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Ion-releasing Resin Composites as Clear Aligner Attachments: Comparison of Caries-inhibition Effect and Shear Bond Strength to Enamel

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Abstract

Objectives: To evaluate mineral loss and lesion depth of enamel adjacent to different ion-releasing resin composites in conjunction with artificial caries induction, and to evaluate shear bond strength to enamel.

Methods: Three ion-releasing resin composites (ACTIVA™ BioACTIVE-RESTORATIVE™, BEAUTIFIL Injectable X_{SL} and Cention® N) with or without adhesive system were investigated in comparison with a conventional resin composite (Filtek™ Z350 XT). Effect of Caries Inhibition in Adjacent Enamel – 36 human premolars with cylindrical cavities and filled with restorative materials underwent 14 days of artificial caries induction and were sectioned into two cross-sectional specimens (n=12). Mineral loss and lesion depth were measured at 10, 260, 510, and 760 µm from the tooth-restoration interface and analyzed using repeated measures ANOVA and pairwise comparison for within group comparisons ($p<0.05$) and One-way ANOVA with Tukey's post hoc test for comparisons among groups ($p<0.05$). Shear bond strength test – 108 human premolars were embedded in self-curing acrylic resin. A polyethylene tube was placed on each surface and filled with restorative materials. Each group was divided into 2 subgroups (n=9) whether the specimens were thermocycled or not. Shear bond strength was tested with the Instron® 5566 universal testing machine. Failure analysis was conducted using a stereomicroscope. Shear bond strength was analyzed using two-way ANOVA and Tukey's multiple comparison test ($p<0.05$).

Results: At 14 days post-caries simulation, ACTIVA™ BioACTIVE-RESTORATIVE™ and Cention® N with and without adhesive demonstrated an ability to inhibit caries formation at 10 µm from the restoration-enamel interface. Shear bond strength to enamel of Cention® N with adhesive had the highest values for both non-thermocycling (21.68±1.86 MPa) and thermocycling (21.17±2.4 MPa) condition, being significantly higher than other groups except for the conventional resin composite (20.3±1.85 MPa for non-thermocycling and 19.16±2.29) MPa for thermocycling condition.

Conclusions: The use of Cention® N with adhesive provides the optimal combination of shear bond strength and caries inhibition effect, which is potentially a superior candidate for clear aligner attachments.

Keywords: caries inhibition, clear aligner attachment, ion-releasing resin composite, shear bond strength

Introduction

Orthodontic treatment is a field that combines both physics and biomechanics to correct malocclusions in addressing three main goals: function, occlusal stability, and aesthetics.[1] In recent years, clear aligner treatment has been considered a more comfortable and esthetic alternative to conventional fixed appliance orthodontics, offering certain advantages such as better aesthetics, removability, and smaller dimensions.[2] Attachments are bonded to the tooth surface and work with clear aligners to move teeth in the desired direction.[3]

The selection of attachment materials must consider both physical and mechanical properties. The attachment materials should resist staining and appear similar to that of natural teeth.[4] Mechanically, the material should maintain its shape and integrity throughout the treatment, enduring the forces exerted during eating and the insertion and removal of clear aligners, to effectively facilitate tooth movement throughout the treatment duration.[5,6]

A common issue during treatment with clear removable appliances is the development of initial caries, or white spot lesions around the attachments. This occurs because the appliances or materials attached to the tooth surface make cleaning difficult, leading to the accumulation of plaque and an increase of cariogenic bacteria, which results in demineralization of the enamel.[7]

The choice of restorative materials can affect the mineral shifts (demineralization and remineralization) of tooth structures. Ion-releasing resin composite that can release ions, such as fluoride ions, calcium ions, and phosphate ions, can help reduce mineral loss and promote remineralization of the tooth structure.[8] Additionally, hydroxyl ions help neutralize the acidic environment created by bacteria.[9] These ions play a crucial role in enhancing the tooth structure's resistance to caries.

ACTIVA™ BioACTIVE-RESTORATIVE™ (Activa) is a self-adhesive, bioactive material in a self-mixing syringe that contains high molecular weight polyacrylic acid, similar to that in resin-modified glass ionomers, but without methacrylate polymerizable groups. It includes urethane dimethacrylate monomers and dimethacrylate phosphate, which enhance its mechanical properties and bond strength.[10] It lacks bisphenol A (BPA), bisphenol A-glycidyl methacrylate (bis-GMA), and BPA derivatives, thus avoiding polymerization shrinkage and stress. The fillers used are silanized fluoroaluminosilicate (FAS) and silanized nonreactive fillers, contributing to the material's wear resistance and esthetics.[11] Moreover, when used in conjunction with an adhesive system, Activa has demonstrated comparable bond strengths to nanocomposite.[12] BEAUTIFIL Injectable X_{SL} (X_{SL}), leveraging Giomer technology, is known for its self-leveling properties, which enhance handling and adaptation to cavity walls. The new nano surface pre-reacted glass-ionomer (S-PRG) fillers offer an optimal balance of light transmission and diffusion for a perfect shade match.[13,14]

Cention® N, an alkasite material supplied as a hand-mixed powder and liquid, can be used in bulk, serving as an alternative to amalgam according to the manufacturer. It has reactive silanized fluoro-alumino-silicate glass similar to those used in glass ionomer cement and advertised as highly reactive especially in an acidic environment. It releases fluoride and calcium ions, preventing demineralization and enhancing remineralization of tooth structure,[15] as well as hydroxyl ions, neutralizing acidic conditions.[16] An *in vitro* study reported that Cention® N can form apatite on its surface, thereby remineralizing the underlying dentin.[11] This property classifies Cention® N as a bioactive material, making it a resin composite with proven bioactivity.[17] In terms of bonding properties, Cention® N showed a superior shear bond strength compared to nanohybrid composite and Fuji IX after being exposed to water aging and exhibited lesser marginal leakage.[18]

Previous studies have characterized the fundamental changes that occur in natural white spot lesions (WSLs) from both materials and microstructural perspectives. Huang *et al.,* demonstrated a correlation between elastic modulus and mineral density for the enamel component of WSLs using nanoindentation and computed x-ray microtomography (micro-CT).[19] Micro-CT is a powerful tool to study the demineralization and remineralization of teeth.[20,21] The data on mineral loss and lesion depth in tooth structures obtained from micro-CT indicate the ability of restorative materials to enhance tooth structures resist mineral loss under simulated caries conditions.[22] Additionally, micro-CT provides distinct advantages, such as non-destructive, high-resolution 3D imaging, enabling precise quantification of mineral loss and lesion depth across different depths of enamel and dentin.[23]

Moreover, choosing the most suitable resin composite type to produce durable attachments is considered challenging. Bonding performance of the attachment material to enamel is important and directly affects the efficiency of tooth movements and treatment outcomes. Research on clear aligner technology is constantly progressing, but studies on the selection of optimal attachment materials to enamel remain relatively limited.(24,25) Two primary types of composite resins are commonly used for fabricating attachments: high-viscosity packable composites and low-viscosity flowable composites. While there is still no consensus on the optimum shear bond strength required for attachments to enamel, previous studies have reported a range of values. High-viscosity composites generally exhibit higher shear bond strength compared to their low-viscosity counterparts.(26) This finding aligns with the study by Chen *et al.* in 2021, which evaluated the shear bond strength of these materials on extracted premolars. The study found that the flowable composite, Filtek™ Z350 XT Flowable, used with two-step total etch adhesive (Adper™ Single Bond 2 Adhesive), demonstrated a lower shear bond strength (15.3±2.33 MPa) compared to the packable composite, Z350 with the two-step total-etch adhesive (20.53±2.59 MPa).(24) Building on this gap, the present investigation aims to analyze three ion-releasing resin composites to compare their effect on caries inhibition in adjacent enamel and shear bond strength to enamel, in order to identify which material is more suitable for attachment reproduction in clear aligner treatment, with and without the use of an adhesive system. The null hypothesis tested was that different restorative materials with or without adhesive would not affect mineral loss and lesion depth on the contiguous enamel or the shear bond strength to enamel.

