* [a,b]*

[a] *Department and Research Institute of Dental Biomaterials and Bioengineering, Yonsei University College of Dentistry, Seoul, Republic of Korea*
[b] *BK21 FOUR Project, Yonsei University College of Dentistry, Seoul, Republic of Korea*

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ABSTRACT

Introduction and aims: This study aimed to evaluate the influence of topical fluoride application temperature on the recharge and rerelease behaviour of three fluoride-containing restorative materials treated with 1.23% acidulated phosphate fluoride (APF) gel and 8% stannous fluoride (SnF_2).

Methods: Specimens of an alkasite-restorative material (Cention N), a resin-modified glass ionomer (Fuji II LC), and a conventional glass ionomer (Fuji IX GP) were prepared ($n = 32$ per material; $n = 8$ for each combination of fluoride agent and temperature). After an initial 64-day fluoride release phase in deionized water, samples were recharged with APF gel or SnF_2 at room temperature (23 ± 2°C) or high temperature (HT) (55 ± 2°C) for 4 minutes. Subsequent fluoride rerelease was measured for 32 days. Physicochemical properties of fluoride agents, including viscosity, temperature, and pH, were analysed. Elemental mapping of fluoride and tin distribution was performed using scanning electron microscope-energy-dispersive X-ray spectroscopy.

Results: All restorative materials initially showed rapid fluoride release that decreased over 64 days. After topical application, cumulative fluoride rerelease was significantly higher under HT than room temperature across all groups ($P < .05$), particularly with APF gel. RMGI demonstrated the greatest fluoride rerelease, while CGI showed the highest initial release. Scanning electron microscope-energy-dispersive X-ray spectroscopy revealed higher fluoride content at HT across all materials in both surface and cross-sectional analyses, regardless of the fluoride agent used. Tin distribution was detected only in SnF_2-treated groups.

Conclusion: Elevated application temperature significantly improves the fluoride recharge and sustained release capacity of restorative materials, with outcomes depending on the fluoride agent and material type.

Clinical relevance: Applying topical fluoride at higher temperatures may enhance its anticariogenic efficacy by promoting greater fluoride uptake and prolonged release from restorative materials. These findings highlight the importance of considering application conditions when optimizing preventive protocols in clinical practice.

Introduction

Dental caries, a common chronic disease affecting 60% to 90% of the global population, is especially prevalent among children and adolescents. It is caused by oral bacteria demineralizing tooth enamel and dentin with acid, which can cause structural damage and other problems.[1-3] It is a serious public

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* *Corresponding author*. Department and Research Institute of Dental Biomaterials and Bioengineering, Yonsei University College of Dentistry, 50-1 Yonsei-ro, Seodaemun-gu, Seoul 03722, Republic of Korea.
E-mail address: jkwon@yuhs.ac (J.-S. Kwon).
Ji-Eun Kim: http://orcid.org/0000-0002-8185-3261
Jae-Sung Kwon: http://orcid.org/0000-0001-9803-7730
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health problem that impacts people's quality of life and incurs costs. Caries can lead to tooth loss, functional impairment, and a substantial financial burden if treatment is not received. One of the most important aspects of dental care is preventing dental caries, which is more effective than treating them. Consequently, the use of fluoride has emerged as one of the most popular and successful preventative measures in contemporary oral healthcare.[4]

Fluoride is recognized for its antibacterial properties and its capacity to release fluoride ions, which are effective in preventing the deposition of calcium fluoride in the oral cavity. It inhibits the enzymatic activity of microorganisms that cause caries, promotes the remineralization of early-stage caries, and helps prevent dental caries.[5,6] This process is crucial in averting the development of secondary or recurrent caries, which is a leading cause of restoration failure over time. For these reasons, fluoride is widely used in dentistry, either applied directly to teeth or incorporated into restorative materials. Fluoride-containing restoratives continuously release fluoride after their initial placement, offering anticariogenic benefits. Additionally, these materials can be recharged with topical fluoride agents, allowing for prolonged fluoride rerelease over time.[7] This capability is not governed by simple diffusion; rather, it is influenced by several physicochemical factors, including matrix composition, porosity, hydrophilicity, filler distribution, and the ionic characteristics of the topical fluoride agents.[8,9] Fluoride-containing restorative materials developed for this purpose include glass ionomers, resin-modified glass ionomers, compomers, and alkasite-restorative material.

To enhance fluoride recharge efficiency, various topical agents such as 1.23% acidulated phosphate fluoride (APF), fluoride varnish, sodium fluoride (NaF), and stannous fluoride (SnF_2) are used clinically.[10,11] This facilitates the sustained release of fluoride ions, which helps prevent dental caries. To ensure this preventive effect is maintained over time, regular fluoride intake is necessary. However, the interaction between these agents and restorative materials depends not only on the composition of the agents but also on environmental factors, such as the temperature at the time of application.[12-14]

Notably, the intraoral tolerance temperature for hot liquids and food is generally reported to be up to approximately 55°C,[15] and transient intraoral temperatures may reach around 70°C when consuming hot beverages.[16] Previous research has evaluated the rerelease of fluoride from restorative materials using NaF at 4, 37, and 55°C,[13] demonstrating a significant increase in both the rate and amount of fluoride recharge at elevated temperatures. Other studies have investigated the effect of APF gel application at 4, 25, and 37°C on enamel microhardness.[17] Nevertheless, the comprehensive roles of application temperature on both surface/cross-sectional fluoride uptake and long-term fluoride release from various restorative materials remain insufficiently explored.

