

Interfacial Evaluation of CAD/CAM Resin Inlays on the Cavity Floor Using Swept-source Optical Coherence Tomography

S-H Han • Y Shimada • A Sadr • J Tagami • S-E Yang

Clinical Relevance

When a resin nanoceramic inlay is cemented using self-adhesive cement, a universal dentin adhesive can be applied to the prepared cavity. The application of the adhesive before self-adhesive cement placement provides similar or better interfacial adaptation than without the adhesive.

SUMMARY

Purpose: The first objective of this study was to determine whether the luting material used

Seung-Hoon Han, DDS, PhD, assistant professor, Department of Conservative Dentistry, St. Vincent Hospital, College of Medicine, The Catholic University of Korea, Gyeonggi-do, South Korea

Yasushi Shimada, DDS, PhD, associate professor, Department of Operative Dentistry, Okayama University, Okayama, Japan; and Department of Cariology and Operative Dentistry, Tokyo Medical and Dental University, Tokyo, Japan

Alireza Sadr, DDS, PhD, associate professor, Department of Restorative Dentistry, School of Dentistry, University of Washington, Seattle, Washington, USA

Junji Tagami, DDS, PhD, professor, Department of Cariology and Operative Dentistry, Tokyo Medical and Dental University, Tokyo, Japan

*Sung-Eun Yang, DDS, PhD, professor, Department of Conservative Dentistry, Seoul St. Mary's Hospital, College of Medicine, The Catholic University of Korea, Seoul, Korea

*Corresponding author: 222, Banpo-daero, Seocho-gu, Seoul 06591, Korea; e-mail: dentyeun@catholic.ac.kr

<https://doi.org/10.2341/19-141-L>

for computer-aided design and computer-aided manufacture resin nanoceramic inlays affected interfacial adaptation. The second objective was to investigate whether application of a universal dentin adhesive before cementation affected interfacial adaptation. The final objective was to compare the inlay-side and dentin-side interfaces in the cement space.

Methods and Materials: Seventy-four class I cavities were prepared on extracted human third molars. Cavities were optically scanned, and resin nanoceramic inlays were milled using Lava Ultimate blocks (3M ESPE). For the control groups, the fabricated inlays were cemented using Panavia V5 (Kuraray Noritake) or FujiCem 2 (GC). For the experimental groups, the teeth were randomly divided into groups I and II. Group I contained four subgroups using different luting materials; in all subgroups, the inlays were cemented and dual cured without pretreatment. Group II contained six subgroups in which inlays were cemented and dual cured after application of

a universal dentin adhesive. After thermocycling, interfacial adaptation was measured using swept-source optical coherence tomography (SS-OCT) imaging and statistically compared among groups.

Results: Interfacial adaptation was different depending on the luting material used ($p<0.05$). After application of a universal adhesive, some subgroups showed improved interfacial adaptation ($p<0.05$). In the comparison of inlay-side and dentin-side interfaces, no difference was found in interfacial adaptation ($p>0.05$).

Conclusions: Interfacial adaptation for resin nanoceramic inlays differed with luting material. For some self-adhesive cements, application of a universal adhesive before cementation improved interfacial adaptation.

INTRODUCTION

Computer-aided design and computer-aided manufacturing (CAD/CAM) restorations, using prefabricated blocks, is one of the latest trends in restorative dentistry. As well as ceramic materials, indirect composite inlays can be milled from prefabricated composite blocks.¹ One advantage of using a composite block is that resin can be less susceptible than ceramic to chipping during the milling process.² Glass ceramic materials are stiff and brittle, making them highly susceptible to chipping and fracture.³ Resin composite restorations do not cause excessive wear on natural dentition. In addition, if resin cement is used as the luting material, the modulus of elasticity of the cement is similar to that of the composite restoration, which can create a more uniform stress distribution throughout the teeth compared to ceramic inlays.^{4,5}

The retention of an indirect restoration largely depends on the adhesive cementation.⁶ Composite resin cement with a multistep application technique has been used to bond restorations. The use of conventional resin cements can improve the physical properties of an indirect restoration; however, a multistep application can be technique sensitive.⁷ Recently, self-adhesive cements (SACs) that do not require pretreatment of the tooth surface have been introduced and are gaining popularity. Their major advantage is simple application with little technique sensitivity. SACs have a characteristic requirement of need for pH increase: when an SAC is mixed, it has an initially low pH to demineralize the tooth material. After a

certain amount of time, that initial acidity should be neutralized to produce a durable cement.

Another current trend in composite restoration is use of a one-step universal adhesive for dentin bonding. One-step self-etch universal adhesives make the bonding procedure simple and fast. However, they create several problems for composite restorations.⁸ Moreover, incompatibility problems between one-step self-etch systems and self-dual-cure resin composites have been reported.^{9,10} It is known that the tertiary amine used as the accelerator in self-dual-cure composites can be neutralized by acidic functional monomers.⁹ Despite those incompatibility issues, the manufacturer of the nanoceramic block material recommends that its universal dentin adhesive be applied to both the restoration and the tooth before cementation. Therefore, it was questioned whether interfacial adaptation could be improved by applying a universal adhesive before placing the luting material. If the universal adhesive is to be applied, a flowable bulk-fill composite could be used as the luting material. It was also wondered whether interfacial adaptation using the flowable bulk-fill composite differed from that cemented with SACs.

The clinical success of indirect restorations depends on a durable bond to the tooth material.^{11,12} Several researchers investigated bond strength and fracture surfaces using light microscopes, scanning electron microscopes, or transmission electron microscopy.¹³⁻¹⁵ In those studies, adhesive, mixed, or cohesive failure at different locations can imply a weak or strong interface in a cemented restoration. However, those studies were performed after interfaces had been detached or separated.

Optical coherence tomography (OCT) can be used to investigate the interfaces inside a restored tooth and to determine whether a microgap is present. When light passes through the interface between two types of media with different refractive indices, a portion of the light is reflected depending on the incidence angle and refractive index (n). The refractive index of air is 1.0 (n), that of water is 1.3, and that of a tooth or resin composite is 1.5-1.6. If there is a microgap between a tooth and a restoration, air or water can be present at the interface. When light transverses the air at the interface, a different portion of light is reflected, and the OCT system shows a higher signal intensity. Swept-source OCT (SS-OCT) is a specific type of OCT and is known to have high image resolution and scan speed. Therefore, SS-OCT can