Materials and Methods

This study was approved by the Human Experimentation Committee, Faculty of Dentistry, Chiang Mai University, Thailand (NO.20/2023).

Materials

Three commercial ion-releasing resin composites: ACTIVA™ BioACTIVE-RESTORATIVE™ (Pulpdent, Massachusetts, USA), BEAUTIFIL Injectable X_{SL} (Shofu, Kyoto, Japan), Cention® N (Ivoclar Vivadent, Schaan, Liechtenstein) and a conventional nanofilled resin composite, Filtek™ Z350 XT Universal Restorative (3M ESPE, Minnesota, USA) were selected. The compositions and product instructions of experimental restorative materials and an adhesive system are shown in Table 1.

Specimen preparation

One hundred and forty-four sound human premolars, extracted for orthodontic and periodontal reasons, were used in this study. All teeth were free of caries, restorations, and dental anomalies. The exclusion criteria were teeth with crown defects, caries, cracks, or restorations. The teeth were immersed in a 0.1% thymol solution at room temperature and used within 3 months of storage.

Effect of caries inhibition in adjacent enamel

The teeth were randomly assigned for two experiments. Thirty-six premolars were sectioned at the cemento-enamel junction using a precision diamond saw (IsoMet™ 1000, Buehler, USA). The buccal sides with enamel surfaces facing up were embedded in self-curing acrylic resin. These surfaces were ground with 600 grit silicon carbide paper to create a flat surface and cleaned with deionized water in an ultrasonic cleaner (BioSonic® UC125: Whaledent Inc., USA) for 10 minutes. Preparations were made at buccal surface with 2 mm wide and 2 mm deep occlusogingivally. The specimens were assigned into six groups based on the restorative materials used: ACTIVA™ BioACTIVE-RESTORATIVE™ (A), BEAUTIFIL Injectable X_{SL} (B), Cention® N (C), and Filtek™ Z350 XT Universal Restorative (F), with Adper™ Single Bond 2 Adhesive (S) as the adhesive system. Activa and Cention® N can be used with or without an adhesive, while X_{SL} and Z350 requires the use of an adhesive. Therefore, the groups were categorized as follows: Group 1: A, Group 2: AS, Group 3: BS, Group 4: C, Group 5: CS, and Group 6: FS. The prepared cavities were filled with the materials according to the manufacturer's recommended directions (Table 1). Filled specimens were stored in deionized water at 37°C for 24 hours. The outer surface of all restored specimens was serially polished with silicon carbine papers of 600, 800, 1000 and 1500 grit to remove the excess and to define the boundary of prepared restoration before being cleaned in ultrasonic cleanser for 10 minutes. Each specimen surface was coated with nail varnish, leaving exposed only half of the restoration and 1 mm of the adjacent enamel beyond the margin on the occlusal side, to allow exposure to the demineralizing and remineralizing solutions. The sound tooth structure on the cervical side, protected by the nail varnish, served as the control within each specimen.

Table 1: The details of the materials and adhesive system.

Materials	Composition	Instructions
ACTIVA™ BioACTIVE-RESTORATIVE™ (Pulpdent, Massachusetts, USA) Lot 210618, shade A2* Lot 230404, shade A3**	Resin matrix: patented ionic resin matrix, shock-absorbing rubberized resin (diurethane and other methacrylates with modified polyacrylic acid 44.6%) Filler: reactive ionomer glass fillers (amorphous silica 6.7%, sodium fluoride 0.75 %, 55.4 wt% of bioactive glass and sodium fluoride)	- Dispense the material into the cavity through a spiral nozzle - Allow the material to self-cure for 20 seconds - Light-cured for 20 seconds
BEAUTIFIL Injectable X_{SL} (Shofu, Kyoto, Japan) Lot 052204, shade B1*,**	Resin matrix: bis-GMA, bis-MPEPP, TEGDMA Filler: F-B-Al-Si-glass (63.4 wt%, 41.7 v%, 0.1-0.8 µm, 0.4 mean) aluminofluoro-borosilicate glass, Al_2O_3, S-PRG filler	- Dispense the material into the cavity - Light-cured for 20 seconds
Cention® N (Ivoclar Vivadent, Schaan, Liechtenstein) Lot Z04YLR, shade A2*,**	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 v%, 0.1–35 µm) Powder/liquid ratio (g/g) = 4.6/1.0	- Mix the material with a powder-to-liquid ratio of 1:1 (mixing time: 40-60 seconds and setting time: 5 minutes) - Light-cured for 20 seconds
Filtek™ Z350 XT Universal Restorative (3M ESPE, Minnesota, USA) Lot 9712738, shade A1*,**	Resin matrix: bis-GMA, UDMA, TEGDMA, bis-EMA (6) resins Filler: non-agglomerated/non-aggregated 20 nm silica filler, non-agglomerated/non-aggregated 4-11 nm zirconia filler, and aggregated zirconia/silica cluster filler (comprised of 20 nm silica and 4-11 nm zirconia particles)	- Place the material into the cavity - Light-cured for 20 seconds
Scotchbond™ Etchant (3M ESPE, Minnesota, USA) Lot 9637865*,**	37% Phosphoric acid	- Apply on enamel for 20 seconds - Rinse thoroughly with water for 10 seconds - Air-dry gently for 2 seconds
Adper™ Single Bond 2 Adhesive (3M ESPE, Minnesota, USA) Lot 9753157*,**	bis-GMA, HEMA, dimethacylates, ethanol, water, photoinitiator, methacrylate functional copolymer of polyacrylic and poly (itaconic) acids, 5 nm spherical silica particles (10 wt%)	- Rub adhesive on enamel for 20 seconds to ensure thorough penetration - Air-dry gently for 5 seconds - Light-cured for 20 seconds

*lot used in the test for effect of caries inhibition in adjacent enamel, **lot used in shear bond strength test

(bis-GMA: bisphenol A-glycidyl methacrylate, DCP: dicalcium phosphate, DMA: dimethacrylate, HEMA: hydroxyethyl methacrylate, PEG: polyethylene glycol, S-PRG: surface pre-reacted glass-ionomer, TEGDMA: triethylene glycol dimethacrylate, UDMA: urethane dimethacrylate)

Artificial caries induction by pH-cycling

All specimens were subjected to 14 days of artificial caries induction or pH-cycling process. Each specimen was submerged in an 8 ml of demineralizing solution (2.2 mM of $CaCl_2$, 2.2 mM of KH_2PO_4 , 0.05 M of acetic acid; pH 4.4) for 6 hours and in an 8 ml of remineralizing solution (1.5 mM $CaCl_2$, 0.9 mM KH_2PO_4, 0.15 M KCl, 20 mM HEPES; pH 7.0) for 18 hours.[27] All specimens were carried out in an incubator at 37°C.

