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The strategies used for curing universal adhesives affect the micro-bond strength of resin cement used to lute indirect resin composites to human dentin

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We evaluated the effect of different curing strategies for universal adhesives on micro-tensile bond strength (μTBS) between resin cement and dentin and/or between resin cement and indirect resin composite. Flat coronal dentin surfaces and composite resin disks were pretreated with silane-containing universal adhesives, with or without light-curing on the dentin-side and/or composite resin disk-side. Resin disks were luted onto the pretreated dentin surfaces with the corresponding dual-cure adhesive resin cements and light-cured, and cut into beams after 24-h water storage. After 0 or 10,000 thermocycles (5ºC/55ºC) in a water bath, the μTBS of the composite resin disk–dentin beam was tested. The μTBS was highest when universal adhesives were applied to both the dentin- and the indirect composite resin disk-side, followed by light-curing. Thermocycling decreased μTBS in all but the Scotchbond Universal-treated group, with light-curing on both sides. The effect of curing strategies is dependent upon the materials.

Keywords: Universal adhesive, Indirect restoration, Dual-cure resin cement, Micro-tensile bond strength, Thermocycle aging

INTRODUCTION

In recent years, indirect tooth-colored restorations comprised of composite resin and all-ceramics have been widely used, because of their superior esthetics and biocompatibility. For these restorations, the dual-cure adhesive resin cement, which is equipped with both photo-cure and self-cure systems, has been frequently been utilized in order to obtain secure polymerization in a region where light hardly reaches under the restorative materials[1]. When bonding an indirect restoration to a tooth structure, two different interfaces need to be considered: between the tooth substrates and resin cement, and between restorative materials and resin cement[2]. Bonding performance of resin cement to both tooth substrates and restorative materials is crucial for improving the fracture strength of the restored tooth[3] and retention of the restorative materials[4], reduction of post-operative sensitivity[5], and its longevity[6]. In order to perform this task, optimal surface treatments for each adhesive substrate is necessary.

Recently, so-called "universal" one-step self-etch adhesives have been developed, which can bond to various restorative materials, such as indirect resin composites, glass ceramics, zirconia, and noble and non-precious alloys, without any additional primer applications, as well as to enamel and dentin[7,8], and are widely used for repairing resin composite restorations as well as for direct resin composite restoration as a photo-cure adhesive. In order to improve bonding to silica filler-containing resin composites and silica-based ceramics, these adhesives contain a silane coupling agent, which has two different functional groups that react with the methacrylate and the silica of the glass structure, respectively. This latter group exists as a non-functional silane (including an alkoxy group [-OR]) that reacts with an inorganic substrate after hydrolysis, but also achieves a three-dimensional crosslinking network of ceramic and resin[9].

These universal adhesives have also been used as a surface primer on tooth substrates and indirect restorative materials for cementation using adhesive resin cements in indirect restorations[10]. According to the manufacturers' instructions, application of light irradiation to the universal adhesives either on the surface of the dentin or that of the indirect restorative materials is not recommended before luting with the corresponding dual-cure resin cement, because this can result in chemical co-polymerization with the cements, due to a specialized accelerator present in the resin cement (a touch-curing system). In clinical application, it is difficult to allow sufficient light energy to reach the adhesive interface of the dentin and the restorative materials *via* the indirect restorative material. Therefore, touch-curing chemical-polymerization of the adhesives is considered to play an important role in the bonding performance of resin cements.

The treatment of one-step self-etching adhesives with an acidic functional monomer would change the surface pH and the remaining H^+ ions on the treated

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surface. When one-step self-etch adhesives are applied to tooth substrates, their acidity is neutralized by the calcium in the demineralized hydroxyapatite of the tooth substrates[11]. On the other hand, when they are applied to a resin composite or ceramic surface, the surface remains acidic. A previous report has demonstrated the polymerization incompatibility of a chemical-cure resin composite with one-step self-etch adhesives on dentin surfaces[12], because the chemical polymerization reaction of the resin composite could be inhibited under acidic conditions. A previous study reported that light-irradiation of the universal adhesives applied to dentin surfaces before luting of the resin cement increased the initial dentin bond strength of the resin cement[13]. This is likely due to the improvement of polymerization compatibility with the chemical-cure resin composite by light-curing the universal adhesives and/or by increasing the mechanical properties of the adhesive layer. On the other hand, a lack of neutralization of the universal adhesive on indirect restorative materials may result in polymerization incompatibility with a chemical-cure resin composite. However, the effect of light irradiation of universal adhesives applied to either the surface of the dentin or that of indirect restorative materials before luting of resin cement on bonding performance of resin cement has not been investigated to date. The purpose of this study was to evaluate the light-curing strategies of universal adhesives applied to dentin and to indirect resin composite material on the μTBS of resin cements used to lute indirect resin composite to dentin, in the presence and absence of thermocycler-based aging. The null hypothesis tested in this study was that the μTBS would not be influenced by light-curing of the adhesives of the resin cement, with or without thermocycle aging, and that μTBS would not be influenced by thermocycle aging.