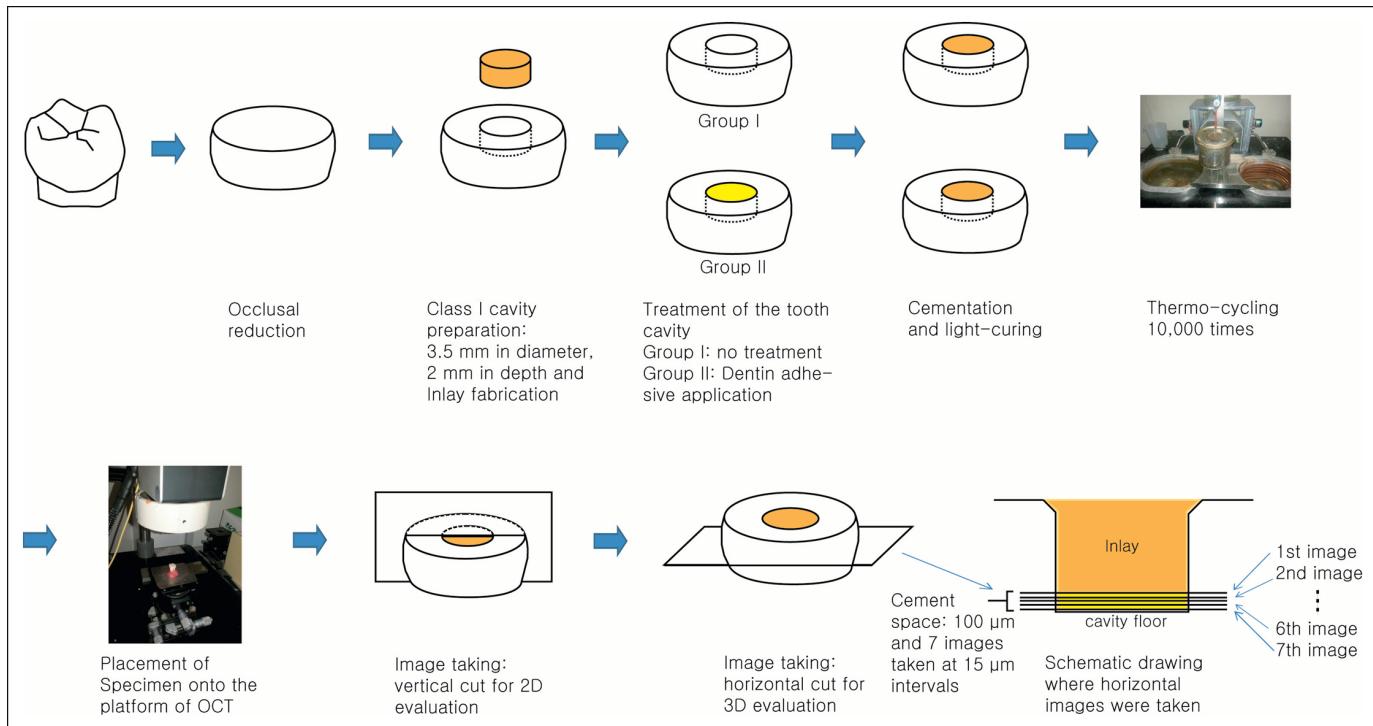


Figure 1. Experimental procedure for this study.

be used to investigate internal interfaces without damaging the specimen.¹⁶

The first objective of this study was to determine whether there was a difference in interfacial adaptation when diverse luting materials were used for resin nanoceramic inlay cementation. The second objective was to investigate whether application of a universal dentin adhesive before placement of the luting material would improve the interfacial adaptation. The final objective of this study was to compare the two interfaces: inlay-side interface between the restorative material and the luting material and dentin-side interface between the tooth and the luting material.

The null hypotheses tested were as follows: 1) there is no difference in the interfacial adaptation of a resin nanoceramic inlay restoration when it is cemented by different luting materials; 2) when a resin nanoceramic inlay is cemented with self-adhesive cement after application of a universal dentin adhesive, the interfacial adaptation does not differ from that without pretreatment; and 3) when SAC is used as the luting material, there is no difference in interfacial adaptation between the restorative material and the luting material compared to the tooth and the luting material.

METHODS AND MATERIALS

Specimen Preparation

The setup used in this study is schematically illustrated in Figure 1. Extracted human third molars, free of cracks, caries, and restorations, were selected and stored in a 0.5% chloramine solution at 4°C and used within two months of extraction. Seventy-four teeth were chosen with a buccolingual dimension of 10.5 ± 0.5 mm. The occlusal surface of each tooth was flattened with a trimmer and 320-grit SiC abrasive paper. Round class I cavities were prepared on the occlusal surface with a flat-end, straight diamond bur (6837-016, Komet Brasseler, Lemgo, Germany). The dimensions were 3.5 ± 0.2 mm in diameter and 2 ± 0.1 mm in depth. For cutting efficacy, a new bur was used every five preparations.

CAD/CAM Inlay Fabrication

Each prepared cavity was optically scanned (Medit Identica hybrid scanner, Medit, Seoul, Korea), and a virtual inlay was created with 100 µm of cement space (Milling software, Excad v 2.0.0.3). A resin nanoceramic block (21×19×14 mm) of Lava Ultimate (A2 HT, 3M ESPE, Neuss, Germany) was used to fabricate the inlays, which were milled using a

Roland Inlab milling machine (DWX-51D, Roland DG, Harmamatsu, Japan).

Groups and Restorative Procedure

For the control-1 group (PV5, n=6), the resin nanoceramic inlays were cemented using Panavia V5 (Kuraray Noritake, Tokyo, Japan). The internal surface of each indirect inlay was air-abraded using 50- μm Al_2O_3 particles 10 mm from the surface and two bars of (30 psi) pressure until the entire bonding surface had a matte appearance. After surface treatment, the particles were removed with alcohol, followed by ultrasonic cleaning in distilled water for three minutes and air drying. Then, Ceramic Primer Plus was applied and dried following the manufacturer's instructions. For tooth treatment, the Tooth Primer in the Panavia V5 kit was applied to the dentin surface of the tooth cavity. Following the manufacturer's instructions, gentle dry air was blasted for 20 seconds after Tooth Primer application. Equal amounts of the base and catalyst of Panavia V5 were mixed and placed into the tooth cavity. After positioning the fabricated inlay into the cavity, a load of 0.5 kg was applied on the inlay surface to allow the extrusion of excess cement. After a transparent celluloid strip was placed onto the top of the inlay, light-curing was performed (1200 mW/cm², Elipar S10, 3M ESPE, St. Paul, MN, USA) on the top surface for 40 seconds.

For the control-2 group (FC2, n=6), Fuji Cem 2 (GC, Tokyo, Japan) was used for inlay cementation. After the same mechanical treatment on the inlay surface as used for the control-1 group, equal amounts of Fuji Cem 2 base and catalyst were mixed and placed into the tooth cavity. After positioning the inlay, the cement was allowed to set for 10 minutes without light-curing.

For the experimental groups, the teeth were randomly divided into two groups (groups I and II). Group I was composed of four subgroups, and the inlays were cemented without pretreatment. Group II was composed of six subgroups, and the inlays were cemented after application of a universal dentin adhesive. For group I, the fabricated inlay was cemented without pretreatment. The prepared teeth and fabricated inlays were randomly assigned to four subgroups (n=6 per subgroup) according to luting material. The cementing procedures are described in Table 1. For group RXU, RelyX U200 (3M ESPE) was used. For group GCL, G-Cem LinkAce (GC) was used. For groups SC2 and MLS, SmartCem2 (Dentsply Caulk, Milford, DE, USA) and Multilink Speed (Ivoclar Vivadent, Schaan, Liechtenstein) were used, respectively.

For group II, the fabricated inlay was cemented after application of a one-step self-etch universal adhesive, Clearfil Universal Bond Quick (CUB). The CUB was applied to the internal portion of the inlay and cavity dentin with a rubbing motion and dried by blowing mild air onto it until the adhesive did not move. The teeth and fabricated inlays in group II were randomly assigned to six subgroups (n=6 teeth per subgroup). For group RXU, RelyX U200 (3M ESPE) was used for cementation after CUB had been applied. For group GCL, G-Cem LinkAce (GC) was used after CUB treatment. For groups SC2 and MLS, SmartCem2 (Dentsply Caulk) and Multilink Speed (Ivoclar Vivadent), respectively, were used in the same way. For group SDR, the SDR (Smart Dentin Replacement, Dentsply Caulk) bulk fill flowable composite was used as the luting material after CUB treatment. For group VBF, Venus Bulk Fill flowable composite (Heraeus Kulzer, Dormagen, Germany) was applied in the same way.

After the luting material was applied, the fabricated inlay was placed. A load of 0.5 kg was applied on the occlusal surface to allow extrusion of excess cement. After a transparent celluloid strip was placed onto the top of the inlay, light-curing was performed (1200 mW/cm², Elipar S10, 3M ESPE) on the top surface for 40 seconds.