Mineral loss and lesion depth measurement

After 14 days of artificial caries induction, each specimen was perpendicularly sectioned through the buccal enamel. The illustration of the cross-sectional specimen, the area of measurements of mineral loss (ΔZ) and lesion depth (LD) are shown in Figure 1 and Figure 2. Both values were performed by a micro computed tomography (microCT35; SCANCO Medical AG, Switzerland) under standardized conditions of 70 kV voltage, 114 µA current,

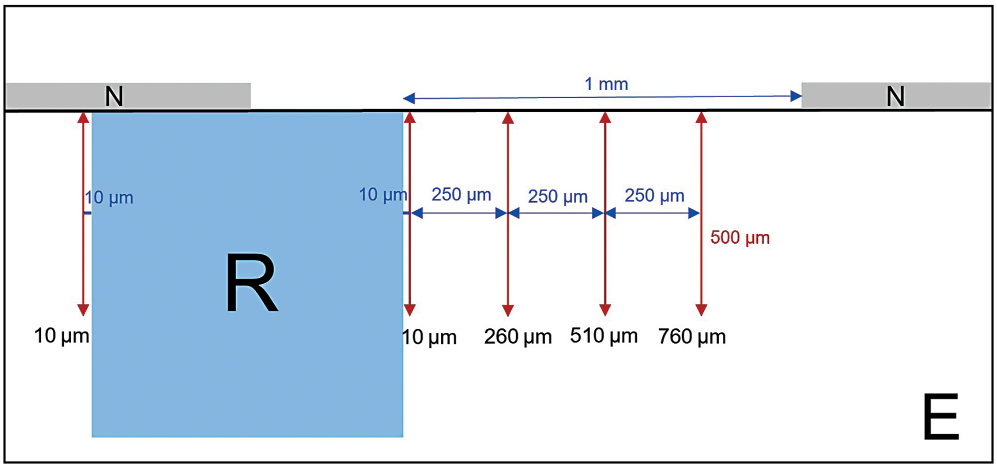

Figure 1: The cross-sectional specimen, along with the locations for mineral loss and lesion depth measurements. E: Enamel, N: Nail varnish, R: Restoration. (Modified from Kuphasuk *et al.,* 2022)

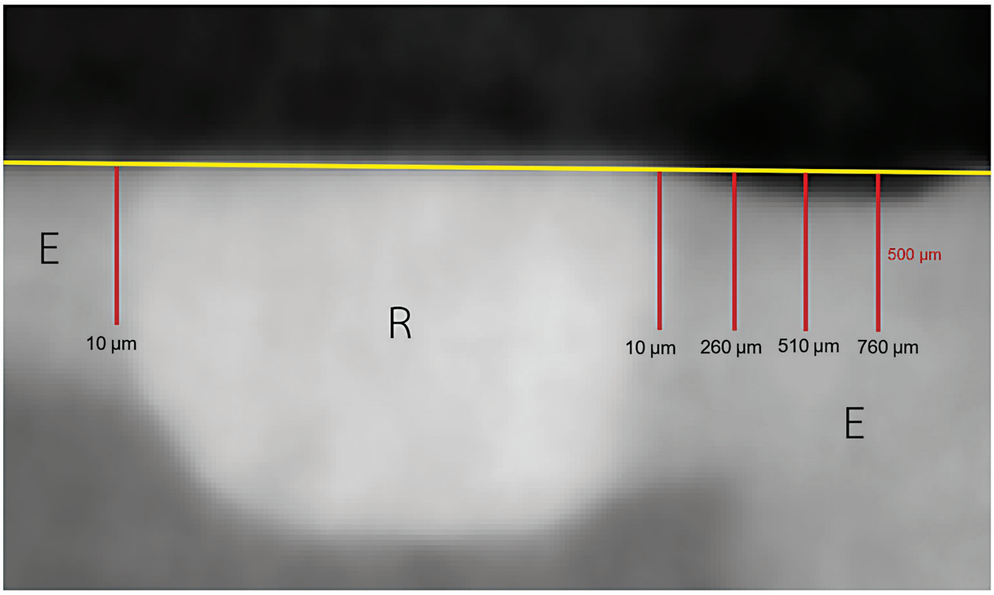

Figure 2: Cross-sectional specimen obtained from micro-CT imaging, along with the locations for mineral loss and lesion depth measurements. E: Enamel, N: Nail varnish, R: Restoration.

and 5 µm voxel dimensions. The radiolucency and radiopacity of the micro-CT images were calibrated against a phantom with known mineral density standards to obtain quantitative mineral profiles for each specimen. Mineral loss was calculated as the integrated difference in mineral volume between the demineralized region and sound enamel using an image processing program (Rasband, W.S., ImageJ, U.S. National Institutes of Health, USA). Lesion depth was determined as the distance where the mineral content dropped below 95% of the mineral density of sound enamel, indicating significant demineralization.[28] Both parameters were evaluated at four predefined distances from the restoration-enamel interface: 10 µm, 260 µm, 510 µm, and 760 µm, measured across a depth of 500 µm from the tooth surface.[29] The evaluation procedure, including the preparation of specimens and the pH cycling model, is summarized and illustrated in Figure 3.

Shear bond strength test

One hundred and eight premolars were sectioned at the cemento-enamel junction using a precision diamond saw. The buccal sides with the enamel surface facing up were embedded in self-curing acrylic resin (18 mm diameter, 10 mm height). The enamel surfaces were

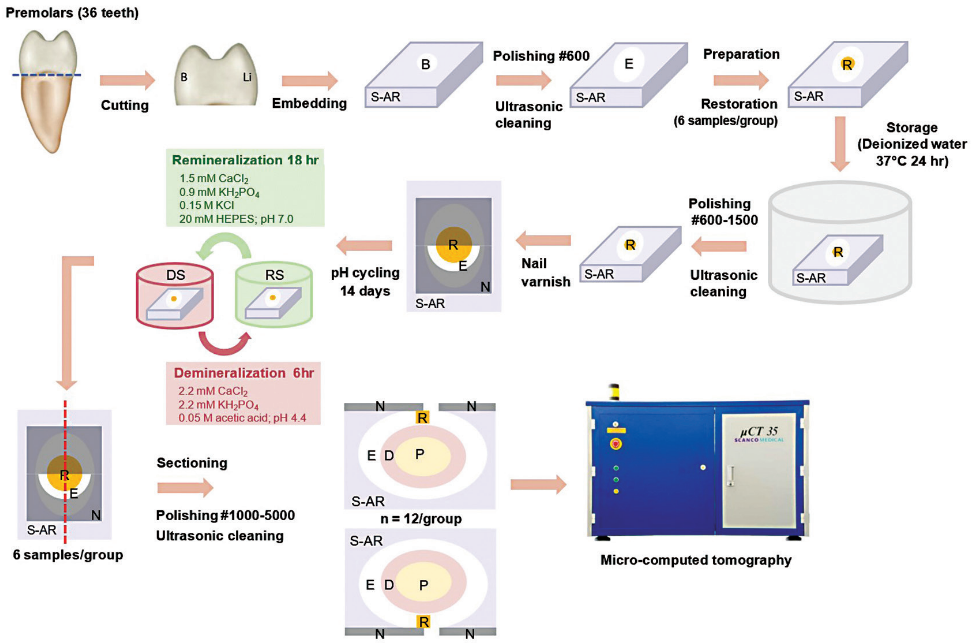

Figure 3: The procedures for specimen preparation to evaluate the effect of ion releasing resin composite material on caries-inhibition potential in conjunction with pH cycling. B: buccal, D: dentin, DS: demineralizing solution, E: enamel, Li: lingual, N: nail varnish, P: pulp, RS: remineralizing solution, S-AR: self-curing acrylic resin.

ground flat with 600-grit silicon carbide paper and cleaned in an ultrasonic cleanser for 10 minutes. A polyethylene tube (1.8 mm internal diameter, 2 mm height) was placed on each surface and filled with different restorative materials per group. All specimens were stored in deionized water at 37°C for 24 hours and inspected for defects under the stereomicroscope. Each group was divided into two subgroups (n=9): one subgroup underwent 10,000 thermocycling cycles, and the other was assigned as non-thermocycling. Shear bond strength was tested with the Instron® 5566 universal testing machine using a knife edge chisel at a cross-head speed of 1 mm/min. Failure mode analysis was performed with a stereomicroscope and digital camera, categorizing failure modes into four types:[30]

- Type 1: Adhesive failure (over 80% at the restoration-enamel interface)
- Type 2: Mixed failure (combination of adhesive failure at the interface and cohesive failure in restoration and/or enamel)
- Type 3: Cohesive failure in enamel (over 80% in the underlying enamel)
- Type 4: Cohesive failure in restoration (over 80% in the adhesive resin and/or restoration)

The procedures for preparing specimens to evaluate the shear bond strength of ion-releasing resin composite to enamel, are summarized and illustrated in Figure 4.