MATERIALS AND METHODS

Specimen preparation

The occlusal dentin surface of 48 human third molars were collected following ethical approval by the Ethics Committee of Tokyo Medical and Dental University under protocol No. 725 and stored in refrigerated distilled water at 4°C before being used. Teeth were transversally sectioned in the middle of the crown using a diamond saw (Isomet, Buehler, Lake Bluff, IL, USA) under water irrigation, exposing areas of the middle coronal dentin. The exposed dentin surfaces were wet-polished with 600-grit silicon carbide paper (DCCS, Sankyo Fuji Star, Saitama, Japan) 30 times to create a flat surface with a standard smear layer before being bonded with the adhesive systems.

Indirect composite resin disks (2 mm in thickness, 10 mm in diameter) were fabricated from a resin composite (PEARLESTE, Shade DA2, Tokuyama Dental, Tokyo, Japan) using a silicone mold. Glass microscope slides were placed over the top and the bottom of the uncured resin composite. Both sides of the disks were cured for 60 s each, for a total of 120 s, using a halogen light source (Optilux 501, Demetron Kerr, Danbury, CT, USA), and were cured for 5 min using a laboratory light curing unit (Alpha Light II, J. Morita, Tokyo, Japan), followed by heat curing at 100ºC for 15 min in a heat curing unit (PEARL CURE HEAT, Tokuyama Dental). The surfaces of indirect composite resin disks were wet-polished with 600-grit silicon carbide paper 30 times. Prepared indirect composite resin disks were cleaned with 37% phosphoric acid (K-etchant, Kuraray Noritake Dental, Tokyo, Japan) for 10 s, rinsed with water and air-dried.

Indirect bonding restorative procedures

Teeth were randomly assigned to 1 of the 8 experimental groups. The schematic diagram for indirect bonding restorative procedures was shown in Fig. 1. Composite resin disks were luted onto dentin using RelyX Ultimate (RU; 3M ESPE, St Paul, MN, USA) and PANAVIA SA cement (PS; Kuraray Noritake Dental). Both dentin and composite resin disk surfaces were pretreated by Scotchbond Universal (SBU; 3M ESPE) and Clearfil Universal Bond (CUB; Kuraray Noritake Dental). The compositions of the resin cement, universal adhesives, and composite resin used in this study are listed in Table 1. The universal one-step adhesive was applied to the composite resin disks and dentin surfaces, and air-thinned according to the manufacturer's instructions. The composite resin disks were adhesively luted onto the dentin surfaces following the use of 4 different curing modes for the applied adhesives. For "$NL_{(D)}NL_{(CR)}$", neither the adhesives on the composite resin disk- nor on the dentin- sides were light-cured before luting. For "$L_{(D)}NL_{(CR)}$", only the adhesive on the dentin- sides were light-cured before luting. For "$NL_{(D)}L_{(CR)}$", only the adhesive applied to the composite resin disk- sides were light-cured before luting. For "$L_{(D)}L_{(CR)}$", the adhesives on both the composite resin disk- and the dentin- sides were light-cured before luting. Light irradiation was performed using a halogen light source (Optilux 501; Demetron Kerr) at 600 mW/cm^2 for 10 s according to the manufacturer's instructions. After applying the adhesive, the composite resin disks were luted using the resin cements, and a halogen light source was positioned against the composite resin disk; each specimen was light-activated at 600 mW/cm^2 for 40 s. All the bonding procedures were carried out at room temperature (23±1°C) and at 60% relative humidity. The specimens were stored in distilled water at 37°C for 24 h.

Microtensile bond strength (μTBS) testing

After storage in distilled deionized water at 37°C for 24 h, the specimens were sectioned into approximately 0.7×0.7 mm^2 beams using a low-speed diamond saw. Only beams from the central part of the dentin surface were used to avoid regional variability. The beams were further divided into 2 groups. Half of the beams were thermal cycled in distilled water for 10,000 cycles at 5 and 55ºC in a water bath, with a dwell-time of 30 s and a transfer time of 5 s. Before μTBS testing, the cross-sectional area of each beam was measured using digital