Thermocycling Procedure

All specimens were stored in water at room temperature for 24 hours before thermocycling. Then, the specimens were fatigued with 10,000 thermocycles to represent approximately one year of clinical functioning.¹⁷ The teeth were cycled between water baths of 5°C and 55°C with a dwell time of 30 seconds and a transfer time of five seconds. After thermocycling, the specimens were stored in water at room temperature.

SS-OCT System

The SS-OCT system (IVS-2000, Santec Co., Komaki, Japan) used in this study was a frequency-domain OCT system integrating a high-speed frequency. To sweep the external cavity, the near-infrared spectrum was swept from 1260 to 1360 nm, with its spectral bandwidth centered at 1310 nm, at a scan rate of 20 kHz. The system has a handheld probe with a power less than 5 mW. Backscattered light-carrying information about the microstructure of the sample was collected, digitized in a time scale, and analyzed in the Fourier domain to reveal information at each location on the x-axis or depth (A-scan). Combining a series of A-scans along a scan path can

Table 1: Composition and Application Method for the Materials Used

Code	Material	Product	Manufacturer	Composition	Application Procedure
CUB	Self-etch universal dentin adhesive	Clearfil universal bond quick	Kuraray Noritake, Tokyo, Japan	Bis-GMA, 10-MDP, HEMA, hydrophilic amide monomer, filler, ethanol, water, NaF, photo initiators, chemical polymerization accelerator	After the dentin was dried, the adhesive was applied. Then, the entire cavity wall was dried by blowing mild air.
PV5	Dual-cure resin cement	Panavia V5	Kuraray Noritake, Tokyo, Japan	Paste A: Bis-GMA, TEGDMA, initiators, accelerators, silanated barium glass filler; Paste B: Bis-GMA, silanated aluminum oxide filler, camphorquinone	The Tooth Primer was applied, left for 20 seconds, and gently air-dried. The mixed paste was placed.
FC2	RMGI (resin modified glass ionomer) cement	Fuji Cem 2	GC, Tokyo, Japan	Paste A: Fluoroaluminosilicate glass, HEMA, dimethacrylate, pigment, initiator; Paste B: Polyacrylic acid, silica powder, initiator	Paste A and paste B were mixed for 10-15 seconds. The cavity wall was coated with the cement.
RXU	Self-adhesive cement	RelyX U200	3M ESPE, St Paul, MN, USA	Methacrylate monomers containing phosphoric acid groups, silanated fillers, initiator, alkaline fillers	Mixed cement was applied to the cavity using an auto-mix syringe.
GCL	Self-adhesive cement	G-Cem LinkAce	GC, Tokyo, Japan	Powder: Fluoroaluminosilicate glass, initiator, pigment; Liquid: 4-META, phosphoric acid ester monomer, UDMA	Mixed cement was applied to the cavity using an auto-mix syringe.
SC2	Self-adhesive cement	Smart-Cem2	Dentsply Caulk, Milford, DE, USA	UDMA, di- and tri-methacrylate resins, fluoroaluminosilicate glass, initiator, accelerators	Mixed cement was applied to the cavity using an auto-mix syringe.
MLS	Self-adhesive cement	Multilink Speed	Ivoclar Vivadent, Schaan, Liechtenstein	Methacrylate monomers containing phosphoric acid groups, dimethacrylate, barium glass, ytterbium trifluoride	Mixed cement was applied to the cavity using an auto-mix syringe.
SDR	Bulk-fill flowable composite	SDR	Dentsply Caulk, Milford, DE, USA	Modified UDMA, EBPADMA, TEGDMA, Ba-Al-F-B silicate glass, St-Al-F silicate glass, photo-initiator	Flowable composite was applied to the cavity
VBF	Bulk-fill flowable composite	Venus Bulk Fill	Heraeus Kulzer, Dormagen, Germany	UDMA, EBPADMA, Bis-EMA, Ba-Al-F silicate glass, YbF ₃ , silicon dioxide	Flowable composite was applied to the cavity

Abbreviations: Bis-EMA, bisphenol-A polyethylene glycol diether dimethacrylate; Bis-GMA, bisphenol-A diglycidyl ether dimethacrylate; EBPADMA, ethoxylated bisphenol A dimethacrylate; HEMA, 2-hydroxyethyl methacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate; 4-META, 4-methacryloyloxyethyl trimellitate anhydride; 10-MDP, 10-methacryloyloxydecyl dihydrogen phosphate.

produce a B-scan. By transforming the raw B-scan data into grayscale, a cross-sectional image can be created. Using serial B-scans over an area, the system produces a 3D image (horizontal cut image in this experiment). The axial resolution of the OCT system was 11 µm in air, which was equivalent to 7 µm in oral hard tissues and resin composites, assuming a refractive index of about $n = 1.5$.

SS-OCT Image Collection and Analysis

Each specimen was positioned on the metal platform of the OCT system. The surface of the specimen was dried using an air duster to standardize the surface conditions. Using the handheld scanning probe connected to the SS-OCT, the light beam was

projected onto the inlay surface of the specimen and scanned across the area.

For vertical-cut image collection, the first SS-OCT image was taken at the center of the restoration. The light beam was projected onto the bottom surface of the specimen and scanned across the area. The OCT probe was set at a fixed distance above the specimen. The first SS-OCT image of the restoration was taken parallel to the buccolingual plane of the cavity at the center of the restoration (Figure 1). After the first image was taken, the platform holding the specimen was moved 0.3 mm distally, followed by acquisition of the second image. The third image was taken after the platform was positioned at 0.3 mm mesially from the original center position. These vertical-cut