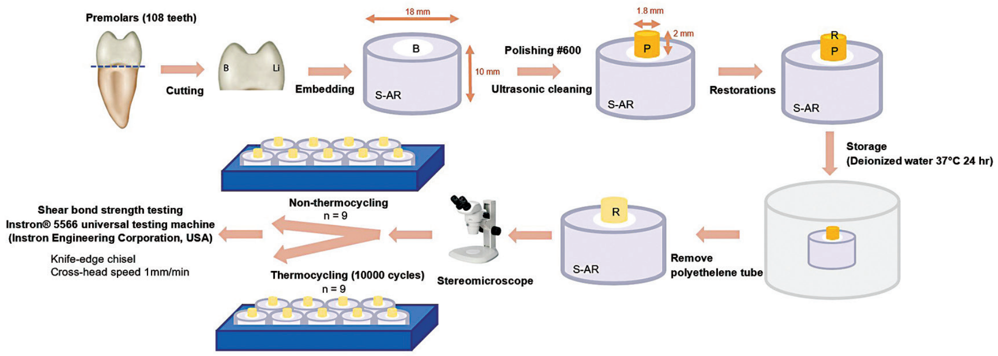

Figure 4: The procedures of specimen preparation to evaluate the effect of ion-releasing resin composite material on shear bond strength to enamel. B: buccal, Li: lingual, P: polyethylene tube, R: restoration, S-AR: self-curing acrylic resin.

Table 2: Means and standard deviations of mineral loss (mgHAP/m^2) at different distances for each group after 14 days of artificial caries induction. Different superscript lowercase letters indicate significant differences within the same row (p<0.05). Different superscript uppercase letters indicate significant differences within the same column (p<0.05).

Groups			Column			
Materials		code	1 (10 µm) (gHAP/m^2)	2 (260 µm) (gHAP/m^2)	3 (510 µm) (gHAP/m^2)	4 (760 µm) (gHAP/m^2)
ACTIVA™ BioACTIVE-RESTORATIVE™	no bonding	A	19.37±2.66^{aA}	104.69±17.25^{bA}	130.13±18.72^{cA}	162.91±18.98^{dA}
	with two-step total etch	AS	48.59±3.22^{aBC}	143.50±13.25^{bBC}	189.10±19.38^{cBC}	221.39±16.73^{dB}
BEAUTIFIL Injectable X_{SL}	with two-step total etch	BS	73.09±5.51^{aD}	125.83±14.12^{bB}	172.44±9.63^{cB}	207.96±11.71^{dB}
Cention® N	no bonding	C	23.21±4.51^{aA}	150.45±15.31^{bCD}	195.33±18.30^{cCD}	249.23±11.79^{dC}
	with two-step total etch	CS	38.16±2.90^{aB}	172.39±18.04^{bDE}	216.19±14.63^{cDE}	255.05±10.74^{dC}
Filtek™ Z350 XT Universal Restorative	with two-step total etch	FS	113.79±19.73^{aE}	223.74±7.22^{bF}	250.21±10.29^{cF}	301.34±15.08^{dD}

Table 3: Means and standard deviations of lesion depth (µm) at different distances for each group after 14 days of artificial caries induction. Different superscript lowercase letters indicate significant differences within the same row (p<0.05). Different superscript uppercase letters indicate significant differences within the same column (p<0.05).

Groups			Column			
Materials		code	1 (10 µm) (µm)	2 (260 µm) (µm)	3 (510 µm) (µm)	4 (760 µm) (µm)
ACTIVA™ BioACTIVE-RESTORATIVE™	no bonding	A	39.39±6.02^{aAB}	162.75±27.06^{bA}	215.29±30.31^{cA}	261.5±30.25^{dA}
	with two-step total etch	AS	63.73±7.76^{aC}	222.42±26.85^{bB}	291.73±21.07^{cBC}	321.62±27.42^{dB}
BEAUTIFIL Injectable X_{SL}	with two-step total etch	BS	120.72±21.69aDE	226.59±28.35^{bB}	278.52±30.58^{cB}	307.53±20.98^{dB}
Cention® N	no bonding	C	37.01±8.74^{aA}	222.7±18.07^{bB}	304.14±11.96^{cBCD}	375.26±41.51^{dC}
	with two-step total etch	CS	60.89±0.25^{aBC}	251.87±34.33^{bBC}	321.03±21.08^{cCD}	370.63±28.06^{dC}
Filtek™ Z350 XT Universal Restorative	with two-step total etch	FS	168.54±26.87^{aF}	324.73±20.23^{bD}	366.35±23.9^{cE}	423.61±22.1^{dD}

Statistical analysis

Statistical analysis was conducted using IBM SPSS Statistics Version 25 (IBM Corporation, Armonk, NY, USA) at a 95% confidence level. Normality and homogeneity tests were performed to confirm normal distribution and homogeneity of variances. Repeated measures ANOVA and pairwise comparison were used to compare mineral loss and lesion depth within group, while One-way ANOVA with Tukey's post hoc test for comparisons among groups (p<0.05). Shear bond strength was analyzed using two-way ANOVA and Tukey's multiple comparison test (p<0.05).

Result

Effect of caries inhibition in adjacent enamel

Mineral loss and lesion depth values of the adjacent enamel at various distances for each group are detailed in Table 2 and Table 3. X-ray images obtained from the micro-CT machine showing the enamel adjacent to each restoration group are presented in Figure 5. Within the same sample, every group exhibited significant differences in mineral loss and lesion depth values at different distances from the restoration-enamel interface (p<0.05). When comparing mineral loss and lesion depth values among groups at a distance of 10 µm, group A showed the

Table 4: Means and standard deviations of shear bond strength (MPa) to enamel before and after thermocycling 10000 cycles and percentage of each mode of failure. Superscript uppercase letters indicate significant differences between different storage conditions within each material tested. Asterisk (*) indicates significant differences between groups within the same material (*p*<0.05).