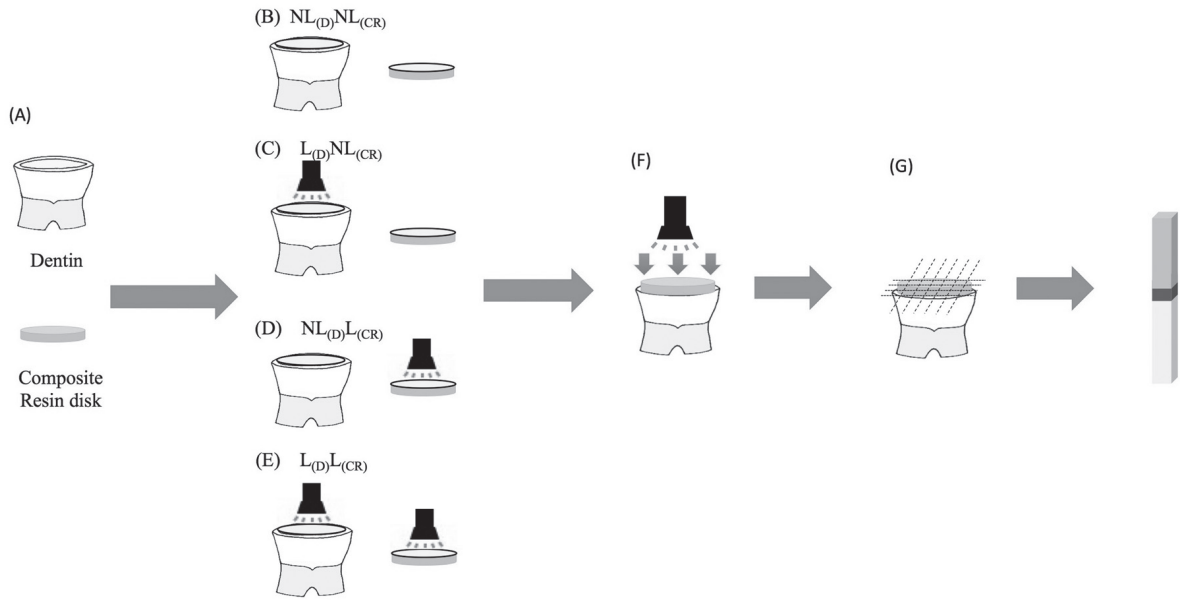

Fig. 1 Specimen preparation and sectioning of specimens for the micro-tensile bond test.
Preparation methods for 4 different modes of curing of the applied adhesives.
(A) Flattened dentin surfaces and composite resin disks (2-mm thickness, 10-mm diameter) that were wet-polished with 600-grit silicon carbide paper were prepared. Universal one-step self-etching adhesive was applied onto dentin and composite resin disk surfaces. (B) The adhesives on neither the composite resin disk- nor the dentin- sides were light-cured before luting. (C) The adhesives on only the dentin- sides were light-cured before luting. (D) The adhesives applied to only the composite resin disk- sides were light-cured before luting. (E) The adhesives on both the composite resin disk- and dentin- sides were light-cured before luting. (F) The composite resin disks were luted using the resin cements, and a halogen light source was positioned against the composite resin disk; each specimen was light-activated at 600 mW/cm^2 for 40 s. (G) The specimens were sectioned into approximately 0.7×0.7 mm^2 beams using a low-speed diamond saw.

calipers (Mitutoyo CD15, Mitutoyo, Kawasaki, Japan). Following this, each specimen was attached to a testing device in a table-top testing machine (EZ Test, Shimadzu, Kyoto, Japan) using cyanoacrylate adhesive (Model Repair II Blue, Dentsply-Sankin, Tokyo, Japan) and subjected to a tensile force at a crosshead speed of 1 mm/min. The value was recorded in kilogram×force (kgf) and transformed to μTBS values in MPa. The μTBS values were statistically analyzed using a three-way ANOVA (resin cements, curing modes of adhesive, thermocycle aging applications) and Student's *t*-test with Bonferroni correction for multiple comparison; significance was set at 0.05.

Fracture analysis

After the μTBS testing, both the resin disk-side and the dentin-side of the fractured beams were mounted on brass tablets and gold sputter-coated (ELIONIX, QUICK AUTO COATER, SC-701AT, Tokyo, Japan). The fracture modes were observed using a scanning electron microscope (SEM, JSM-5310, JEOL, Tokyo, Japan) with an accelerating voltage of 15 kV under 50× magnification. Fracture modes at the interface between the dentin and the resin were classified into 6 categories as "cohesive failure in dentin", "adhesive failure at the dentin–resin cement interface", "cohesive failure in resin cement", "adhesive failure at the resin cement–composite resin disk interface", "cohesive failure in composite resin disk", or "mixed failure."