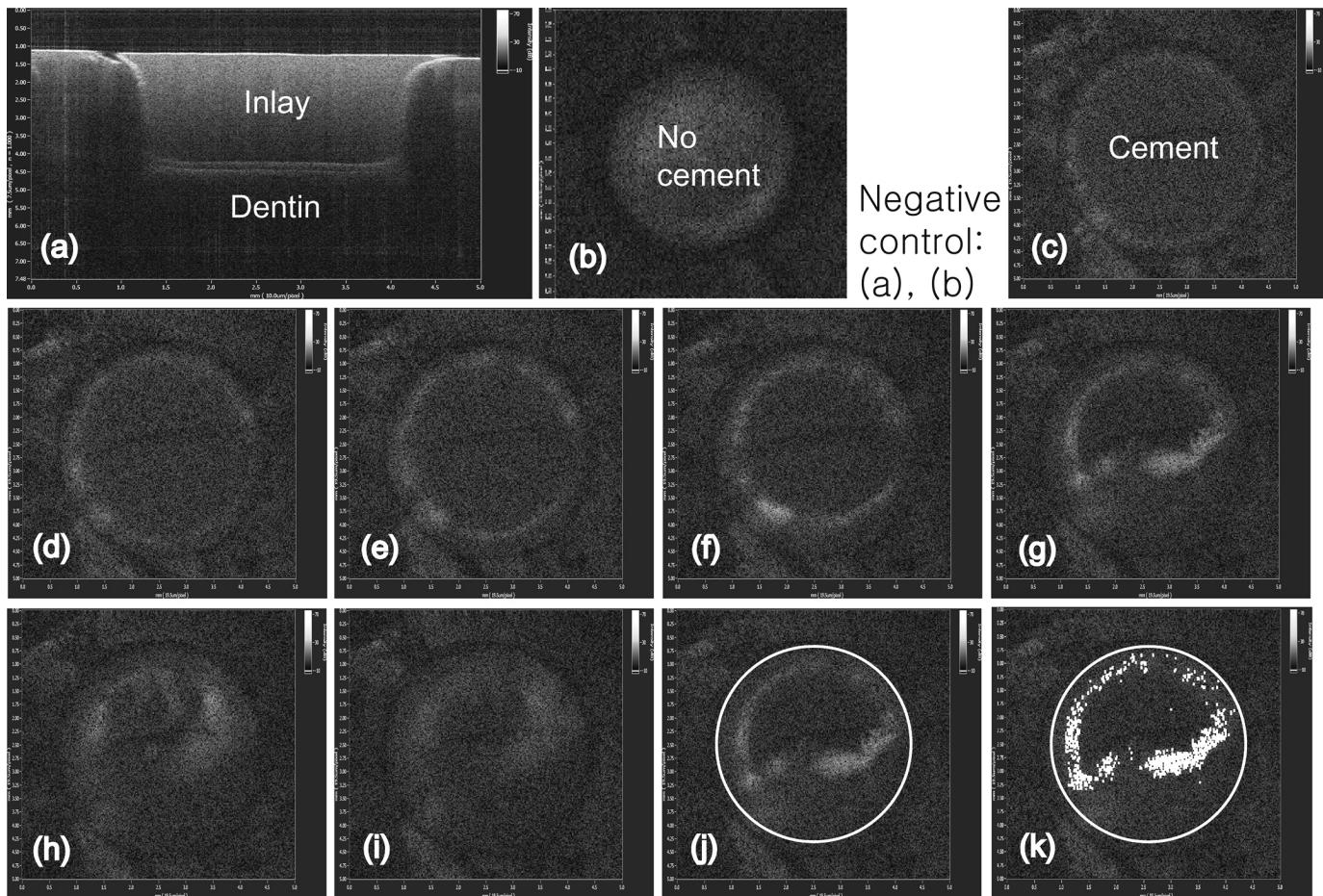


Figure 2. SS-OCT images for interfacial adaptation measurement. (a) Representative OCT image of a negative control without luting material: vertical cut. (b) A negative control image of the cement space: horizontal cut. (c) The first horizontal-cut image with luting material (PV5) in the cement space. The luting material shows an intensity value similar to that of dentin. The first image was taken parallel to the cavity floor 5 μm down from the inlay base. (d) The second image with PV5 in the cement space. The second image was taken 15 μm down from the first image. (e-i) The third, fourth, fifth, sixth, and seventh images, respectively, which were each taken 15 μm down from the previous image. j and k are the same images as in g, which are presented for the calculation of HB% of image g. (j) The same image as in g with a circle representing the cavity border. (k) The same image as in j processed by GapAnalyzer. The white dots on image k are brighter pixels that have higher signal intensity than the threshold to indicate a microgap at the cement space. The signal intensity of the threshold value was determined using the negative control image. On image k, the area of white dots was measured and then divided by the area of the circle to calculate the interfacial adaptation (HB%). On image k, the HB% was calculated to be 11.9%.

images were used as references and not applied in the statistical analysis.

For horizontal-cut image collection, the first SS-OCT image of the restoration was taken parallel to the cavity floor 5 μm below the inlay base. After the first image was taken, the platform holding the specimen was moved upward 15 μm , followed by acquisition of the second image. A total of seven images were taken in the cement space of each specimen at 15- μm intervals (Figure 1).

To evaluate the resin–tooth interfacial adaptation, raw OCT data were imported into image analysis software (ImageJ, ver. 1.48, National Institutes of Health, Bethesda, MD, USA). If air or water was present at the tooth–restoration interface, part of the

light reflected from the interface was visualized as a bright spot on the image (Figure 2). The high brightness (HB%) parameter was defined as the percentage of brighter pixels with a signal intensity above the threshold value in the signal intensity profile^{16,18} and calculated to indicate microgap or nonadapted area in the cement space (Figure 2k). To calculate the percentage of pixels brighter than the threshold, images were processed using GapAnalyzer, plug-in software that uses a binarization process described previously.^{19,20} Using the seven horizontal-cut cross-sectional images of each specimen, the mean HB% per sample was calculated. A higher HB% represents inferior interfacial adaptation in the cement space.

Table 2: Mean High Brightness Values (HB%) in Each Group^a

Group	Control ^b		Experimental Subgroup					
	PV5	FC2	RXU	GCL	SM2	MLS	Adhesive ^c + SDR	Adhesive ^c + VBF
I: Cementation without pretreatment	10.2 (3.4) a	24.6 (4.6) d	10.7 (3.4) a,b,A	11.1 (3.6) a,b,A	18.2 (4.0) c,B	13.0 (3.8) a,b,B		
II: Cementation with dentin adhesive	10.2 (3.4) a		9.4 (3.2) a,A	10.4 (2.9) a,A	14.8 (3.4) c,A	10.4 (3.0) a,A	11.1 (3.2) a,b	12.9 (3.5) b,c

^a Standard deviations are indicated in parentheses. In each row, values marked by identical lowercase letters are not significantly different (one-way ANOVA and Tukey test, $p>0.05$). In each column, values marked by identical uppercase letters are not significantly different (independent t-test, $p>0.05$).

^b The results of PV5 (control-1) for Groups I and II and FC2 (control-2) for Group I are presented for comparison with other luting materials.

^c The adhesive was CUB.

Comparative Analysis of the Inlay-side Interface and Dentin-side Interface

The CAD/CAM inlay was fabricated with a 100- μm cement space. For inlay-side interfacial adaptation, which can represent the interface between the inlay and luting material, the first and second horizontal-cut images were measured (Figure 1). As explained earlier, the first image was taken parallel to the cavity floor 5 μm down from the inlay base (Figure 2c). The second image was taken 15 μm down from the previous image (Figure 2d). The inlay-side interfacial adaptation was the average HB% of the first and second images. The dentin-side interfacial adaptation, which may represent the interface between the tooth and the luting material, was calculated as the mean of the sixth and seventh images. Inlay-side and dentin-side interfacial adaptations were calculated for each specimen.

Statistical Analysis

Because the distribution of measurements in each group was normal (Shapiro-Wilk test, $p>0.05$), parametric statistical tests were performed. To test for homogeneity of variances of the groups, Levenes test was used ($p>0.05$). The HB% of the SAC groups

(RXU, GCL, SM2, and MLS) was analyzed using two-way analysis of variance (ANOVA) to test the effects of universal adhesive application and luting material, as well as their interaction. For the comparison of subgroups in groups I and II, one-way ANOVA and the Tukey test were performed. To compare the SAC subgroups between groups I and II, an independent t-test was used (Table 2). For analysis of interfacial adaptation at the inlay-side and dentin-side interfaces, one-way ANOVA and post hoc Tukey tests were performed (Table 3). All statistical analyses were conducted using PASW Statistics 18 software (SPSS for Windows, SPSS, Inc, Chicago, IL, USA) with the significance level set at $\alpha=0.05$.

RESULTS

The interfacial adaptations (HB%) are summarized in Table 2. The two-way ANOVA showed the significance of universal adhesive application, the luting material, and their interaction, at 0.008, less than 0.001, and 0.036 ($p<0.05$), respectively. After one-way ANOVA and Tukey analysis, interfacial adaptation was different depending on the luting material in both groups I and II (Table 2; each row,

Table 3: Mean HB% of control (PV5) and Self-adhesive Cements at the Inlay-side and Dentin-side Interfaces^a

Group	Control, PV5 ^b		Experimental Subgroup			
		RXU	GCL	SC2	MLS	
I						
Inlay-side interface	9.1 (1.6) a,A	9.8 (4.0) a,A	11.7 (4.6) a,A	17.2 (2.0) b,A,B	13.0 (2.4) a,B	
Dentin-side interface	11.1 (2.3) a,A	10.6 (2.2) a,A	11.1 (2.5) a,A	19.1 (3.6) b,B	12.6 (4.4) a,A,B	
II						
Inlay-side interface	9.1 (1.6) a,A	11.4 (3.8) a,A	11.0 (2.5) a,A	15.9 (3.0) b,A,B	11.3 (3.3) a,A,B	
Dentin-side interface	11.1 (2.3) a,b,A	8.3 (2.0) a,A	8.8 (2.1) a,A	14.1 (3.6) b,A	9.1 (2.4) a,A	

^a Standard deviations are indicated in parentheses. In each row, values marked by identical lowercase letters are not significantly different (one-way ANOVA, $p>0.05$). In each column, values marked by identical uppercase letters are not significantly different (one-way ANOVA, $p>0.05$).