Materials		Thermocycling	code	SBS (MPa)
ACTIVA™ BioACTIVE-RESTORATIVE™	no bonding	24 hours	A-t_0	14.10 ± 2.03^{D}
		10,000 thermocycles	A-t_1	$7.10 \pm 0.80^{E*}$
	with two-step total etch	24 hours	AS-t_0	15.13 ± 1.76^{D}
		10,000 thermocycles	AS-t_1	13.99 ± 1.74^{D}
BEAUTIFIL Injectable X_{SL}	with two-step total etch	24 hours	BS-t_0	17.90 ± 1.45^{BC}
		10,000 thermocycles	BS-t_1	16.38 ± 1.54^{CD}
Cention® N	no bonding	24 hours	C-t_0	7.52 ± 1.19^{E}
		10,000 thermocycles	C-t_1	$4.08 \pm 0.51^{F*}$
	with two-step total etch	24 hours	CS-t_0	21.68 ± 1.86^{A}
		10,000 thermocycles	CS-t_1	21.17 ± 2.40^{A}
Filtek™ Z350 XT Universal Restorative	with two-step total etch	24 hours	FS-t_0	20.30 ± 1.85^{AB}
		10,000 thermocycles	FS-t_1	19.16 ± 2.29^{AB}

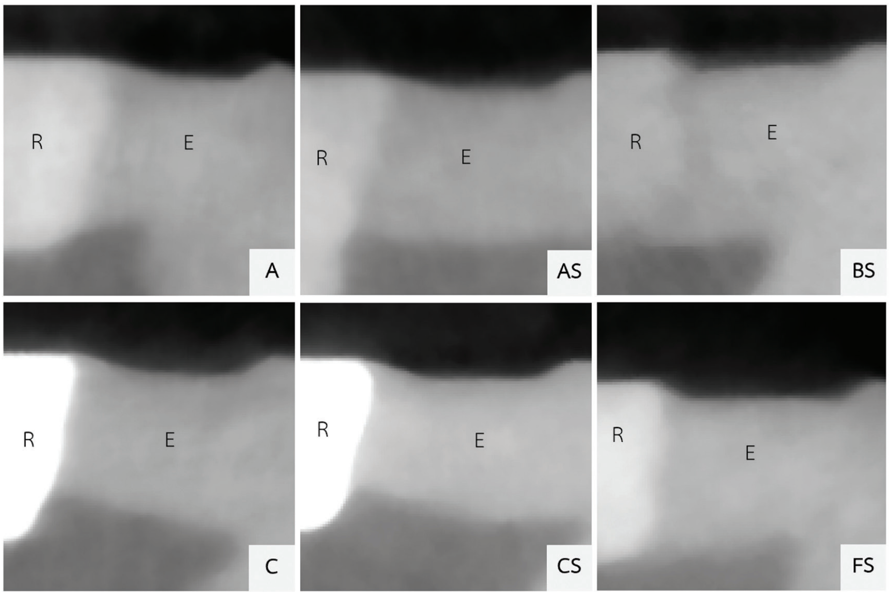

Figure 5: X-ray images obtained from the micro-CT machine show the enamel adjacent to each restoration group after 14 days of pH cycling. A: ACTIVA™ BioACTIVE-RESTORATIVE™, AS: ACTIVA™ BioACTIVE-RESTORATIVE™ with two-step total etch, BS: BEAUTIFIL Injectable XSL with two-step total etch, C: Cention® N, CS: Cention® N with two-step total etch, E: enamel, FS: Filtek™ Z350 XT with two-step total etch, R: restoration.

lowest value and significant differences compared to the other groups, except for the group C. Group FS exhibited the highest, showing significant differences compared to the other groups (*p*<0.05). At distances of 260, 510 and 760 µm, group A remained the lowest and Group FS remained the highest in mineral loss and lesion depth value and showed significant differences compared to the other groups.

Shear bond strength test

The comparison of shear bond strength values between groups as shown in Table 4 revealed that Cention® N with an adhesive system had the highest values for both thermocycling and non-thermocycling conditions, which was significantly higher than other groups except that of

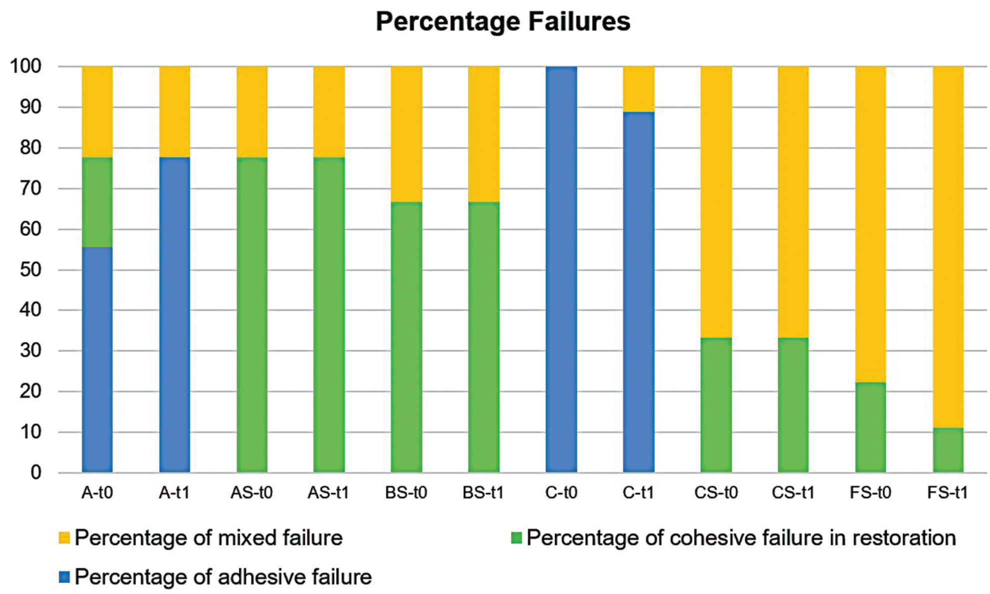

Figure 6: Percentage of failure mode.

the conventional resin composite with an adhesive system. Conversely, Cention® N without an adhesive system had the lowest values, significantly different from all other groups (p<0.05).

When considering the shear bond strength within the group, by comparing conditions with and without thermocycling, it was found that the group subjected to 10,000 cycles of thermocycling exhibited lower shear bond strength than the group that did not undergo thermocycling. The lower value was statistically significant in the group without an adhesive system (p<0.05). Whereas in the group with adhesive system, although a lower value of shear bond strength was observed, the difference was not statistically significant (p>0.05).

Failure mode analysis (Figure 6) showed that adhesive failure was predominant in the groups without an adhesive system. Cohesive failure within the restoration was predominantly observed in group AS and BS, whereas mixed failure was primarily noted in group CS and FS.

Discussion

Composite attachments serve as a component that bonds to the tooth surface, working in conjunction with clear aligners; it increases the surface area in contact with the aligners, allowing for better control of tooth movement in the desired direction.[(3)] Therefore, selection of composite attachment plays a crucial role for the long-term stability of the attachments' shape and for their structural integrity.

Ion-releasing resin composites signify a notable advancement in dental restorative materials. These innovative composites are designed to restore the structural integrity of teeth while actively preventing secondary caries through the release of therapeutic ions.[(31)]

The caries inhibition effects of restorative materials manifest in two distinct forms based on the alterations in tooth structure. The first form is the caries inhibition zone, which develops adjacent to fluoride-releasing materials due to ion infiltration and fluoride accumulation, enhancing the acid resistance in that area.[(32,33)] The second form is the acid-base resistant zone beneath the hybrid layer, which shows greater resistance to environmental acids and bases than normal tooth structure.[(34,35)] Previous studies have demonstrated that 10-MDP in primers or bonding agents forms stable, low-solubility salts with calcium in hydroxyapatite, which are crucial for creating an acid-base resistant zone in the enamel.[(36,37)]

When considering the lesion depth of enamel columns at a distance of 10 µm from the restoration-enamel interface, it was found that groups A, AS, C, and CS exhibited lesion depths of less than 100 µm (Table 3). In contrast, groups BS and FS exhibited lesion depths of more than 100 µm at all distances. Studies have shown that the lesion depth of initial caries ranges from 100 to 500 µm.[(19,38)] This indicates that at 14 days post-caries simulation, the use of Activa and Cention® N with and without adhesive demonstrated the ability to inhibit caries formation in columns 10 μm from the restoration-enamel interface. According to the study by Ruengrungsom *et al.,* Cention® N exhibited a higher and more substantial fluoride ion release compared to Activa.[8] The results align with the trend observed in recent studies.[11] This is due to the fluoride-containing fillers in Cention® N, which release fluoride ions in deionized water, whereas bioactive glass typically releases fluoride ions under acidic conditions.[8,11] Additionally, Cention® N was found to release a higher amount of calcium ions than Activa, attributable to its composition of calcium fluorosilicate glass or bioactive glass-like phase, and calcium barium aluminum fluorosilicate glass or ionomer glass-like phase, similar to those in glass ionomer cements. Activa, on the other hand, contains only the ionomer phase. However, Activa was found to release a high amount of phosphate ions, due to its phosphate-containing fillers.[8]