Scanning electron microscopy (SEM) observation of the dentin–composite resin disk interface

The dentin–composite resin disk interfaces, treated with each resin–cement combination, was observed. The specimens were prepared using the same procedure as for μTBS measurement, as described above. After the specimens were stored in distilled water at 37°C for 24 h, each specimen was embedded with an epoxy resin (Epoxicure Resin, Buehler), and was sectioned perpendicular to the dentin–composite resin disk interface with a diamond saw, and polished with diamond paste (Struers, Copenhagen, Denmark), to a 0.25-μm particle size. The specimens were then gold-sputter coated, and the dentin–composite resin disk interfaces were observed using SEM with an accelerating voltage of 15 kV under 1,000× magnification.

Table 1 Chemical composition and application mode of the materials tested in this study

Material	Composition	Procedure	pH
Scotchbond Universal Adhesive (SBU; 3M ESPE, St.Paul, MN, USA)	10-MDP, Bis-GMA, Dimethacrylate resins, HEMA, Vitrebond™ Copolymer, Silane, Ethanol, Water, Filler, Initiators	Composite pre-treatment: Apply Scotchbond Universal Adhesive, leave for 20 s, Mild air blow Tooth pre-treatment: Apply (agitate) 20 s, 5 s air dry.	2.7
RelyX Ultimate Adhesive Resin Cement (RU; 3M ESPE)	Base Paste: Methacrylate monomers, Radiopaque Silanated fillers, Initiator components, Stabilizers, Rheological additives Catalyst Paste: Methacrylate monomers, Radiopaque Alkaline fillers, Initiator components, Stabilizers, Pigments, Rheological additives, Fluorescence dye	Cementation: Mix Paste A+B, place on the composite surface, gently press and remove excess cement, light cure for 20 s/surf.	—
CLEARFIL Universal Bond (CUB; Kuraray Noritake Dental, Tokyo, Japan)	Bis-GMA, HEMA, Ethanol ,10-MDP, Hydrophilic aliphatic dimethacrylate, Colloidal silica, dl Camphorquinone, Silane-coupling agent, Accelerators, Initiators, Water	Apply bond and rub 10 s, dry by blowing mild air for 5 s.	2.3
PANAVIA SA CEMENT (PS; Kuraray Noritake Dental)	PASTE A: 10-MDP, Bis-GMA, TEGDMA, Hydrophobic aromatic dimethacrylate, Silanated barium glass filler, Silanated colloidal silica, dl-Camphorquinone, Benzoyl peroxide, Initiators PASTE B: Bis-GMA, Hydrophobic aromatic dimethacrylate, Hydrophobic aliphatic dimethacrylate, Silanated barium glass filler, Silanated colloidal silica, Surface treated sodium fluoride, Accelerators, Pigments	Light-cure the entire surface and margins of the prosthetic restoration for 20 s.	—
Pearl Este (Tokuyama Dental, Tokyo, Japan)	Bis-MPEPP, TEGDMA, UDMA, Silica-zirconia filler, Silica-titanium filler	Light-cure the entire surface for 60 s and 5 min, followed by heat curing at 100°C for 15 min.	—

Abbreviations; 10-MDP: 10-Methacryloyloxydecyl dihydrogen phosphate, Bis-GMA: bisphenol-A-glycidyl methacrylate, HEMA: 2-hydroxyethyl methacrylate, TEGDMA: Triethyleneglycol dimethacrylate, UDMA: urethane dimethacrylate

RESULTS

Microtensile bond strengths

There were no pretesting failures during specimen preparation for μTBS testing. The results of μTBS testing are presented in Table 2. ANOVA revealed that the μTBSs were significantly influenced by resin cements ($p<0.01$), curing strategies for universal adhesives ($p<0.01$), and the use or not of thermocycle aging ($p<0.01$). There were significant interactions among materials, curing modes, and thermocycle aging applications ($p=0.001$). An interaction between materials and curing modes ($p<0.01$), and between curing modes and thermocycle aging ($p=0.001$), whereas there was no interaction between materials and thermocycle aging applications ($p=0.70$).

Among the SBU-treated specimens, the $L_{(D)}L_{(CR)}$ group showed significantly higher bond strength ($p<0.01$) than the other groups, and there was no significant difference among the $L_{(D)}NL_{(CR)}$, $NL_{(D)}L_{(CR)}$, and $NL_{(D)}NL_{(CR)}$ groups ($p>0.05$). In the CUB-treated specimens, the highest μTBS values were arranged in the order $L_{(D)}L_{(CR)}>NL_{(D)}L_{(CR)}>L_{(D)}NL_{(CR)}=NL_{(D)}NL_{(CR)}$. Thermocycle aging using 10,000 cycles significantly decreased the μTBS of all experimental groups, except for the $L_{(D)}L_{(CR)}$ group treated with SBU.