^b The results of PV5 in Groups I and II are presented for comparison with other subgroups.

$p<0.05$). The independent *t*-test showed that application of a universal dentin adhesive significantly improved adaptation in some of the SAC groups (Table 2; each column, $p<0.05$).

In the comparison of inlay-side and dentin-side interfaces in groups I and II, no different interfacial adaptation was found (Table 3; $p>0.05$). In the comparison of inlay-side interfacial adaptations between groups I and II, no difference was found (Table 3; first and third rows, $p>0.05$). However, a different adaptation was found at the dentin-side interface between a subgroup of group I and a subgroup of group II (Table 3; second and fourth rows, $p<0.05$).

DISCUSSION

The first null hypothesis was rejected because HB% (internal adaptation) was different depending on luting material. Some of the SACs showed interfacial adaptation similar to that of the control group, whereas other SACs had inferior interfacial adaptation. Thus, SACs' self-adhesive capacities can be different by product features.²¹ SACs have a complex composition, including methacrylate monomers, acidic functional monomers, fillers, and initiators (Table 1). The selection and proportion of each component may determine the final properties of the materials.²² The second null hypothesis was partially rejected because application of a universal dentin adhesive improved interfacial adaptation, but only in some subgroups. The third null hypothesis was accepted because the inlay-side and dentin-side interfaces showed no difference in interfacial adaptation.

SACs are reported to have a limited capacity to demineralize dentin and have no significant infiltration more than a micrometer into the dentin.²³ However, some SACs showed interfacial adaptation comparable to that of conventional resin cement (Table 2). It was previously reported that RXU can make cement bind with the calcium in hydroxyapatite due to the phosphate group in its functional monomer.²⁴ GCL is known to contain two functional monomers: 4-META (4-methacryloyloxyethyl trimellitate anhydride) and a phosphoric-acid ester monomer.²⁵ 4-META can be hydrolyzed to form 4-MET (4-methacryloxyethyl trimellitic acid), which can have a chelating reaction with hydroxyapatite.^{26,27} SC2 and MLS had relatively inferior adaptation (Table 2). In other studies, SC2 and MLS showed relatively low micro-tensile or shear bond strength to dentin and high pretest failures.^{22,28,29} One study indicated that the bonding ability of SACs depends mainly on

the presence of an acid-functionalized monomer.³⁰ Thus, SACs' adhesive properties are material dependent and could be related to the composition of each material.^{21,28}

SAC manufacturers have challenges to address. SACs need two opposite characteristics to achieve stable cementation: hydrophilicity for tooth bonding and hydrophobicity for resin matrix durability. For the bonding mechanism, SACs include acidic, hydrophilic monomers that can chemically interact with the hydroxyapatite in the tooth. Carboxylic or phosphoric acid groups in the monomers, which can show a low pH before reaction, form a complex with the calcium of hydroxyapatite.^{23,31} To neutralize this initially acidic condition, a glass-ionomer concept was adopted.³² The acid–base reaction of the acidic functional group with the basic inorganic filler in the cement or the mineralized tooth surface can lead to neutralization.³⁰ However, some SACs still have low pH values (less than 4) even two to seven days after mixing.^{31,33} This acidity could compromise the curing of the resin, increase water sorption, and lower the SAC's mechanical properties.^{30,34} Another problem for SAC bonding can be dependence on the condition of the bonding substrate.³⁵ SACs showed different bond durability depending on dentin surface conditions such as moisture and smear layer.²⁹

Despite the problems with one-step universal adhesives, application of the universal adhesive produced similar or better interfacial adaptation than not using it (Table 2). Applying a universal adhesive to dentin provides an acidic treatment to the dentin. In terms of pH, SACs could be applied to the adhesive-treated surface because the acidic functional monomer in the SAC has a mechanism similar to that of a self-etch universal dentin adhesive.³⁰ When using a universal dentin adhesive in this study, one reason for the improved adaptation could be modification of the smear layer on the dentin surface. The presence of a smear layer has been recognized as a weak link to dentin.³⁶ It was reported that mild acidic treatment, which could remove the superficial, loosely bound fraction of the smear layer, could enhance adhesion.²³ Some SACs may not be able to modify the smear layer, so an additional treatment of universal adhesive could improve interfacial adaptation.

No differences in adaptation were found in the comparison of the inlay-side and dentin-side interfaces in groups I and II (Table 3). Generally, the dentin-side interface showed inferior adaptation to the inlay-side; however, no statistically significant difference was found. For the comparison of the

inlay-side of group I and that of group II, no difference was found. It could indicate that universal adhesive treatment on the resin inlay surface did not significantly improve adaptation at the inlay-side interface. It was previously reported that mechanical treatments such as sandblasting would be the most determining factor for improving the retention of indirect composite restorations.²² Another paper presented that sandblasting Lava Ultimate contributed most of the bonding, whereas the effects of other treatments remained unclear.¹³ In the comparison of dentin-side interfaces between groups I and II, one significant difference was found (Table 3). It can suggest that a universal adhesive may improve adhesion to the dentin interface depending on the luting material.

The horizontal OCT images show the interfacial debonding pattern in the cement space under the inlay. The most frequent debonding pattern at the inlay side was circular along the border of the cavity, while that at the dentin side was irregular (Figures 3 and 4). The circular pattern can be due to polymerization shrinkage of the luting material toward the center of the cement space and the relatively even bond strength on the inlay-side interface. However, the debonding pattern on the dentin side was somewhat different and frequently irregular, which might indicate that bonding to the dentin surface was not consistent. Several other variables might also affect interfacial adaptation at the dentin surface: different characteristics of regional dentin, different properties of dentin adhesives, surface defects, air bubbles, phase separation, and a non-uniform adhesive layer. Those variables could make the bond strength on the dentin side nonuniform to produce an irregular debonding pattern.

Drawbacks and limitations should be considered when the interfacial adaptation is evaluated by the SS-OCT image. First, SS-OCT has depth limitations. Even though SS-OCT shows very clear images within the penetrating depth of the laser, it cannot be used in deep cavities or with a non-light-transmitting material such as metal. The imaging depth of SS-OCT systems had been reported to be in the range of 2-3 mm.³⁷ Therefore, the cavity design for SS-OCT evaluation can be limited depending on the light penetration capability. The second thing to consider is the threshold for the gap decision. It would be ideal to evaluate the images with one signal intensity threshold. However, defining a SS-OCT threshold was very complicated because the signal intensity was affected by the light intensity, scattering, attenuation, and transmittance properties of the

material.^{16,19} In this experiment, the threshold was determined after the signal intensity was measured on the negative control image.