The caries-inhibitory potential of ion-releasing restorative materials is primarily linked to their ability to release beneficial ions, with fluoride playing a crucial role in this process.[39,40] Studies indicate that Cention® N releases significantly greater quantities of fluoride and calcium ions compared to conventional resin composites, with concentrations approximately 300-400 times higher.[8,11,41] This enhanced ion release is associated with a notable reduction in Streptococcus mutans colonization, a key contributor to biofilm formation and the progression of carious lesions.[42] This substantial ion release may account for the reduced lesion depth and lesser demineralization at all depths, compared to the control resin composite.[43] Conversely, Activa has been shown to release minimal amounts of calcium and relatively low concentrations of fluoride, which are unlikely to significantly reduce biofilm formation.[8,11,42]

Giomer incorporates pre-reacted glass (PRG) filler technology, utilizing ionomer-like fluorosilicate glasses that pre-reacted with polyacrylic acid and are dispersed in the resin matrix.[44] Fluoride release in giomers occurs through water diffusion. When bioactive glass containing fluoride is included ion-releasing resin composite, the bioactive glass functions as an ion source, releasing ions such as Ca, P, and F. This process is initiated upon contact with fluid, even under neutral pH conditions, enabling controlled fluoride release.[11] The level of fluoride release from ion-releasing resin composite is affected by the hydrophilic and acidic properties of their resin matrices.[45] However, PRG fillers combined with bis-GMA/TEGDMA resin have shown a lower capacity to facilitate fluoride diffusion in deionized water compared to other materials.[8]

When comparing the use of an adhesive system with the same restorative material, the micro-CT evaluation revealed that at a distance of 10 μm from the restoration-enamel interface, group A exhibited significantly less mineral loss than group AS. Similarly, group C showed significantly less mineral loss than group CS (Table 2). These findings align with several studies that have reported that the presence of an adhesive layer can interfere the release of fluoride ions from fluoride-releasing resin composites.[46,47] This interference occurs because fluoride release is facilitated by the infiltration and diffusion of water into the material. Therefore, when an adhesive is used, water must first penetrate the adhesive layer. A study by Burrow *et al.,* found that physical properties such as water sorption and desorption of the adhesive are crucial factors in controlling fluoride release.[48]

Analysis of mineral loss and lesion depth in enamel for the same restorative material group, at varying distances from the restoration-enamel interface, revealed that all groups exhibited a gradient of increasing mineral loss and lesion depth from column 1 to column 4 (Table 2 and Table 3). Each column showed statistically significant differences, indicating that enamel closer to the restoration-enamel interface had less mineral loss and shallower lesion depths than enamel further away from the interface. This effect is attributed to higher fluoride exposure near the restorative material, consistent with multiple studies reporting that the tooth structure near fluoride-releasing restorative materials had mineral density and surface hardness similar to normal tooth structure, and higher than tooth structure further from the restoration-enamel interface.[29,49] The ion-releasing mechanisms of the experimental restorative materials vary significantly. Activa releases fluoride, calcium, and phosphate ions through an acid-base reaction, similar to glass ionomers, and promotes remineralization via its polyacrylic acid component.[8] Cention® N releases fluoride, calcium, and hydroxyl ions primarily under acidic conditions, aiding in remineralization and neutralizing oral acids. It achieves this through its unique alkaline fillers.[8,15,16] X_{SL} features S-PRG fillers that release fluoride and other ions. These differences in ion releasing behavior directly influence the gradient of mineral loss observed in the enamel with materials that have more active and targeted ion release, such as Activa and Cention® N, showing localized effects near the restoration-enamel interface and leading to reduced lesion depth and mineral loss in adjacent enamel.

Selecting an attachment material with optimal bonding properties to the enamel surface is crucial for the success of clear aligner orthodontic treatment. The effectiveness of clear aligners in moving teeth to the desired positions depends on the stability of the attachment bond.[50] If the attachment bond is insufficient, the attachments may detach, resulting in poor control of tooth movement and prolonged treatment duration.[51]

The analysis of shear bond strength values to enamel indicated that ion-releasing resin composites exhibited significantly lower shear bond strength compared to conventional resin composites. However, Cention® N, when used with an adhesive system, demonstrated no significant difference in shear bond strength compared to the conventional resin composite group in both thermocycling and non-thermocycling conditions (Table 4). One key factor contributing to the differences in shear bond strength among various materials is the amount of inorganic filler. Higher inorganic filler content results in lower polymerization shrinkage, thereby enhancing bond strength.[52] Specifically, the inorganic filler content is 78.5% by weight for Z350, 78.4% for Cention® N, 63.4% for X_{SL}, and 55.4% for Activa. The higher filler content in Z350 and Cention® N contributes to their improved bond strength due to reduced polymerization shrinkage, compared to X_{SL} and Activa.

Activa exhibits bend before failure due to its low flexural modulus.[53] A low flexural modulus leads to high distortion, an undesirable property in materials.[54] This can affect material strength and cause uneven stress distribution from chewing forces.[53,54] This study found that using Activa with an adhesive resulted in cohesive failure within the composite resin layer during shear bond strength tests. In contrast, groups using Cention® N and Z350 exhibited mixed failure modes. This difference is likely due to the higher mechanical strength and shear bond strength of Cention® N and the conventional resin, resulting in a combination of cohesive failure within the composite resin layer and adhesive failure at the bonding interface. Across all experimental groups, the observed failure modes included adhesive failure, mixed failure, and cohesive failure within the composite resin layer. Notably, no cohesive failures were observed within the enamel layer, indicating that the enamel remained intact. This finding highlights the safety and clinical suitability of the adhesives and restorative materials evaluated in this study for use in attachment bonding in clear aligner therapy.

Factors such as depth of cure (DoC) and Knoop microhardness also impact the bond strength to tooth structure. A study by Daabash *et al.,* found that Cention® N has a greater DoC than Activa and Z350.[54] Cention® N initiates polymerization with both chemical and light activation, aided by Ivocerin™ and acyl phosphine oxide, which absorb visible light between 370 and 460 nm, and its high translucency enhances light transmission. In contrast, despite manufacturer claims, Activa, with a stated DoC of 4 mm and a combination of acid-base and photopolymerization reactions, showed no DoC enhancement after 24 hours, indicating a less effective chemical cure compared to Cention® N.[54] This aligns with a study by Hughes *et al.,* which reported Activa's limited self-curing ability despite being marketed as a dual-cure material.[55]

Selecting restorative materials for orthodontic attachments requires balancing adequate bond strength and the added benefit of caries inhibition. The study results indicate that Cention® N, with its high filler content and fluoride release, excels in both bond strength and caries inhibition, although its hand-mixing requirement can be a downside. Z350 also demonstrates superior mechanical properties, making it a reliable choice for durable attachments. X_{SL} exhibited a lower shear bond strength than Cention® N, comparable to Z350, and higher than Activa, but lacks caries inhibition effects, similar to Z350. Activa, while beneficial for its caries inhibition effect, shows limitations in mechanical strength and shear bond strength. Notably, all materials showed significantly lower shear bond strength without an adhesive system, which is insufficient to withstand the insertion and removal of clear aligner trays. These insights guide the selection of materials to improve the efficacy and safety of clear aligner treatments.