Failure mode analysis

Figure 2 presents the failure mode percentage of the debonded specimens. The $L_{(D)}NL_{(CR)}$ group specimens for both resin cements combinations failed predominantly at the interface of the composite resin disk and the resin cement. In contrast, the specimens in the $NL_{(D)}L_{(CR)}$ group failed predominantly at the interface of the dentin and the resin cement for SBU/RU and CUB/PS combinations. Varying failure patterns occurred in the $L_{(D)}L_{(CR)}$ and the $NL_{(D)}NL_{(CR)}$ groups specimens treated with the SBU/RU combination. For the CUB/PS combination, mixed failure and composite resin disk–resin cement interface failure were the predominant failure modes in the $L_{(D)}$

Table 2 Microtensile bond strength to dentin (MPa)

Materials	Storage condition	Curing mode			
		$NL_{(D)}NL_{(CR)}$	$L_{(D)}NL_{(CR)}$	$NL_{(D)}L_{(CR)}$	$L_{(D)}L_{(CR)}$
SBU	24 h	51.0±9.0 (56)^{A*}	51.5±8.0 (50)^{A*}	51.4±8.6 (58)^{A*}	74.3±7.6 (58)^{B*}
		$p<0.05$	$p<0.05$	$p<0.05$	*NS*
	TC	38.6±6.0 (44)$^{A+}$	39.2±7.1 (60)$^{A+}$	40.8±9.5 (58)$^{A+}$	71.9±9.0 (60)$^{B+}$
CUB	24 h	29.9±7.0 (57)^{A*}	31.4±7.8 (52)^{A*}	40.1±8.3 (46)^{B*}	51.0±9.2 (48)^{C*}
		$p<0.05$	$p<0.05$	$p<0.05$	$p<0.05$
	TC	20.3±4.2 (45)$^{A+}$	21.9±4.2 (49)$^{A+}$	31.8±4.5 (51)$^{B+}$	41.8±6.2 (59)$^{C+}$

Values are mean±SD (number of tested beams). Upper-case letters refer to row. In row, different upper-case letters indicate statistically significant differences. In columns, same symbols between the same storage condition of each material indicate statistical significance ($p<0.05$). *NS*=no significant difference ($p>0.05$) between 24 h and TC within the material. SBU: Scotchbond Universal Adhesive, RU: RelyX Ultimate Adhesive Resin Cement, CUB: CLEARFIL Universal Bond, PS: PANAVIA SA CEMENT, 24 h: without thermocycle aging, TC: with thermocycle aging.

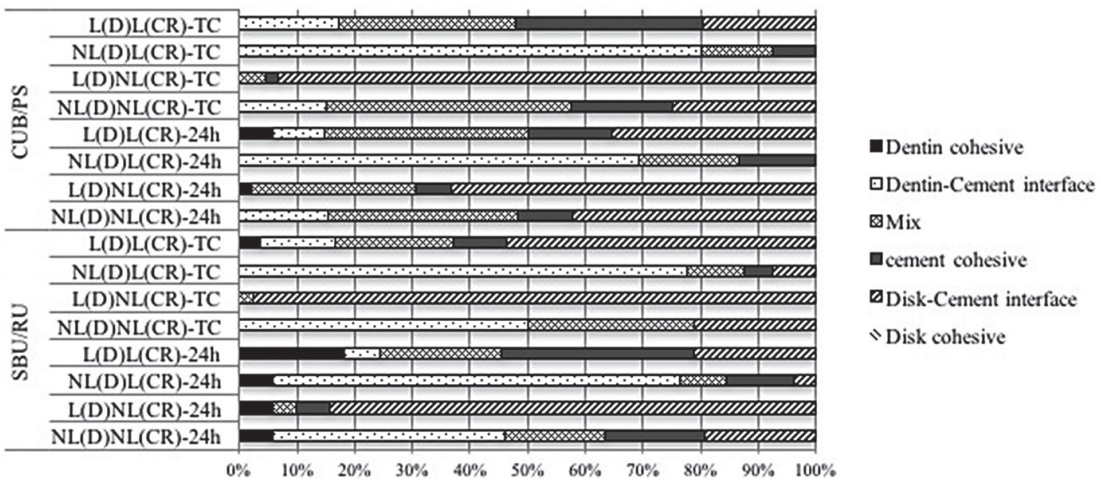

Fig. 2 Results of the SEM failure analysis for the different experimental groups.
SBU: Scotchbond Universal Adhesive, RU: RelyX Ultimate Adhesive Resin Cement, CUB: CLEARFIL Universal Bond, PS: PANAVIA SA CEMENT, $NL_{(D)}NL_{(CR)}$: neither the adhesives applied to the composite resin disks nor that applied to the dentin sides were light-cured before luting. $NL_{(D)}L_{(CR)}$: only the adhesive applied to the composite resin disk-sides was light-cured before luting. $L_{(D)}NL_{(CR)}$: only the adhesive applied to the dentin-side was light-cured before luting. $L_{(D)}L_{(CR)}$: the adhesives applied to both the composite resin disk- and dentin-sides were light-cured before luting, TC: with thermocycle aging.