Therefore, to explore a method applicable to clinical practice, this study aims to compare the fluoride recharge and rerelease behaviour of three fluoride-containing restorative materials (alkasite-restorative material, Resin-modified glass-ionomer cement, Conventional glass-ionomer cement) after treatment with 1.23% APF gel and 8% SnF_2 under room temperature (RT; 23 ± 2 °C) and high temperature (HT; 55 ± 2 °C) conditions. By examining the interaction between material type, fluoride agent, and application temperature, this study seeks to provide fundamental insights for optimizing clinical fluoride application strategies. The null hypothesis is that the temperature at which fluoride agents are applied has no significant effect on the fluoride recharge and rerelease performance of fluoride-containing restorative materials.

Materials and methods

Materials

Three commercially available fluoride-containing restorative materials were used in this study: Cention N (Ivoclar Vivadent), Fuji II LC Capsule (GC Corporation), and Fuji IX GP Fast Capsule (GC Corporation). Two types of topical fluoride agents were applied: 1.23% APF gel (Natural-F Gel, Denbio Co) and 8% stannous fluoride (SnF_2) (Sigma-Aldrich) solution. The APF gel was used to simulate tray-based application, while the SnF_2 solution was used to mimic brush-based topical application. The list of investigated materials, along with their compositions provided by the manufacturers, is presented in Table 1.

Study design

Initially, specimens were immersed in deionized water, and fluoride release was measured for 64 days to decrease fluoride content and simulate clinical ageing. Following the first release phase, all specimens underwent fluoride uptake using either APF gel or SnF_2 solution under HT or RT conditions. The APF gel was applied for 4 minutes utilizing tray contact simulation, and SnF_2 was applied to the teeth for the same time. After fluoride treatment, the specimens were washed and kept in deionized water. The subsequent fluoride rerelease was monitored for another 32 days. To assess elemental absorption, the concentrations of fluoride and tin ions at the surface and cross-section were measured using energy-dispersive X-ray spectroscopy (EDS). The experimental protocol and workflow are shown schematically in Figure 1.

Physicochemical properties

Viscosity

Viscosity tests were performed using an MCR 702e dynamic shear rheometer (Anton Paar, GmbH) equipped with 25 mm parallel plates. The gap between rheometer plates was set to 1 mm. APF gel viscosity was also measured at RT and HT and at shear rates ranging from 0.1 to 100 s^{-1}.

pH and temperature change

Fluoride agents stored under HT and RT conditions were analysed using a pH meter (Orion 4 Star, Thermo Fisher Scientific). The electrode was calibrated using standard buffer solutions at pH 4.01, 7.00, and 10.01. To measure temperature change under conditions identical to the fluoride application process, 1 g of each fluoride agent was placed in a 24-well plate, stored at HT, and then transferred to a 37 ± 1°C environment. The temperature was recorded every 30 seconds for 4 minutes using a data logger (GL840-20CH, GRAPHTEC), and

Table 1 – Materials used in this study, including restorative materials containing fluoride and topical fluoride agents.

Category	Materials	Classification (code)	Composition
Fluoride-containing materials	Cention N (Ivoclar Vivadent, Schaan, Liechtenstein)	Alkasite-restorative material (ARM)	- Resin matrix: UDMA, DCP, Aromatic aliphatic-UDMA, PEG-400 DMA - Filler: Barium aluminium silicate glass, ytterbium trifluoride, Isofiller, calcium barium aluminium fluorosilicate glass, calcium fluorosilicate glass (78.4 wt%, 57.6% of inorganic filler) particle size range of 0.1-35 μm Powder/liquid ratio (g/g) = 4.6/1.0
	Fuji II LC (GC Co)	Resin-modified glass-ionomer cement (RMGI)	- Resin matrix: Polyacrylic acid, 2-HEMA, dimethacrylate - Filler: Al-Si-glass (58 wt%) powder/liquid ratio (g/g) = 3.3/1.0
	Fuji IX GP Fast Capsule (GC Corporation)	Conventional glass-ionomer cement (CGI)	- Powder: Fluoroaluminosilicate glass, polyacrylic acid powder - Liquid: Polyacrylic acid, polybasic carboxylic acid
Topical fluoride agents	Natural-F Gel (Denbio Co, Ltd)	1.23% acidulated phosphate fluoride gel (APF gel)	Ethanol, sodium fluoride, phosphoric acid, xanthan gum, sodium benzoate, glycerin, tocopheryl acetate, water
	Tin(II) fluoride (Sigma-Aldrich)	8% stannous fluoride (SnF_2)	≥95% (CAS No. 7783-47-3)

pH was measured simultaneously. All measurements were performed in triplicate, and the average values at each time point were used for analysis.

Specimen preparation and initial fluoride release

Thirty-two specimens per material were prepared according to manufacturers' instructions using Teflon moulds (2 mm thickness, 5 mm diameter). To prevent wall contact and ensure free ion release, nylon threads were inserted through each specimen. Capsule-type materials (RMGI, CGI) were mixed for 10 s using an amalgamator (Ultramat S, SDI). Light-curable materials (ARM, RMGI) were cured for 20 s on each side; CGI was self-cured for 10 min at RT.

Initial fluoride release was measured on days 1, 2, 4, 8, 16, 32, and 64. All samples were stored in 2 mL of fresh deionized water, which was changed daily. The fluoride concentration was determined using a pH/ion meter (Orion 4 Star pH/ISE Benchtop, Agilent, Thermo Fisher Scientific Inc) that was attached fluoride ion-specific electrode (Orion 9609BNWP, Thermo Fisher Scientific Inc). Calibration was performed with fluoride standard solution of 0.1, 1, 10, and 100 ppm before measurement.