In vitro pH cycling models remain widely used as they simulate daily pH changes in the oral cavity and mimic the dynamic processes of mineral changes associated with caries formation.[27] The duration of the demineralization phase was regulated to replicate both high- and low-cariogenic pH cycling scenarios, as demonstrated in Wierich's study.[56] Longer demineralization process are more representative of individuals at high caries risk, where the oral environment remains predominantly in a state of demineralization.[56,57] A six-hour demineralization period is commonly employed in in vitro studies to replicate the acidic exposure that occurs during and after meals, particularly in high-cariogenic environments.[29,58,59] This duration reflects typical acid challenge scenarios within the oral cavity and provides a controlled framework for evaluating the material's response under conditions that mimic real-life clinical situations.[57] Moreover, after 14 days of pH-cycling, the formation of demineralized lesions was the result of a continuous process involving both demineralization and remineralization. The ion-releasing resin composite demonstrated reduced lesion depth and mineral loss compared to the conventional resin composite.

The limitations of this *in vitro* study include its inability to fully replicate the complexity of *in vivo* conditions, such as the ionic composition of dietary foods and drinks, intraoral pH fluctuations, and the presence of salivary enzymes.[60,61] While this study used a controlled environment to isolate and assess specific factors, future investigations should incorporate biological elements such as salivary proteins and enzymes, which play significant roles in remineralization.[62] While the ion-releasing effects of the materials tested are likely influenced by interactions with both tooth structure and the surrounding media, the controlled environment employed in this study provides a close approximation of the material's behavior in clinical scenarios. Future studies should develop experimental models that better simulate the oral environment.

Conclusions

The null hypothesis of this study was rejected. The results of this *in vitro* study indicated that Cention® N, when used with an adhesive system, provides the optimal combination of shear bond strength and caries inhibition effect for the clear aligner attachments.

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Conflicts of Interest

The authors declare no conflict of interest.

References

1. Jamilian A, Kiaee B, Sanayei S, Khosravi S, Perillo L. Orthodontic treatment of malocclusion and its impact on oral health-related quality of life. Open Dent J. 2016;10:236-41.
2. Morton J, Derakhshan M, Kaza S, Li C, Chen V. Design of the invisalign system performance. Semin Orthod. 2016;23(1):3-11.
3. Jedliński M, Mazur M, Greco M, Belfus J, Grocholewicz K, Janiszewska-Olszowska J. Attachments for the orthodontic aligner treatment-atate of the art-a comprehensive systematic review. Int J Environ Res Public Health. 2023;20(5):4481.
4. Feinberg KB, Souccar NM, Kau CH, Oster RA, Lawson NC. Translucency, stain resistance, and hardness of composites used for invisalign attachments. J Clin Orthod. 2016;50(3):170-6.
5. Barreda GJ, Dzierewianko EA, Muñoz KA, Piccoli GI. Surface wear of resin composites used for Invisalign® attachments. Acta Odontol Latinoam. 2017;30(2):90-5.
6. Kravitz ND, Kusnoto B, Agran B, Viana G. Influence of attachments and interproximal reduction on the accuracy of canine rotation with invisalign. a prospective clinical study. Angle Orthod. 2008;78(4):682-7.
7. Julien KC, Buschang PH, Campbell PM. Prevalence of white spot lesion formation during orthodontic treatment. Angle Orthod. 2013;83(4):641-7.
8. 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.
9. Persson A, Lingstrom P, van Dijken JW. Effect of a hydroxyl ion-releasing composite resin on plaque acidogenicity. Caries Res. 2005;39(3):201-6.
10. Porenczuk A, Jankiewicz B, Naurecka M, Bartosewicz B, Sierakowski B, Gozdowski D, *et al*. A comparison of the remineralizing potential of dental restorative materials by analyzing their fluoride release profiles. Adv Clin Exp Med. 2019;28(6):815-23.
11. Tiskaya M, Al-Eesa NA, Wong FSL, Hill RG. Characterization of the bioactivity of two commercial composites. Dent Mater. 2019;35(12):1757-68.
12. Rifai H, Qasim S, Mahdi S, Lambert MJ, Zarazir R, Amenta F, *et al*. *In vitro* evaluation of the shear bond strength and fluoride release of a new bioactive dental composite material. J Clin Exp Dent. 2022;14(1):e55-e63.
13. Ltd. SDA-PP. BEAUTIFIL Injectable X : The one true bioactive injectable restorative [Internet]. 2021 [Available from:

https://www.shofu.com.sg/wp-content/uploads/2021/06/BInjectableX-ref-1.pdf.
14. Bai X, Chen Y, Zhou T, Pow EHN, Tsoi JKH. The chemical and optical stability evaluation of injectable restorative materials under wet challenge. J Dent. 2024;146:105031.
15. Ilie N. Comparative effect of self- or dual-curing on polymerization kinetics and mechanical properties in a novel, dental-resin-based composite with alkaline filler. running title: resin-composites with alkaline fillers. Materials (Basel). 2018;11(1):108.
16. 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. 2019;22(3):296-9.
17. Francois P, Fouquet V, Attal JP, Dursun E. Commercially available fluoride-releasing restorative materials: a review and a proposal for classification. Materials (Basel). 2020;13(10):2313.
18. Witty D, Kumaran P, Varma B, J SK, Xavier AM, Venugopal M, *et al*. Effect of prolonged water aging on the bond strength and marginal seal of three novel restorative materials. J Contemp Dent Pract. 2023;24(9):632-7.
19. Huang TT, He LH, Darendeliler MA, Swain MV. Correlation of mineral density and elastic modulus of natural enamel white spot lesions using x-ray microtomography and nanoindentation. Acta Biomater. 2010;6(12):4553-9.
20. Cochrane NJ, Cai F, Huq NL, Burrow MF, Reynolds EC. New approaches to enhanced remineralization of tooth enamel. J Dent Res. 2010;89(11):1187-97.
21. Pires PM, Santos TPD, Fonseca-Gonçalves A, Pithon MM, Lopes RT, Neves AA. A dual energy micro-CT methodology for visualization and quantification of biofilm formation and dentin demineralization. Arch Oral Biol. 2018;85:10-5. doi: 10.1016/j.archoralbio.2017.09.034.
22. Mahoney E, Ismail FS, Kilpatrick N, Swain M. Mechanical properties across hypomineralized/hypoplastic enamel of first permanent molar teeth. Eur J Oral Sci. 2004;112(6): 497-502.
23. Swain MV, Xue J. State of the art of micro-CT applications in dental research. Int J Oral Sci. 2009;1(4):177-88.
24. Chen W, Qian L, Qian Y, Zhang Z, Wen X. Comparative study of three composite materials in bonding attachments for clear aligners. Orthod Craniofac Res. 2021;24(4):520-7.
25. Kircelli BH, Kilinc DD, Karaman A, Sadry S, Gonul EY, Gögen H. Comparison of the bond strength of five different composites used in the production of clear aligner attachments. J Stomatol Oral Maxillofac Surg. 2023;124(6):101481.
26. Alsaud BA, Hajjaj MS, Masoud AI, Abou Neel EA, Abuelenain DA, Linjawi AI. Bonding of clear aligner composite attachments to ceramic materials: an *in vitro* study. Materials (Basel). 2022;15(12):4145.
27. ten Cate JM, Duijsters PP. Alternating demineralization and remineralization of artificial enamel lesions. Caries Res. 1982;16(3):201-10.
28. Arends J, ten Bosch JJ. Demineralization and remineralization evaluation techniques. J Dent Res. 1992;71 Spec No: 924-8.
29. Kuphasuk S, Kunawarote S. *In vitro* caries inhibition in enamel adjacent to ion-releasing resin composite. CM Dent J. 2022;43(2):50-61.
30. Thitthaweerat S, Klinklao K, Senawongse P. Effect of modified smear layer on the bond strength of all-in-one adhesives to dentin. M Dent J. 2018;38(1):11-21.
31. Dionysopoulos D, Koliniotou-Koumpia E, Helvatzoglou-Antoniades M, Kotsanos N. *In vitro* inhibition of enamel demineralisation by fluoride-releasing restorative materials and dental adhesives. Oral Health Prev Dent. 2016;14(4):371-80.
32. Nagamine M, Itota T, Torii Y, Irie M, Staninec M, Inoue K. Effect of resin-modified glass ionomer cements on secondary caries. Am J Dent. 1997;10(4):173-8.
33. Itota T, Nakabo S, Iwai Y, Konishi N, Nagamine M, Torii Y, *et al*. Effect of adhesives on the inhibition of secondary caries around compomer restorations. Oper Dent. 2001;26(5):445-50.
34. Nikaido T, Takahashi R, Ariyoshi M, Sadr A, Tagami J. Protection and reinforcement of tooth structures by dental coating materials. Coatings. 2012;2(4):210-20.
35. Nikaido T, Weerasinghe DD, Waidyasekera K, Inoue G, Foxton RM, Tagami J. Assessment of the nanostructure of acid-base resistant zone by the application of all-in-one adhesive systems: super dentin formation. Biomed Mater Eng. 2009;19(2-3):163-71.
36. Li N, Nikaido T, Takagaki T, Sadr A, Makishi P, Chen J, *et al*. The role of functional monomers in bonding to enamel: acid-base resistant zone and bonding performance. J Dent. 2010;38(9):722-30.
37. Nikaido T, Ichikawa C, Li N, Takagaki T, Sadr A, Yoshida Y, *et al*. Effect of functional monomers in all-in-one adhesive aystems on formation of enamel/dentin acid-base resistant zone. Dent Mater J. 2011;30(5):576-82.
38. Cochrane NJ, Anderson P, Davis GR, Adams GG, Stacey MA, Reynolds EC. An x-ray microtomographic study of natural white-spot enamel lesions. J Dent Res. 2012;91(2): 185-91.
39. Donly KJ, Segura A. Fluoride release and caries inhibition associated with a resin-modified glass-ionomer cement at varying fluoride loading doses. Am J Dent. 2002;15(1):8-10.
40. Kozai K, Suzuki J, Okada M, Nagasaka N. *In vitro* study of antibacterial and antiadhesive activities of fluoride-containing light-cured fissure sealants and a glass ionomer liner/base against oral bacteria. ASDC J Dent Child. 2000;67(2):117-22, 82-3.
41. Di Lauro A, Di Duca F, Montuori P, Dal Piva AMO, Tribst JPM, Borges ALS, *et al*. Fluoride and calcium release from alkasite and glass ionomer restorative dental materials: *in*