PV5, which is a dual-cure resin cement, was used as a control. It is claimed that PV5 contains a novel amine-free redox initiator that makes it possible to avoid the oxidation of amine. Unlike the previous version (Panavia F2), PV5 does not contain 10-MDP in the cement paste, but it is included in the Tooth Primer. Essentially, 10-MDP is a hydrophilic, acidic monomer that can lead the cement to a low degree of conversion. FC2, which was used as a second reference control, showed much more debonding on the dentin side than on the inlay side. For the SDR and VBF groups, a bulk-fill flowable composite which has no acid-base reaction for polymerization, was tested as the luting material. Those groups showed interfacial adaptation comparable to those of the resin-based cements (Table 2). However, the debonding pattern was somewhat different and frequently showed an agglomerated pattern. Bulk-fill flowable composites do not have self-curing potential and are fully light dependent, highlighting the importance of transmitting light through the whole restorative material.

SACs are known to contain specific components to prevent the acid-base reaction problem.³⁰ As explained in the Introduction, the problem with self-/dual-cure resins is incompatibility between the residual acidic monomer of the universal dentin adhesive and the tertiary amine catalyst.³⁸ For SACs, an acid-tolerant oxidant such as cumene hydroperoxide is included in the acidic part, and a reductant such as benzoyl thiourea is used in the nonacidic part.^{30,39} Adverse acid-base reactions can also be circumvented by using acid-resistant catalysts such as aryl sulphate salts.^{9,30,40} The sodium salt of aryl sulfinic acid can react with acidic monomers to produce phenyl free radicals, which can initiate the self-curing process of the resin.⁴⁰ This acid-tolerant composition can help to lessen the adverse acidic effects of the universal adhesive.

Immediate light-curing can be important to reduce adverse effects when using a one-step self-etch universal adhesive. Studies have shown that light-curing improved the degree of conversion and bond strength compared with self-curing alone.⁴¹⁻⁴³ Photo-initiators such as camphorquinone (CQ) and diphenyl-(2,4,6-trimethylbenzoyl) phosphine oxide (TPO) are used in the acidic components of SACs. After acid contamination, light-curing of resin was inhibited to a much lower extent than that of self-curing,⁹ possibly because photo-initiation of free radicals is

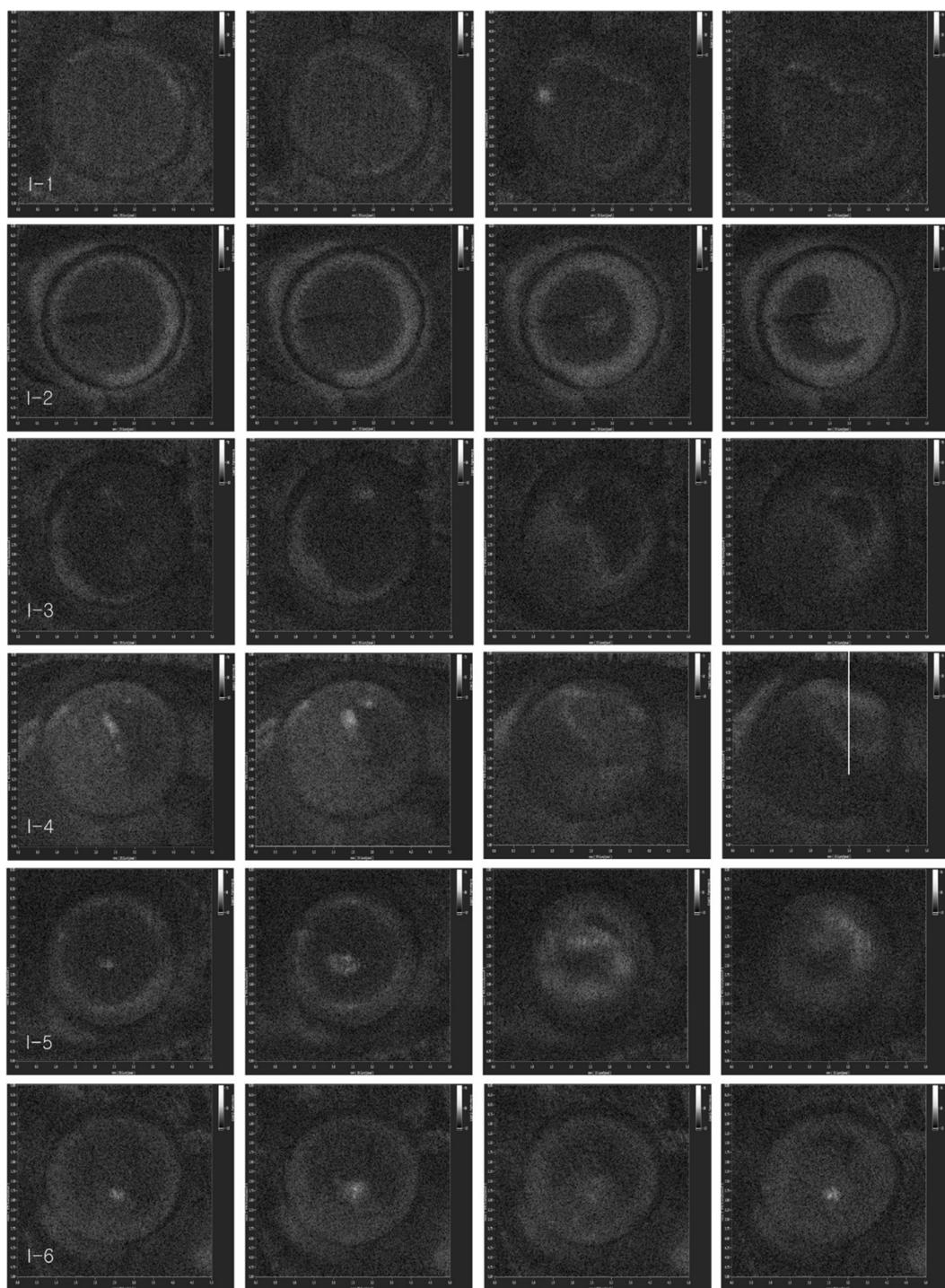


Figure 3. Interfacial horizontal-cut images of group I at the cement space. Row I-1 represents the first image, second image, sixth image, and seventh image of the control-1 group (PV5). Row I-2 represents the same four images of control-2 group (FC2). Row I-3 represents the same four images from the RelyX U200 group (RXU). Row I-4 represents the same four images from the G-cem LinkAce group (GCL). Row I-5 represents the same four images from the Smartcem2 group (SC2). Row I-6 represents the same four images from the Multilink Speed group (MLS).

much faster than that of chemical initiators.⁴⁴ Another problem of one-step adhesives is that the permeable adhesive layer can trigger osmotic fluid movement.⁸ When the adhesive layer was not

immediately cured, water sorption from the dentinal tubule increased.⁴⁴⁻⁴⁶ It is thought that immediate light-curing with adequate light should be performed whenever a universal dentin adhesive is applied.