$L_{(CR)}$ and the $NL_{(D)}NL_{(CR)}$ groups specimens.

Thermocycling increased the cohesive failure in the resin cement in the $L_{(D)}L_{(CR)}$ group specimens treated with the CUB/PS combination and interface failure specimens in the $L_{(D)}NL_{(CR)}$ and the $NL_{(D)}L_{(CR)}$ groups specimens treated with either of the SBU/RU or CUB/PS combinations. Varying failure patterns were seen in the $L_{(D)}L_{(CR)}$ and $NL_{(D)}NL_{(CR)}$ group specimens for both resin cement systems.

SEM observation of the dentin-resin cement-composite resin disk interfaces

The SEM images of the dentin–resin cement–composite resin disk interfaces revealed the thickness of the adhesive layer at the dentin–composite resin disk interfaces (Figs. 3 and 4). When the universal adhesives were light-cured at the flat dentin surface and the composite resin disks, the film thickness of SBU was about 5–10 μm, while that of CUB was<5 μm. The light-cured adhesives layers of SBU and CUB were markedly thinner than each cement thickness (*ca.* 30 μm). When the universal adhesives were not light-cured, the layers of the universal adhesives were not clearly visible, because it was mixed with the overlaying luting resin cements.

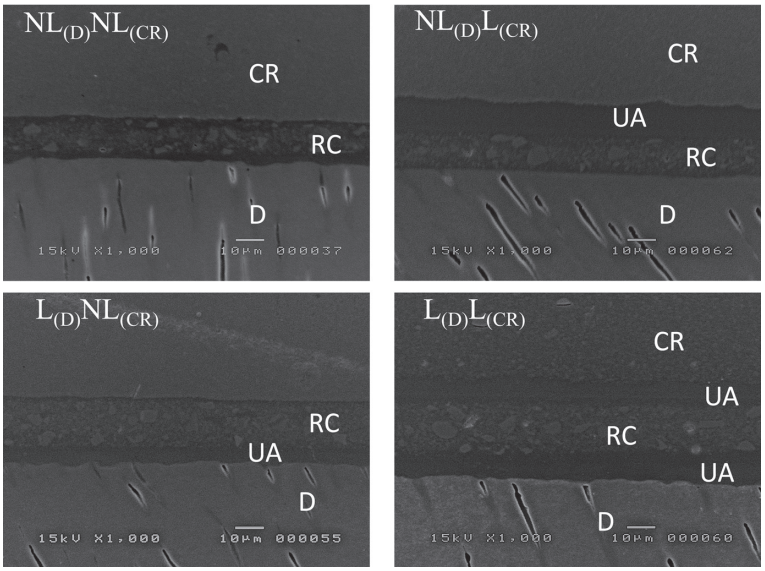

Fig. 3 Representative scanning electron micrographs of the adhesive and resin cement layer of specimens luted with SBU/RU in each curing mode group.
Each figure shows the dentin (D), composite resin disk (CR), universal adhesive (UA) and resin cement (RC).

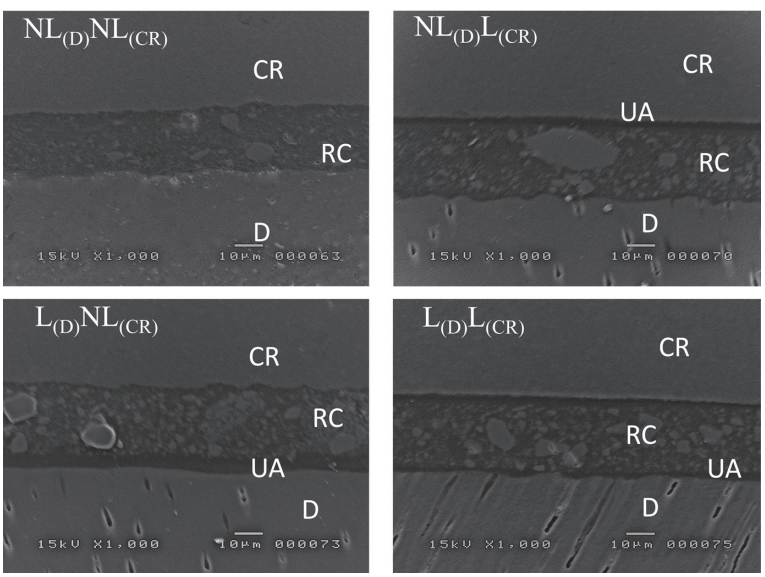

Fig. 4 Representative scanning electron micrographs of the adhesive and resin cement layer of specimens luted with CUB/PS in each curing mode group.
Each figure shows the dentin (D), composite resin disk (CR), universal adhesive (UA) and resin cement (RC).