Recharge of fluoride and rerelease

After fluoride release for 64 days, specimens of each material were randomly divided into four subgroups (n = 8 each)

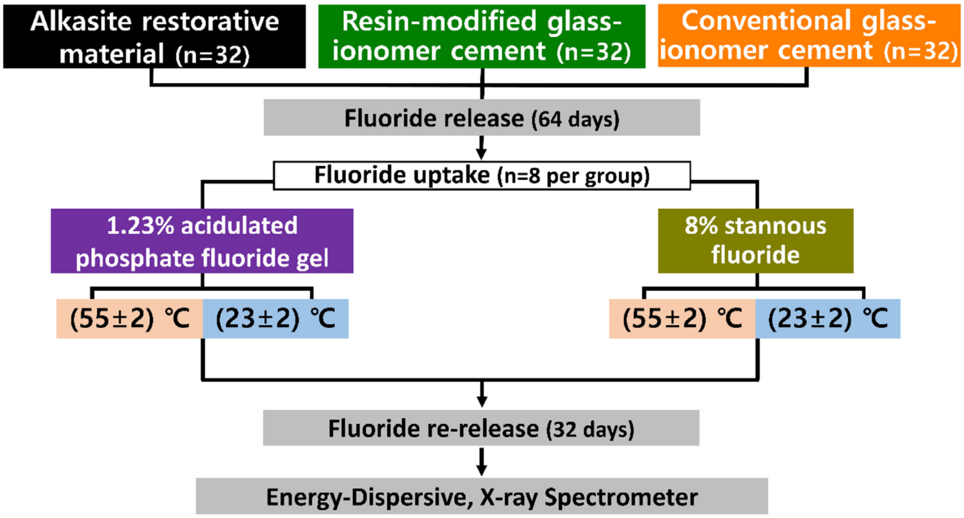

Fig. 1 – Flowchart depicting the study design and experiment workflow.

according to fluoride agent type (APF gel or SnF_2) and agent storage temperature (RT or HT). Each fluoride agent, prestored at RT or HT, was applied to the specimen surface for 4 minutes. Specimens were immediately transferred to a 37 ± 1°C incubator to simulate intraoral conditions. The specimens were then rinsed under running distilled water for 1 minute and air-dried for 10 seconds.

Fluoride rerelease was measured on days 1, 2, 4, 8, 16, and 32 after recharging. Specimen storage and fluoride quantification were performed using the same protocol described in the section ‘Specimen preparation and initial fluoride release’.

Fluoride (F-) and tin (Sn) distribution patterns analysis

After completing the 32-day fluoride rerelease, one specimen was randomly selected from each group and recharged on one surface only, under the same conditions as the previous fluoride application. The agents stored at RT or HT were applied to the specimen surface for 4 minutes and then immediately placed in a 37 ± 1°C incubator to simulate intraoral conditions, as described in the section ‘Recharge of fluoride and rerelease’. After rinsing with distilled water, the specimens were sectioned in half to expose the cross-section and then dried at RT in a desiccator for 3 days. A representative specimen from each group was observed using a scanning electron microscope coupled with an EDS (JEOL JSM-7800F) for fluoride atomic (%) characterization. Elemental mapping was performed for fluoride (F) and tin (Sn) to assess their distribution in the specimens. Before the EDS analysis, the specimens were sputter-coated with platinum for 90 seconds.

Statistical analysis

Statistical analysis was performed using IBM SPSS Statistics version 27.0 (IBM Corp.). Viscosity and temperature values were analysed using an independent samples *t* test to compare the RT and HT groups. The nonparametric Kruskal–Wallis test was performed to compare fluoride release across different time points within each material group for the initial fluoride release (over 64 days), and to compare temperature-dependent differences between the RT and HT groups at each time point for the fluoride rerelease (over 32 days). Mann–Whitney *U* tests were conducted for multiple comparisons following the Kruskal–Wallis test.

Results

Physicochemical properties of fluoride agents

In the shear rate range from 0.1 to 100 s^{-1}, the average value of RT was 185.54 ± 26.56 Pa·s, and the average value of HT was 176.85 ± 8.92 Pa·s (Figure 1A). There was no significant difference in viscosity between temperatures at each shear rate ($P > .05$). Figure 2B shows the temperature changes of APF and SnF_2 measured at 30-second intervals for 4 minutes after transferring from 55 ± 2°C storage to a 37 ± 1°C environment. After 4 minutes, the temperatures of APF gel and SnF_2 decreased to 44.60 ± 0.69°C and 44.43 ± 0.67°C, respectively, with no significant difference between them ($P > .05$). Figure 2C presents the pH changes of both fluoride agents stored under HT and RT conditions, measured at 30-second intervals for 4 minutes. Regardless of temperature conditions, SnF_2 maintained approximately pH 2.0, while APF gel maintained approximately pH 5.0. Both agents showed lower pH values when stored under HT conditions compared to RT conditions.

Initial fluoride release

All fluoride-containing restorative materials initially exhibited rapid fluoride release, followed by a significant gradual decrease and nonconstant release pattern over the subsequent 64 days (Figure 3). On day 1, CGI showed the highest fluoride release, 22.13 ± 5.38 ppm, which was greater than that of RMGI, 11.67 ± 1.60 ppm, and ARM, 5.28 ± 1.28 ppm. From day 4 onward, the release rate declined more gradually, and after day 16, all materials exhibited fluoride release levels below 3 ppm, reaching less than 1 ppm by day 64.

Fluoride rerelease

The cumulative fluoride rerelease after topical application of 1.23% APF gel and 8% SnF_2 is presented in Figure 4. All fluoride-containing restorative materials showed the greatest difference between HT and RT on day 1. Although the daily fluoride release decreased over time, the cumulative release continued to increase gradually. Overall, the HT group consistently exhibited higher cumulative fluoride release than the RT group across all measurement days. Notably, in the RMGI group treated with SnF_2, a significant difference between HT and RT was observed only on day 1, whereas all other fluoride-containing restorative material groups showed significant differences at all time points ($P < .05$).