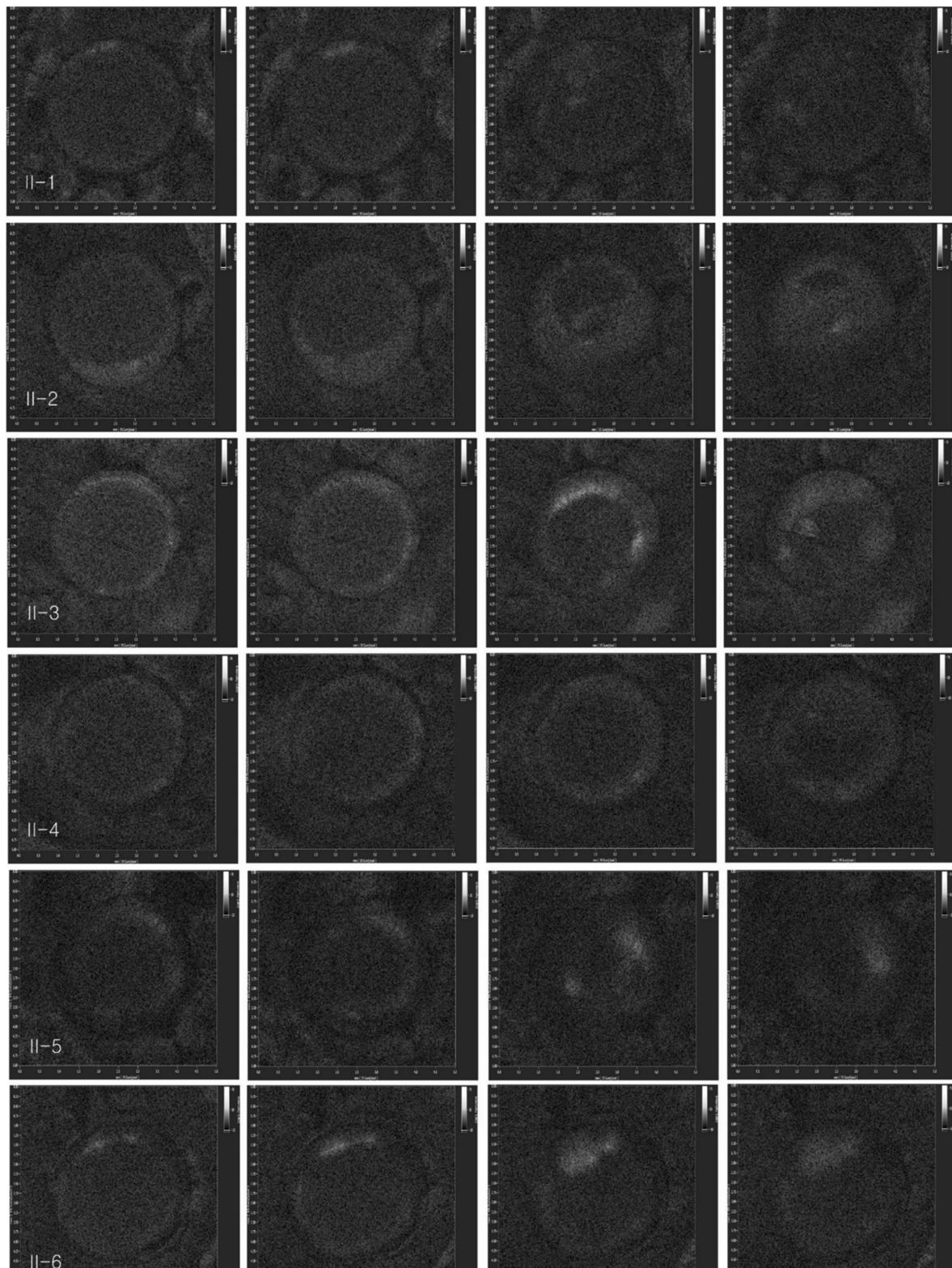


Figure 4. Interfacial horizontal-cut images of group II at the cement space. Row II-1 represents the first image, second image, sixth image, and seventh image of the RelyX U200 group (RXU). Row II-2 represents the same four images from the G-cem LinkAce group (GCL). Row II-3 represents the same four images from the Smartcem2 group (SC2). Row II-4 represents the same four images from the Multilink Speed group (MLS). Row II-5 represents the same four images when the inlays were cemented by SDR flowable resin after application of the universal dentin adhesive. Row II-6 represents the same four images when the inlays were cemented by VBF flowable resin after application of the universal dentin adhesive.

CONCLUSION

Under the limitations of this study, it can be concluded that the interfacial adaptation of a resin nanoceramic inlay restorations were different depending on luting material. For the resin nanoceramic inlay cementation, the application of a universal dentin adhesive before SAC placement showed similar or better interfacial adaptation than without the dentin adhesive. When SAC was used as a luting material without any pretreatment, the interfacial adaptation of the inlay-side was not different from that of the dentin-side interface. The comparison of the interfacial adaptation with the adhesive and that without the adhesive was performed on the inlay-side and the dentin-side. On the inlay-side, the interfacial adaptation with the universal adhesive application was not different from that without the adhesive. However, on the dentin-side, the interfacial adaptation with the adhesive was better in some groups.

Acknowledgement

The authors acknowledge the financial support of St Vincent's Hospital, Research Institute of Medical Science (SVHR-2017-11).

Regulatory Statement

This study was conducted in accordance with all the provisions of the local human subjects oversight committee guidelines and policies of St Vincent Hospital, the Catholic University of Korea. The approval code issued for this study is VC17OESI0173.

Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

(Accepted 7 October 2019)

REFERENCES

1. Fasbinder DJ (2012) Chairside CAD/CAM: an overview of restorative material options *Compendium of Continuing Education in Dentistry* **33**(1) 50, 52-58.
2. Tsitrou EA, Northeast SE, & van Noort R (2007) Brittleness index of machinable dental materials and its relation to the marginal chipping factor *Journal of Dentistry* **35**(12) 897-902.
3. Ruse ND & Sadoun MJ (2014) Resin-composite blocks for dental CAD/CAM applications *Journal of Dental Research* **93**(12) 1232-1234.
4. Ausiello P, Rengo S, Davidson CL, & Watts DC (2004) Stress distributions in adhesively cemented ceramic and resin-composite Class II inlay restorations: a 3D-FEA study *Dental Materials* **20**(9) 862-872.
5. St-Georges AJ, Sturdevant JR, Swift EJ, Jr., & Thompson JY (2003) Fracture resistance of prepared teeth restored with bonded inlay restorations *Journal of Prosthetic Dentistry* **89**(6) 551-557.
6. Kumbuloglu O & Ozcan M (2015) Clinical survival of indirect, anterior 3-unit surface-retained fibre-reinforced composite fixed dental prosthesis: up to 7.5-years follow-up *Journal of Dentistry* **43**(6) 656-663.
7. Mak YF, Lai SC, Cheung GS, Chan AW, Tay FR, & Pashley DH (2002) Micro-tensile bond testing of resin cements to dentin and an indirect resin composite *Dental Materials* **18**(8) 609-621.
8. Van Meerbeek B, Yoshihara K, Yoshida Y, Mine A, De Munck J, & Van Landuyt KL (2011) State of the art of self-etch adhesives *Dental Materials* **27**(1) 17-28.
9. Suh BI, Feng L, Pashley DH, & Tay FR (2003) Factors contributing to the incompatibility between simplified-step adhesives and chemically-cured or dual-cured composites. Part III. Effect of acidic resin monomers *Journal of Adhesive Dentistry* **5**(4) 267-282.
10. Cheong C, King NM, Pashley DH, Ferrari M, Toledano M, & Tay FR (2003) Incompatibility of self-etch adhesives with chemical/dual-cured composites: two-step vs one-step systems *Operative Dentistry* **28**(6) 747-755.
11. Inokoshi S, Willems G, Van Meerbeek B, Lambrechts P, Braem M, & Vanherle G (1993) Dual-cure luting composites: part I: filler particle distribution *Journal of Oral Rehabilitation* **20**(2) 133-146.
12. Arrais CA, Rueggeberg FA, Waller JL, de Goes MF, & Giannini M (2008) Effect of curing mode on the polymerization characteristics of dual-cured resin cement systems *Journal of Dentistry* **36**(6) 418-426.
13. Luhrs AK, Pongprueksa P, De Munck J, Geurtzen W, & Van Meerbeek B (2014) Curing mode affects bond strength of adhesively luted composite CAD/CAM restorations to dentin *Dental Materials* **30**(3) 281-291.
14. El Zohairy AA, De Gee AJ, Mohsen MM, & Feilzer AJ (2003) Microtensile bond strength testing of luting cements to prefabricated CAD/CAM ceramic and composite blocks *Dental Materials* **19**(7) 575-583.
15. Shimada Y, Yamaguchi S, & Tagami J (2002) Micro-shear bond strength of dual-cured resin cement to glass ceramics *Dental Materials* **18**(5) 380-388.
16. Han SH, Sadr A, Tagami J, & Park SH (2016) Non-destructive evaluation of an internal adaptation of resin composite restoration with swept-source optical coherence tomography and micro-CT *Dental Materials* **32**(1) e1-e7.
17. Gale MS & Darvell BW (1999) Thermal cycling procedures for laboratory testing of dental restorations *Journal of Dentistry* **27**(2) 89-99.
18. Makishi P, Thithaweerat S, Sadr A, Shimada Y, Martins AL, Tagami J, & Giannini M (2015) Assessment of current adhesives in class I cavity: nondestructive imaging using optical coherence tomography and micro-tensile bond strength *Dental Materials* **31**(9) e190-e200.
19. Bista B, Sadr A, Nazari A, Shimada Y, Sumi Y, & Tagami J (2013) Nondestructive assessment of current one-step