vitro study. J Funct Biomater. 2023;14(2):109.

42. Daabash R, Alqahtani MQ, Price RB, Alshabib A, Niazy A, Alshaafi MM. Surface properties and *Streptococcus mutans* biofilm adhesion of ion-releasing resin-based composite materials. J Dent. 2023;134:104549.
43. Albelasy EH, Chen R, Fok A, Montasser M, Hamama HH, Mahmoud SH, *et al*. Inhibition of caries around restoration by ion-releasing restorative materials: an *in vitro* optical coherence tomography and micro-computed tomography evaluation. Materials (Basel). 2023;16(16):5558.
44. Colceriu Burtea L, Prejmerean C, Prodan D, Baldea I, Vlassa M, Filip M, *et al*. New pre-reacted glass containing dental composites (giomers) with improved fluoride release and biocompatibility. Materials (Basel). 2019;12(23):4021.
45. Asmussen E, Peutzfeldt A. Long-term fluoride release from a glass ionomer cement, a compomer, and from experimental resin composites. Acta Odontol Scand. 2002;60(2):93-7.
46. Itota T, Nakabo S, Narukami T, Tashiro Y, Torii Y, McCabe JF, *et al*. Effect of two-step adhesive systems on inhibition of secondary caries around fluoride-releasing resin composite restorations in root dentine. J Dent. 2005;33(2):147-54.
47. Vercruysse CW, De Maeyer EA, Verbeeck RM. Fluoride release of polyacid-modified composite resins with and without bonding agents. Dent Mater. 2001;17(4):354-8.
48. Burrow MF, Inokoshi S, Tagami J. Water sorption of several bonding resins. Am J Dent. 1999;12(6):295-8.
49. Serra MC, Cury JA. The *in vitro* effect of glass-ionomer cement restoration on enamel subjected to a demineralization and remineralization model. Quintessence Int. 1992;23(2):143-7.
50. Rossini G, Parrini S, Castroflorio T, Deregibus A, Debernardi CL. Efficacy of clear aligners in controlling orthodontic tooth movement: a systematic review. Angle Orthod. 2015;85(5):881-9.
51. Kravitz ND, Kusnoto B, BeGole E, Obrez A, Agran B. How well does invisalign work? a prospective clinical study evaluating the efficacy of tooth movement with invisalign. Am J Orthod Dentofacial Orthop. 2009;135(1):27-35.
52. Pereira R, Lima D, Giorgi MCC, Marchi GM, Aguiar FHB. Evaluation of bond strength, nanoleakage, and marginal adaptation of bulk-fill composites submitted to thermomechanical aging. J Adhes Dent. 2019;21(3):255-64.
53. Ruengrungsom C, Burrow MF, Parashos P, Palamara JEA. Comprehensive characterisation of flexural mechanical properties and a new classification for porosity of 11 contemporary ion-leaching dental restorative materials. J Mech Behav Biomed Mater. 2021;121:104615.
54. Daabash R, Alshabib A, Alqahtani MQ, Price RB, Silikas N, Alshaafi MM. Ion releasing direct restorative materials: key mechanical properties and wear. Dent Mater. 2022;38(12):1866-77.
55. Hughes KO, Powell KJ, Hill AE, Tantbirojn D, Versluis A. Delayed photoactivation of dual-cure composites: effect on cuspal flexure, depth-of-cure, and mechanical properties. Oper Dent. 2019;44(2):e97-e104.
56. Wierichs RJ, Rupp K, Meyer-Lueckel H, Apel C, Esteves-Oliveira M. Effects of dentifrices differing in fluoride content on remineralization characteristics of dentin *in vitro*. Caries Res. 2020;54(1):75-86.
57. Fu Y, Ekambaram M, Li KC, Zhang Y, Cooper PR, Mei ML. *In vitro* models used in cariology mineralisation research-a review of the literature. Dent J (Basel). 2024;12(10):323.
58. Chen H, Zhang J, Hill R, Baysan A. Evaluation of toothpastes for treating root carious lesions - a laboratory-based pilot study. BMC Oral Health. 2024;24(1):484.
59. Takagi S, Liao H, Chow LC. Effect of tooth-bound fluoride on enamel demineralization/ remineralization *in vitro*. Caries Res. 2000;34(4):281-8.
60. Lynch RJ, Mony U, ten Cate JM. Effect of lesion characteristics and mineralizing solution type on enamel remineralization *in vitro*. Caries Res. 2007;41(4):257-62.
61. Birant S, Gümüştaş B. The effect of thermal aging on microhardness and SEM/EDS for characterisation bioactive filling materials. BMC Oral Health. 2024;24(1):1142.
62. Farooq I, Bugshan A. The role of salivary contents and modern technologies in the remineralization of dental enamel: a narrative review. F1000Res. 2020;9:171.