Result of EDS

The results of EDS mapping for O, F, Al, Si, Ca, and Sn are presented in Table 2. In the APF gel application group, all fluoride-containing restorative materials consistently exhibited higher fluoride atomic (%) values on the surface compared to the cross-section (inside), with the HT condition yielding higher values than RT in both regions. In the SnF_2 application group, the surface also showed an increased fluoride content under the HT condition. However, a contrasting result was observed in the cross-section of ARM, where a higher fluoride atomic (%) value was detected under the RT condition. Overall, regardless of the type of fluoride agent used, the average fluoride atomic (%) values in both the surface and inside regions followed the order of CGI, RMGI, ARM, and the values were consistently higher under HT than RT conditions.

Discussion

This investigation examined fluoride recharge and rerelease characteristics of three fluoride-containing restorative materials following treatment with 1.23% APF gel and 8% SnF_2 at varying temperatures. The rheological characteristics and

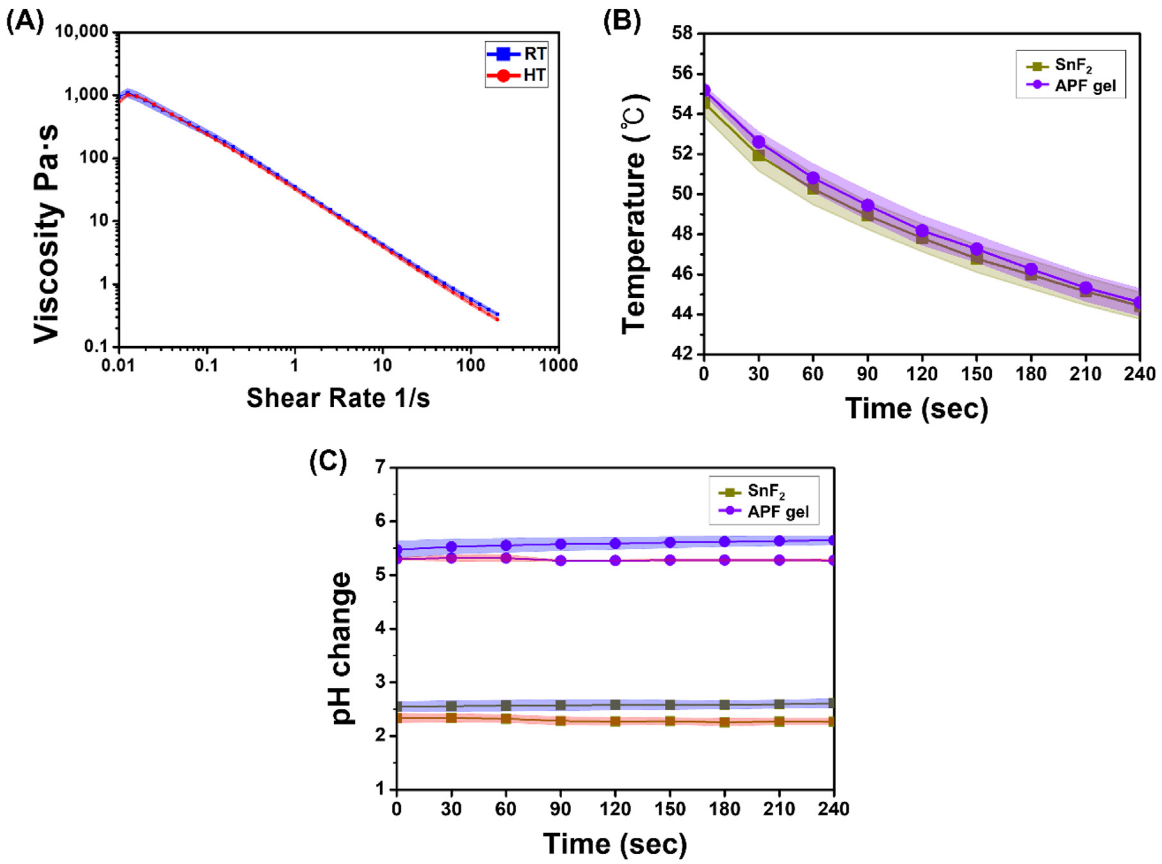

Fig. 2 – (A) Change in the viscosity of 1.23% acidulated phosphate fluoride (APF) gel as a function of shear rate at room temperature (RT) and high temperature (HT). (B and C) Temperature and pH changes of SnF_2 and APF gel: (B) temperature was measured after transferring samples from HT storage to a 37 ± 1°C environment; (C) pH was measured for samples stored under HT and RT conditions.

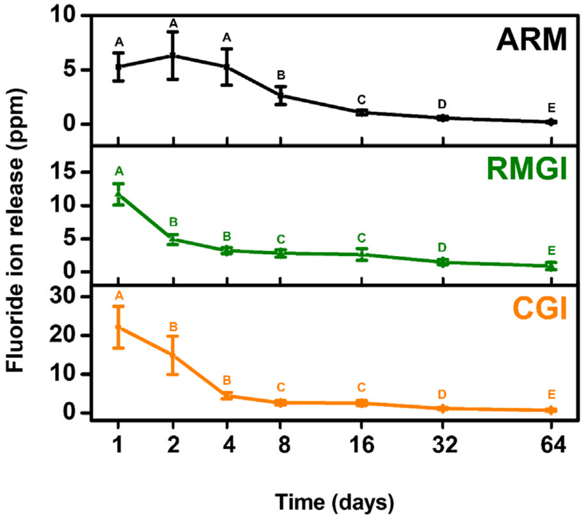

Fig. 3 – Fluoride ion rerelease (ppm) from ARM, RMGI, and CGI measured on days 1, 2, 4, 8, 16, 32, and 64. Different uppercase letters indicate statistically significant differences in fluoride release within each material group over time ($P < .05$). ARM, alkasite-restorative material; CGI, conventional glass-ionomer cement; RMGI, resin-modified glass-ionomer cement.