DISCUSSION

In this study, we investigated the effect of 3 experimental factors, *i.e.*, the type of resin cement, the curing strategies used for the universal adhesive, and the effect of thermocycle aging, on the μTBS of resin cements luting composite resin disks to dentin. Significant differences in μTBS were found among all the experimental groups. Thus, both null hypotheses tested in this study, *i.e.*, that the μTBS would not be influenced by light-curing

of the adhesives of the resin cement, with or without thermocycle aging, and that μTBS would not be influenced by thermocycle aging, were rejected.

In the $L_{(D)}L_{(CR)}$ group, in which the adhesives on both composite resin disks and dentin surfaces were light-cured before luting, μTBS was statistically significantly higher for both resin cements, than for the other curing strategy groups. The higher μTBS is likely due to photo-polymerization of the adhesive resin before luting, which would improve the mechanical properties of each adhesive layer on the dentin and indirect composite resin surfaces[14]. In this study, after luting the composite resin disk onto the dentin, light irradiation of the resin cements was performed from the composite resin disk-side only, using a halogen light source at 600 mW/cm^2 for 40 s to simulate the clinical situation. A previous study reported that the intensity of transmitted light was decreased by 92% by composite resin disks with a thickness of 2 mm[15]. As the composite resin disk used in this study was 2 mm thick, with an opaque dentin shade (DA2), the effect of "indirect" transmitted light energy through the resin disk on the polymerization of the resin cements is likely to be limited. In a situation in which it is difficult for light to reach through the indirect restoration material, light activation of the adhesives prior to luting can impact the bonding performance of the resin cement more than chemical activation does[16,17].

Both the adhesive/resin cement combinations (SBU/RU and CUB/PS combinations) used in this study adopt a touch-curing system, in which the polymerization of the adhesive starts chemically when the adhesive comes into contact with the resin cement. However, we found that the effect of touch-curing between the resin cement and the universal adhesive on the μTBS was limited, because the μTBS in the $NL_{(D)}NL_{(CR)}$ group was significantly lower than that of the $L_{(D)}L_{(CR)}$ group. A previous study using resin core systems in combination with a corresponding touch-curing adhesive, also reported higher bond strengths to root canal dentin when using light-curing of the adhesives[18]. Therefore, the "direct" light irradiation of adhesives was more effective for improving μTBS of resin cement than only a touch-curing chemical polymerization reaction between the universal adhesive and resin cement.

Statistically significant interaction between materials and curing modes was revealed in this study ($p<0.01$). For SBU, the $L_{(D)}NL_{(CR)}$ and $NL_{(D)}L_{(CR)}$ specimens, in which the applied adhesives were light-irradiated on a single side only (either the dentin- or the composite resin disk- side) before luting, showed similar μTBS to that of the $NL_{(D)}NL_{(CR)}$ group. This could be because the breakage due to tensile load during μTBS testing occurred at the non-irradiated side, where the polymerization had not been improved, although the polymerization of the adhesive on the irradiated side had been improved. This was confirmed by the SEM failure mode analysis, where the predominant failures occurred at the interface between the composite resin disk and the resin cement in the $L_{(D)}NL_{(CR)}$ group, while the predominant failures occurred at the interface between dentin and resin cement in the $NL_{(D)}L_{(CR)}$ group.

The CUB group also showed a similar tendency in failure mode to the SBU group, in which breakage caused by tensile load during testing mainly occurred at the interface on the non-irradiated side. Nevertheless, the $NL_{(D)}L_{(CR)}$ group revealed significantly higher μTBS than did the $L_{(D)}NL_{(CR)}$ and $NL_{(D)}NL_{(CR)}$ groups. These results indicate that the bond strength value in the CUB group was dependent upon the polymerization behavior of the adhesive on the indirect composite resin- side. Acidic monomers in universal adhesives are thought to induce polymerization incompatibility with chemical-cure resin cement on dentin surfaces under acidic conditions[12]. According to the manufacturer's specifications, the pH of CUB (pH=2.3) is lower than that of SBU (pH=2.7). Self-etching adhesives (pH approximately 2 or above) partially demineralize the smear layer-covered dentin subsurface and increase the pH itself due to neutralization by calcium in demineralized hydroxyapatite crystals[12]. On the other hand, when one-step self-etching adhesives are applied to resin composite or ceramic surfaces, the pH of the surface remains unchanged. Therefore, it may be unavoidable that polymerization incompatibility would be induced on indirect restoration surfaces[19]. Indeed, it has been reported that the higher acidity of the surface decreases the bond strength of resin cement to indirect ceramic materials, due to the inhibition of the polymerization[20]. Thus, we speculate that the higher acidity of CUB might have strongly compromised the bond strength to indirect resin composite, as compared to SBU.

