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Microtensile bond strengths of a dual-cure resin cement to dentin resin-coated with an all-in-one adhesive system using two curing modes

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This study evaluated the effect of resin coating using an all-in-one adhesive system on the dentin bond strength of a dual-cure resin cement after different curing modes. Human molars were ground to obtain flat dentin surfaces and divided into three groups: untreated as a control and resin-coated with either a single- or double-application of an all-in-one adhesive (Tokuyama Bond Force). The specimens were bonded to indirect composite disks using a dual-cure resin cement (Bistite II) activated by dual-cure or self-cure modes. Each specimen was sectioned into beams for the microtensile bond strength test. The data were analyzed by two-way ANOVA with Bonferroni's correction (p=0.05). Resin coating with a double-application of the all-in-one adhesive system significantly improved the bond strength of the dual-cure resin cement to dentin. In addition, dual-curing of the resin cement enhanced the bond strengths to dentin.

Keywords: Resin coating, Resin cement, All-in-one adhesive

INTRODUCTION

Exposed dentinal tubules after cavity preparation are permeable, since tooth preparations for a crown is usually carried out for intact dentin, which is more permeable than caries-affected dentin[1]. The closer the contents of dentinal tubules to the pulp, the more influenced they are by thermal stress and osmotic gradients, which causes tooth sensitivity[2] and pulpal inflammation due to the presence of bacteria on the prepared surface[3]. Therefore, optimal sealing of the prepared dentin is important in protecting the pulp tissue. The barrier-like film layer created by the adhesive material has the potential to minimize pulpal irritation and postoperative sensitivity.

Currently available resin cements do not always bond to dentin as strong as dentin bonding systems for direct resin composite restorations[4,5]. A relatively weak bond of resin cement may cause poor adaptation, gap formation[6] and postoperative sensitivity[7].

In the early 90s, a resin coating technique was introduced for indirect restorations to minimize pulpal irritation and postoperative sensitivity[8]. Resin coating in combination with a dentin bonding agent (DBA) and a low-viscosity resin composite (LVR) has been recommended for the prepared cavity immediately after tooth preparation, just before taking the final impression[9]. This technique also enables better bonding[10,11], sealing, adaptation to dentin[12], and durability of the indirect restoration[13]. However, the combination of a DBA and a LVR creates a layer of more than 100 µm thickness on the dentin[12], which is too thick for the coating of crown preparations.

In order for the resin coating technique to be applied after crown preparation, a much thinner coating film is required. An experimental, all-in-one adhesive, coating material, RZII (recently launched in the market as Hybrid Coat; Sun Medical, Moriyama, Japan) was developed to create a thin coating film on the dentin surface[5].

Previous studies have reported that resin coating with an all-in-one adhesive which improved the µTBS of resin cement to dentin beneath composite crown restorations[14], also prevented marginal leakage beneath full cast crowns[15] and reduced coronal leakage scores in endodontically treated teeth[16].

Recently, an alcohol-water based all-in-one adhesive system, Tokuyama Bond Force, was introduced to the market. It contains a unique acidic monomer so-called 3D-SR monomer (three dimensional self-reinforcing monomer), which has several functional groups per molecule, enabling a multiple point interaction with dentin calcium[17] [Tokuyama Technical Bulletin]. Therefore, this material may offer the possibility to create a thin coating material for the crown preparation.

Generally, dual-cure resin cements have routinely been used for the cementation of tooth-colored restorations because of their light transparency. Light irradiation of the adhesive cement is important to obtain good bonding even in the case of dual-cure resin cements. However, the thickness of the restoration can influence the polymerization of dual-cure resin cement[18].

There is little information on the dentin bonding performance of dual-cure resin cements used after resin

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coating with an all-in-one adhesive system. Therefore, the purpose of this study was to investigate the bond strengths of resin cements to human dentin resin-coated with an all-in-one adhesive system using two different curing modes.