thermal behaviour of the fluoride agents are critical determinants that may explain the observed differences in fluoride uptake and rerelease efficiency under varying temperature conditions. APF gel, commonly applied inside the mouth using trays, must possess specific viscosity and flow properties to ensure effective contact with tooth surfaces and facilitate efficient fluoride ion penetration.[18] These characteristics allow the gel to reach all areas of the teeth, including interdental spaces, thereby providing fluoride to help prevent cavities. In this study, rheological tests of APF gel performed at 55°C – close to the highest temperature experienced inside the mouth – showed minimal changes in viscosity across all shear rates when compared to measurements at RT. This result highlights the excellent thermal stability of APF gel and supports earlier research indicating that commercial fluoride gels display non-Newtonian flow characteristics with stable viscosity between 25 and 42°C.[19] Conversely, because SnF_2 solutions are inherently low-viscosity aqueous solutions applied topically to the teeth using a toothbrush or a similar device, the effect of temperature on their viscosity is considered clinically negligible. Therefore, rheological analysis of SnF_2 was not included in this investigation.

Professional application of topical fluoride agents, such as APF gel and SnF_2 solution, typically follows a standardized exposure duration of 4 minutes to achieve optimal

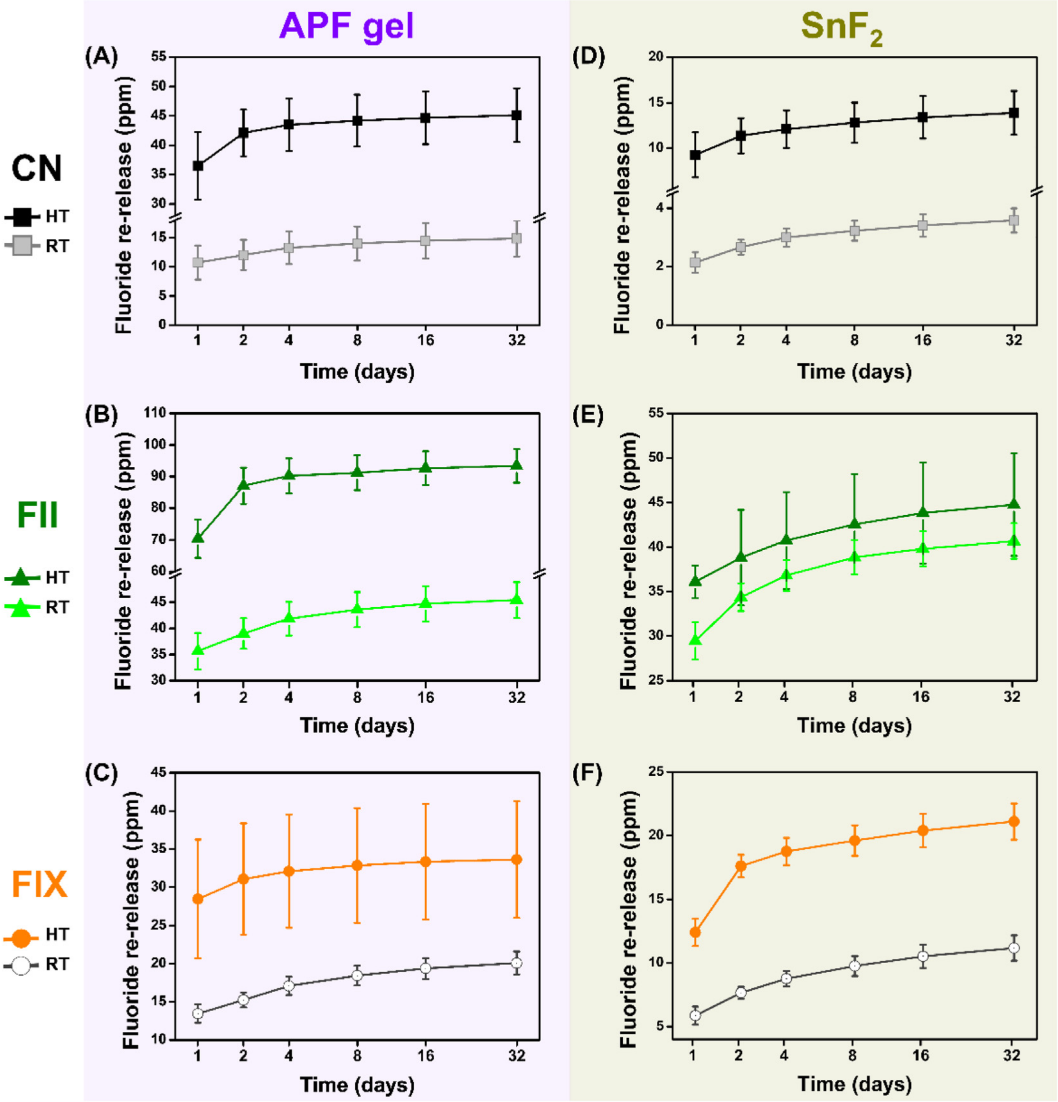

Fig. 4 – Cumulative fluoride rerelease (ppm) measured on days 1, 2, 4, 8, 16, and 32 after topical application of 1.23% acidulated phosphate fluoride gel (APF gel) or 8% stannous fluoride (SnF_2). (A-C) Cumulative rerelease after fluoride uptake with APF gel, (D-F) Cumulative rerelease after fluoride uptake with SnF_2. ARM, alkasite-restorative material; CGI, conventional glass-ionomer cement; HT, high temperature; RMGI, resin-modified glass-ionomer cement; RT, room temperature.