- self-etch dental adhesives using optical coherence tomography *Journal of Biomedical Optics* **18(7)** 76020.
20. Turkistani A, Sadr A, Shimada Y, Nikaido T, Sumi Y, & Tagami J (2014) Sealing performance of resin cements before and after thermal cycling: evaluation by optical coherence tomography *Dental Materials* **30(9)** 993-1004.
 21. Sarr M, Mine A, De Munck J, Cardoso MV, Kane AW, Vreven J, Van Meerbeek B, & Van Landuyt KL (2010) Immediate bonding effectiveness of contemporary composite cements to dentin *Clinical Oral Investigations* **14(5)** 569-577.
 22. Fuentes MV, Escribano N, Baracco B, Romero M, & Ceballos L (2016) Effect of indirect composite treatment microtensile bond strength of self-adhesive resin cements *Journal of Clinical and Experimental Dentistry* **8(1)** e14-e21.
 23. Monticelli F, Osorio R, Mazzitelli C, Ferrari M, & Toledano M (2008) Limited decalcification/diffusion of self-adhesive cements into dentin *Journal of Dental Research* **87(10)** 974-979.
 24. Gerth HU, Dammaschke T, Zuchner H, & Schafer E (2006) Chemical analysis and bonding reaction of RelyX Unicem and Bifix composites: a comparative study *Dental Materials* **22(10)** 934-941.
 25. Mazzitelli C, Monticelli F, Osorio R, Casucci A, Toledano M, & Ferrari M (2008) Effect of simulated pulpal pressure on self-adhesive cements bonding to dentin *Dental Materials* **24(9)** 1156-1163.
 26. Nagakane K, Yoshida Y, Hirata I, Fukuda R, Nakayama Y, Shirai K, Ogawa T, Suzuki K, Van Meerbeek B, & Okazaki M (2006) Analysis of chemical interaction of 4-MET with hydroxyapatite using XPS *Dental Materials Journal* **25(4)** 645-649.
 27. Yoshida Y, Nagakane K, Fukuda R, Nakayama Y, Okazaki M, Shintani H, Inoue S, Tagawa Y, Suzuki K, De Munck J, & Van Meerbeek B (2004) Comparative study on adhesive performance of functional monomers *Journal of Dental Research* **83(6)** 454-458.
 28. Hitz T, Stawarczyk B, Fischer J, Hammerle CH, & Sailer I (2012) Are self-adhesive resin cements a valid alternative to conventional resin cements? A laboratory study of the long-term bond strength *Dental Materials* **28(11)** 1183-1190.
 29. Suyama Y, de Munck J, Cardoso MV, Yamada T, & Van Meerbeek B (2013) Bond durability of self-adhesive composite cements to dentine *Journal of Dentistry* **41(10)** 908-917.
 30. Ferracane JL, Stansbury JW, & Burke FJ (2011) Self-adhesive resin cements: chemistry, properties and clinical considerations *Journal of Oral Rehabilitation* **38(4)** 295-314.
 31. Roedel L, Bednarzic V, Belli R, Petschelt A, Lohbauer U, & Zorzin J (2017) Self-adhesive resin cements: pH-neutralization, hydrophilicity, and hygroscopic expansion stress *Clinical Oral Investigations* **21(5)** 1735-1741.
 32. Radovic I, Monticelli F, Goracci C, Vulicevic ZR, & Ferrari M (2008) Self-adhesive resin cements: a literature review *Journal of Adhesive Dentistry* **10(4)** 251-258.
 33. Han L, Okamoto A, Fukushima M, & Okiji T (2007) Evaluation of physical properties and surface degradation of self-adhesive resin cements *Dental Materials Journal* **26(6)** 906-914.
 34. Vrochari AD, Eliades G, Hellwig E, & Wrba KT (2010) Water sorption and solubility of four self-etching, self-adhesive resin luting agents *Journal of Adhesive Dentistry* **12(1)** 39-43.
 35. Park JW (2012) 'Wet or Dry tooth surface?' - for self-adhesive resin cement *Restorative Dentistry and Endodontics* **37(4)** 249-250.
 36. Al-Assaf K, Chakmakchi M, Palaghias G, Karanika-Kouma A, & Eliades G (2007) Interfacial characteristics of adhesive luting resins and composites with dentine *Dental Materials* **23(7)** 829-839.
 37. Shimada Y, Sadr A, Burrow MF, Tagami J, Ozawa N, & Sumi Y (2010) Validation of swept-source optical coherence tomography (SS-OCT) for the diagnosis of occlusal caries *Journal of Dentistry* **38(8)** 655-665.
 38. Sanares AM, Itthagaran A, King NM, Tay FR, & Pashley DH (2001) Adverse surface interactions between one-bottle light-cured adhesives and chemical-cured composites *Dental Materials* **17(6)** 542-556.
 39. Moszner N & Salz U (2007) Recent developments of new components for dental adhesives and composites *Macromolecular Materials and Engineering* **292(3)** 245-271.
 40. Ikemura K & Endo T (1999) Effect on adhesion of new polymerization initiator systems comprising 5-monosubstituted barbituric acids, aromatic sulfinate amides, and tert-butyl peroxymaleic acid in dental adhesive resin *Journal of Applied Polymer Science* **72(13)** 1655-1668.
 41. Kumbuloglu O, Lassila LV, User A, & Vallittu PK (2004) A study of the physical and chemical properties of four resin composite luting cements *International Journal of Prosthodontics* **17(3)** 357-363.
 42. Vrochari AD, Eliades G, Hellwig E, & Wrba KT (2009) Curing efficiency of four self-etching, self-adhesive resin cements *Dental Materials* **25(9)** 1104-1108.
 43. Zorzin J, Belli R, Wagner A, Petschelt A, & Lohbauer U (2014) Self-adhesive resin cements: adhesive performance to indirect restorative ceramics *Journal of Adhesive Dentistry* **16(6)** 541-546.
 44. Tay FR, King NM, Suh BI, & Pashley DH (2001) Effect of delayed activation of light-cured resin composites on bonding of all-in-one adhesives *Journal of Adhesive Dentistry* **3(3)** 207-225.
 45. Van Landuyt KL, Snaauwaert J, De Munck J, Coutinho E, Poitevin A, Yoshida Y, Suzuki K, Lambrechts P, & Van Meerbeek B (2007) Origin of interfacial droplets with one-step adhesives *Journal of Dental Research* **86(8)** 739-744.
 46. Tay FR, Pashley DH, Suh B, Carvalho R, & Miller M (2004) Single-step, self-etch adhesives behave as permeable membranes after polymerization. Part I. Bond strength and morphologic evidence *American Journal of Dentistry* **17(4)** 271-278.