The bond strengths of both resin cements decreased after thermocycle aging, regardless of the curing strategies used for the adhesives. The decrease in bond strengths of adhesive resin cements to CAD/CAM composite resin blocks after thermocycle aging has also been reported previously[21]. In the specimens of the $L_{(D)}L_{(CR)}$ group, thermocycle aging significantly reduced the μTBS of CUB/PS-treated specimens, with an increase in cohesive failure in the cement, but it did not reduce the μTBS of SBU/RU-treated specimens. RU cement, used in combination with SBU, contains hydrophobic monomers. On the other hand, PS cement contains acidic functional monomers as well as hydrophobic monomers, which can be used together with CUB as a primer, and can also be used as a self-adhesive cement, without any pretreatment of the tooth substrates. Previous studies have reported that hydrophilicity and acidity of self-adhesive cements could cause water absorption, leading to reduced mechanical properties[22-24]. In this study, PS may have had an adverse effect on polymerization and allowed water absorption during thermocycle aging, resulting in reduced mechanical properties after thermocycle aging.

In $L_{(D)}NL_{(CR)}$ and $NL_{(D)}L_{(CR)}$ group specimens, for both resin cements, thermocycle aging decreased the bond strength, with increased interfacial failure on the non-irradiated side. The incomplete polymerization of the adhesive layer on the non-irradiated side could accelerate diffusion of water[25,26]. Water sorption causes

a softening of the resin polymer by swelling the network and reducing the frictional forces between the polymer chains, resulting in decreased mechanical properties. Moreover, increased water sorption decreases the mechanical properties over time[27], which would compromise the stability of bond strength[28]. Sato *et al.* have reported that adhesive layers with a higher degree of polymerization decrease the water sorption of the polymerized adhesives themselves, and increased the μTBS to dentin, and even after 1 year of water storage[29]. Therefore, in order to improve the bonding durability of resin cement, light-curing the universal adhesives after application to both tooth substrate and indirect resin composite is recommended.

Clinically, the cavity adaptation of the restoration should be taken into account. As light irradiation of the adhesives produces an adhesive layer on the adhesive substrate, a thicker adhesive layer may compromise the adaptation of indirect restorations[30,31]. It has been reported that the marginal and internal mean cement thicknesses of MOD inlays manufactured by using monolithic lithium disilicate, polymer-infiltrated ceramic, and nano-ceramic CAD/CAM materials ranged between 60.6 and 95.2 μm[32]. Additionally, another study reported marginal adaptation, ranging between 50 and 100 μm, for conventional crowns[33]. From the observation of the dentin–resin cement–composite resin sandwich specimens used in this study, the thickness of the light-cured adhesives layer depended on the materials used. The thickness of the light-cured SBU was about 5–10 μm, while the thickness of the light-cured CUB was <5 μm. When the universal adhesive applied on both sides are light-irradiated (*i.e.*, the $L_{(D)}L_{(CR)}$ group), the total thickness of the adhesive layer would not exceed 20 μm. This suggested that the light-curing universal adhesives applied to both the dentin- and indirect composite resin- sides may not affect the marginal adaptation in the indirect restoration. However, future studies should explore the cavity adaptation of the restoration in more complicated cavities when universal adhesives are applied to dentin surfaces and indirect restorations that are light-cured before luting.

CONCLUSIONS

Within the limitations of this study, the following conclusion can be drawn:

1. Light-curing universal adhesives (SBU and CUB) applied on both dentin- and indirect composite resin- sides resulted in the highest μTBS among the curing strategies used for the adhesives.
2. The effects of curing strategies used for universal adhesives on the bond strengths of the resin cements used to lute indirect resin composites to dentin were dependent upon the materials present within each bonding substrate (dentin or indirect resin composite), which might affect the polymerization behavior of the adhesive on each substrate.
3. Thermocycle aging decreased the μTBS of resin cements, particularly at the non-light-irradiated side, but it did not significantly decrease that of the SBU group after light-curing of the adhesive on both the dentin- and indirect composite resin-sides.

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