anticariogenic efficacy.[17,20] In this in vitro study, the thermal behaviour of APF gel and SnF_2 was monitored during this critical 4-minute window after being transferred immediately from elevated storage temperature 55 ± 2°C to an intraoral simulation environment 37 ± 1°C. Both agents exhibited a gradual decrease in temperature, reaching approximately 44 to 45°C by the end of 240 seconds (Figure 2B). The temperature levels of both agents stayed consistently high during the entire application period, demonstrating a sustained thermal benefit when used warm. However, given the clinical differences, it is important to consider that while studies often assume a 55 ± 2°C storage condition for topical application,[17] in actual practice, fluoride agents are applied directly to the tooth surfaces, either transferred to a tray or using a cotton applicator. This process is influenced by factors such as saliva, oral airflow, and the tray itself, which all facilitate heat dissipation. Therefore, the temperature of the fluoride agents applied to the tooth would likely decrease more rapidly than observed under laboratory conditions, where the agents were kept warm throughout the application, and this possibility should not be disregarded.

Both agents exhibited lower pH values when stored and applied under HT conditions compared to RT. This trend is primarily attributed to the temperature-dependent increase in acid dissociation. As temperature rises, the ionization constant (Ka) of weak acids increases, enhancing the release of H^+ ions in aqueous solutions.[21] For APF gel, which contains acidic phosphate and fluoride in a buffered formulation, the measured pH in this study was above 5 under both temperature conditions; however, elevated temperature still enhanced dissociation, resulting in a further pH decrease. For SnF_2, hydrolysis and dissociation of tin(II) fluoride in aqueous solution increase H^+ release, thereby intensifying acidity.

Table 2 – Surface and internal tin (Sn) atomic percentage (%) measured by SEM-EDS after SnF_2 application under high temperature (HT) and room temperature (RT) conditions for each restorative material.

Fluoride application	Temperature	Fluoride-containing restorative materials	Section	Element (%)					
				O	F	Al	Si	Ca	Sn
APF gel	HT	ARM	Surface	63.19	21.63	3.95	10.59	0.64	-
			Inside	64.10	8.97	4.70	20.68	1.56	-
	RT		Surface	69.10	14.53	3.60	12.02	0.76	-
			Inside	64.59	6.70	3.78	18.62	6.31	-
	HT	RMGI	Surface	68.45	19.65	5.03	1.62	5.24	-
			Inside	61.34	15.01	12.14	11.29	0.22	-
	RT		Surface	73.04	13.85	5.42	1.02	6.67	-
			Inside	60.41	14.68	12.74	11.99	0.18	-
	HT	CGI	Surface	24.91	62.30	10.34	2.25	0.20	-
			Inside	59.73	19.79	12.77	7.40	0.31	-
	RT		Surface	56.37	30.33	10.74	2.41	0.14	-
			Inside	59.89	18.75	13.59	7.72	0.05	-
SnF_2	HT	ARM	Surface	76.27	10.47	1.94	10.02	0.94	0.36
			Inside	57.96	6.85	1.07	23.54	10.58	0.00
	RT		Surface	76.81	8.15	2.27	12.03	0.33	0.42
			Inside	64.84	8.43	2.65	17.97	5.90	0.22
	HT	RMGI	Surface	75.93	5.00	2.99	1.33	0.00	14.75
			Inside	64.54	17.10	9.18	9.09	0.00	0.09
	RT		Surface	78.25	2.75	3.86	1.51	0.00	13.62
			Inside	65.18	16.51	9.29	8.98	0.00	0.04
	HT	CGI	Surface	72.01	8.14	4.27	0.03	0.51	15.04
			Inside	59.88	18.94	12.87	8.12	0.07	0.11
	RT		Surface	75.24	5.94	7.24	1.14	0.13	10.30
			Inside	57.22	17.42	14.55	10.29	0.48	0.03

In the initial fluoride release phase, all restorative materials exhibited the highest fluoride release on day 1, followed by a progressively decreasing, nonlinear release pattern. This trend is consistent with findings from previous studies.[22,23] The initial release amounts followed the order: FIX > FII > ARM. The superior initial fluoride release of FIX can be attributed to its high content of fluoroaluminosilicate glass particles and the absence of resin components. As a conventional glass ionomer, FIX undergoes an acid–base reaction that allows rapid leaching of fluoride ions from the glass matrix during early setting, resulting in an initial burst effect.[24] FII, a resin-modified glass ionomer (RMGIC), exhibited moderate fluoride release. Although it also contains fluoroaluminosilicate fillers, the presence of resin components such as HEMA can slow water diffusion and ionic exchange, leading to a lower but sustained fluoride output compared to conventional glass ionomer cements.[25] Since its encapsulated ion-releasing glass and less hydrophilic resin matrix allow for slower and more limited fluoride release, Cention N – an alkasite-restorative material based on UDMA – exhibits a modest initial fluoride release.[26]

Fluoride release behaviour was evaluated under various temperature conditions following the topical application of APF gel and SnF_2 to three fluoride-containing restorative materials. Interestingly, while FIX exhibits the highest initial fluoride release, FII demonstrates the greatest fluoride rerelease following APF and SnF_2 treatments. This result contrasts with previous reports suggesting that conventional glass ionomer cements, due to their greater porosity and hydrophilicity, have superior fluoride uptake and rerelease capabilities compared to RMGIC.[7,27] This reversal implies that fluoride rerelease is influenced by more complex factors than baseline fluoride levels alone, including the material's porosity, matrix composition, and fluoride recharge capacity. All fluoride-containing restorative materials exhibited higher fluoride release under the HT condition compared to the RT condition, with this trend being particularly pronounced in the APF gel application group. This temperature-dependent effect can be explained by Fick's law of diffusion, which states that elevated temperatures increase ion mobility,[28] thereby enhancing fluoride penetration into both the surface and interior of the restorative materials.[29] It is worth noting that, under the HT condition with APF gel application, ARM demonstrated a higher cumulative fluoride release than FIX. ARM, a resin-based restorative material containing alkaline glass, barium aluminium silicate glass, and calcium fluorosilicate fillers,[30] has a chemical composition distinct from conventional fluoride-releasing materials, which may influence its interactions with fluoride ions under specific temperature and pH conditions.[31]

EDS analysis revealed distinct differences in fluoride distribution between the surface and cross-sectional regions. In the APF gel-treated groups, all restorative materials exhibited consistently higher fluoride atomic (%) values on the surface than in the cross-section, as expected. In contrast, the SnF_2-treated groups showed an inverse pattern in most materials, where the cross-sectional regions contained higher fluoride concentrations than the surface. Notably, the fluoride concentration in the cross-section decreased under high-temperature conditions. This phenomenon can be attributed to the chemical instability of SnF_2 at low pH (≈ 2) and elevated temperatures in the presence of moisture and oxygen.[32,33] Under these conditions, Sn^{2+} ions are prone to oxidation to Sn^{4+} or conversion into insoluble precipitates such as SnO_2, thereby reducing the availability of free fluoride ions for diffusion. At RT, this oxidative degradation is limited, allowing for greater ionic penetration and sustained retention of fluoride within the bulk material. These findings demonstrate that fluoride distribution within restorative materials is influenced not only by the intrinsic chemistry of the topical agent but also by temperature-dependent changes in viscosity, diffusivity, and redox stability. After SnF_2 application, little Sn was detected on the ARM surface, which can be attributed to the resin-based matrix of the material. ARM, as an alkasite-restorative containing a UDMA resin matrix, exhibits low hydrophilicity and lacks chemical affinity for Sn^{2+} ions, unlike conventional glass ionomer-based materials such as FII and FIX.[34] In contrast, substantial surface Sn was observed in FII and FIX, suggesting that these materials are more reactive in acidic conditions and readily participate in cation-exchange reactions.

Therefore, this study rejects the null hypothesis that the application temperature of fluoride agents does not significantly affect the fluoride recharging and re-emission behaviour of fluoride-containing restoratives. The findings show that temperature is not merely an environmental factor but a critical determinant influencing the physicochemical stability of fluoride agents, their diffusion behaviour, and fluoride deposition within the material. This study's limitations stem from its in vitro technique, which does not fully simulate the complex oral environment, including salivary flow, pH variations, and mechanical wear. Furthermore, the therapeutic implications of fluoride therapy parameters such as frequency, duration, and concentration were not investigated. Future research should include long-term in vivo trials using a variety of application methods. Nonetheless, this study systematically controlled and contrasted three crucial variables restorative material type, fluoride agent, and application temperature under standard settings, resulting in evidence-based recommendations for therapeutic fluoride applications.

Conclusion

This study demonstrated that fluoride application under elevated temperatures significantly enhances the recharge capacity and long-term fluoride rerelease of restorative materials. In particular, APF gel showed the ability to maintain high fluoride concentrations both on the surface and within the material. These findings underscore the importance of considering multiple factors – such as formulation type, pH, and biocompatibility – rather than relying solely on increased temperature when designing clinical fluoride recharge protocols.

From a clinical perspective, for patients with high caries risk or multiple restorations, prewarming topical fluoride agents to approximately 55°C prior to application may be considered to maximize the protective effects of fluoride-releasing materials. Although intraoral conditions such as saliva and rapid heat dissipation are likely to reduce gel temperature quickly, thereby minimizing the risk of pulpal thermal damage, clinicians should still adjust the application temperature appropriately according to individual patient tolerance. This evidence may serve as a valuable foundation for optimizing fluoride application strategies and developing personalized preventive treatments in the future.

Author contributions

Ji-Eun Kim (First author): Conceptualization, methodology, investigation, data curation, formal analysis, writing – original draft. Ji-Yeon Hong: Methodology, validation, formal analysis. Kwang-Mahn Kim: Conceptualization, resources, supervision, writing – review and editing. Jae-Sung Kwon (Corresponding author): Conceptualization, project administration, supervision, funding acquisition, writing – review and editing.

Conflict of interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this article.

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REFERENCES

1. Xu Y, You Y, Yi L, et al. Dental plaque-inspired versatile nanosystem for caries prevention and tooth restoration. Bioact Mater 2023;20:418–33. doi: 10.1016/j.bioactmat.2022.06.010.
2. Collaborators GOD, Bernabe E, Marcenes W, et al. Global, regional, and national levels and trends in burden of oral conditions from 1990 to 2017: a systematic analysis for the global burden of disease 2017 study. J Dent Res 2020;99(4):362–73.
3. Pitts NB, Zero DT, Marsh PD, et al. Dental caries. Nat Rev Dis Primers 2017;3(1):1–16.
4. Samaranayake L, Porntaveetus T, Tsoi J, Tuygunov N. Facts and fallacies of the fluoride controversy: a contemporary perspective. Int Dent J 2025;75(4):100833. doi: 10.1016/j.identj.2025.04.013.
5. Tenuta LMA, Cury JA. Fluoride: its role in dentistry. Braz Oral Res 2010;24:9–17.
6. Bansal R, Bansal T. A comparative evaluation of the amount of fluoride release and re-release after recharging from aesthetic restorative materials: an in vitro study. J Clin Diagn Res: JCDR 2015;9(8):ZC11.
7. Wiegand A, Buchalla W, Attin T. Review on fluoride-releasing restorative materials—fluoride release and uptake characteristics, antibacterial activity and influence on caries formation. Dent Mater 2007;23(3):343–62. doi: 10.1016/j.dental.2006.01.022.
8. Tiwari S, Kenchappa M, Bhayya D, et al. Antibacterial activity and fluoride release of glass-ionomer cement, compomer and zirconia reinforced glass-ionomer cement. J Clin Diagn Res: JCDR 2016;10(4):ZC90.

9. Gao W, Smales R. Fluoride release/uptake of conventional and resin-modified glass ionomers, and compomers. J Dent 2001;29(4):301–6.
10. Shashikiran N, Subba Reddy V, Patil R. Evaluation of fluoride release from teeth after topical application of NaF, SnF2 and APF and antimicrobial activity on mutans streptococci. J Clin Pediatr Dent 2006;30(3):239–45.
11. Chan AKY, Tamrakar M, Jiang CM, Tsang YC, Leung KCM, Chu CH. Clinical evidence for professionally applied fluoride therapy to prevent and arrest dental caries in older adults: a systematic review. J Dent 2022;125:104273.
12. Sen M, Madhyastha PS, Bangera MK, Natarajan S, Kotian R. Effect of time, temperature, and storage on fluoride release and recharge of esthetic restorative materials. J Orofac Sci 2021;13(2):90–5.
13. Yan Z, Sidhu S, Mahmoud G, Carrick T, McCabe J. Effects of temperature on the fluoride release and recharging ability of glass ionomers. Oper Dent 2007;32(2):138–43.
14. Yoda A, Nikaido T, Ikeda M, Sonoda H, Foxton RM, Tagami J. Effect of curing method and storage condition on fluoride ion release from a fluoride-releasing resin cement. Dent Mater J 2006;25(2):261–6.
15. Ernst C-P, Canbek K, Euler T, Willershausen B. In vivo validation of the historical in vitro thermocycling temperature range for dental materials testing. Clin Oral Investig 2004;8(3):130–8.
16. Barclay CW, Spence D, Laird WR. Intra-oral temperatures during function. J Oral Rehabil 2005;32(12):886–94. doi: 10.1111/j.1365-2842.2005.01509.x.
17. Milani S, Shahrabi M, M-Abdi N. Effect of temperature of 1.23% acidulated phosphate fluoride gel on enamel microhardness of permanent teeth. Int J Dent 2024;2024(1):8227152.
18. Keratibumrungpong K, Trairatvorakul C, Jirakran K, Govitvattana N. A comparison of the fluoride 'Paint-On' vs tray application techniques for enamel remineralisation. Int Dent J 2024;74(5):1006–15.
19. Plaizier-Vercammen J, Lecluse E, Boute P, De Neve RE. Rheological properties of topical fluoride gels. Dent Mater 1989;5(5):301–5.
20. Nayak P. Topical fluoride for prevention of dental caries: a review. Indian J Forensic Med Toxicol 2020;14(4):9120–3.
21. Ferrari V, Cutler DJ. Temperature dependence of the acid dissociation constants of chloroquine. J Pharm Sci 1987;76(7):554–6. doi: 10.1002/jps.2600760714.
22. Nigam AG, Jaiswal J, Murthy R, Pandey R. Estimation of fluoride release from various dental materials in different media—an in vitro study. International J Clin Pediatr Dent 2009;2(1):1.
23. Garcez RMVdB, Buzalaf MAR, Araújo PAd. Fluoride release of six restorative materials in water and pH-cycling solutions. J Appl Oral Sci 2007;15:406–11.
24. Forsten L. Fluoride release and uptake by glass-ionomers and related materials and its clinical effect. Biomaterials 1998;19(6):503–8.
25. Kumari PD, Khijmatgar S, Chowdhury A, Lynch E, Chowdhury CR. Factors influencing fluoride release in atraumatic restorative treatment (ART) materials: a review. J Oral Biol Craniofac Res 2019;9(4):315–20.
26. Ruengrungsom C, Burrow MF, Parashos P, Palamara JEA. Evaluation of F, Ca, and P release and microhardness of eleven ion-leaching restorative materials and the recharge efficacy using a new Ca/P containing fluoride varnish. J Dent 2020;102:103474. doi: 10.1016/j.jdent.2020.103474.
27. Singh H, Rashmi S, Pai S, Kini S. Comparative evaluation of fluoride release from two different glass ionomer cement and a novel alkasite restorative material-an in vitro study. Pesqui Bras Odontopediatria Clin Integr 2020;20:e5209.
28. Crank J. The mathematics of diffusion. New York, NY: Oxford Univ. Press; 1975. p. 414.
29. Lee SY, Dong DR, Huang HM, Shih YH. Fluoride ion diffusion from a glass–ionomer cement. J Oral Rehabil 2000;27(7):576–86.
30. Gupta N, Jaiswal S, Nikhil V, Gupta S, Jha P, Bansal P. Comparison of fluoride ion release and alkalizing potential of a new bulk-fill alkasite. J Conserv Dent Endod 2019;22(3):296–9.
31. Aliberti A, Di Duca F, Triassi M, et al. The effect of different pH and temperature values on Ca2+, F−, PO43−, OH−, Si, and Sr2+ release from different bioactive restorative dental materials: an in vitro study. Polymers 2025;17(5):640.
32. Zhang S, Govindaraju GV, Cheng CY, et al. Oxidative stability of chelated Sn(II)((aq)) at neutral pH: the critical role of NO(3)(-) ions. Sci Adv 2024;10(40):eadq0839. doi: 10.1126/sciadv.adq0839.
33. Huang Y, Liu Y, Pandey NK, et al. Iron oxide nanozymes stabilize stannous fluoride for targeted biofilm killing and synergistic oral disease prevention. Nat Commun 2023;14(1):6087. doi: 10.1038/s41467-023-41687-8.
34. Mederos M, León E, García A, et al. In vitro characterization of a novel resin-based restorative material containing alkaline fillers. J Appl Oral Sci 2024;32:e20230219. doi: 10.1590/1678-7757-2023-0219.