The null hypotheses proposed were that the resin coating with an all-in-one adhesive system did not improve the microtensile bond strength of a dual-cure resin cement to dentin, and also that dual-curing did not increase the microtensile bond strengths to dentin.

MATERIALS AND METHODS

Materials used in this study

The resin coating material and the resin cement used in this study are listed in Table 1. For the resin coating, Tokuyama Bond Force was used, which is a fluoride-releasing one-bottle adhesive. The pH value is 2.3. For cementation, the dual-cure resin cement, Bistite II (Tokuyama Dental) was used, which consists of a two-step self-etch system and a resin cement. The pH value of the mixture of the self-etching primers 1A and 1B is 1.7.

Specimen preparation

The present study's protocol was designed according to the guidelines of the Ethics Committee of the Graduate School and Hospital, Tokyo Medical and Dental University.

Eighteen intact human molars, freshly extracted for clinical reasons, were used. The method of specimen preparation is illustrated in Fig. 1. Preparation of the tooth surfaces involved making a flat surface in superficial dentin on a model trimmer (Y-230; Yoshida, Tokyo, Japan). The region to be bonded was then finished with #600-grit silicon carbide paper under running water. The teeth were then randomly divided into three groups according to the surface treatment as follows: (1) the dentin surface was left untreated as a control, (2) Tokuyama Bond Force was applied to the dentin surface for 20 s, dried with strong air-dried after gentle air-dried, and light-cured of resin coating for 10 seconds using a halogen light curing unit (Optilux 501; Demetron Kerr, Danbury, CT, USA) (single-application group), and (3) after the first application and light-curing of resin coating, Tokuyama Bond Force was reapplied to the coated surface and light-cured with the same procedure as the first application (double-application group).

Following this, each specimen surface of all groups was cleaned with alcohol-soaked cotton pellets for 10 seconds, covered with a temporary filling material (Caviton; GC, Tokyo, Japan), and immersed in water at 37°C for 24 hours. After that, the temporary filling material was carefully removed from the surface with a spoon excavator. Then the surface was cleaned with alcohol-soaked cotton pellets for 10 seconds. For the resin-coated specimens, the surface to be bonded was cleaned with 38% phosphoric acid (Palfique Etching Agent; Tokuyama Dental) for 20 seconds, rinsed with water and air-dried to remove any debris, whereas for the control group, the dentin surfaces were left untreated.

Indirect composite resin disks (1 mm thick, 10 mm

Table 1 Materials used in this study

Material	pH	Batch No.	Material composition	Procedure
Resin coating material				
Tokuyama Bond Force	2.3	020067S	3D-SR monomer, TEGDMA, Bis-GMA, HEMA, Glass fillers, Isopropyl alcohol, Photo-initiator, Water	Apply for 20 s, strong air-blast after gentle air-blast, light cure for 10 s
Resin cement				
Bistite II	1.7	001037	Primer 1 (A and B): Phosphoric acid monomer, Acetone, Initiator Primer 2: HEMA, Acetone, Initiator Paste-A: NPGDMA, Bis-MPEPP, Silica-zirconia filler Paste-B: MAC-10, Silica-zirconia filler, Benzoyl peroxide, Photo-initiator	Apply Primer 1A+1B, leave for 30 s, air-dry, apply Primer 2, leave for 20 s, air-dry, place mixed Paste A+B, light cure for 20 s

Manufacturer: Tokuyama Dental, Tsukuba, Japan
3D-SR monomer: three dimensional self-reinforcing monomer; TEGDMA: triethylene glycol dimethacrylate; Bis-GMA: bisphenol-A- diglycidylmethacrylate; HEMA: hydroxyethyl methacrylate; NEPGDMA: neopentyl glycol dimethacrylate; Bis-MPEPP: 2,2-bis[4- (methacryloxy polyethoxy)phenyl]propane; MAC-10: methacryloxyundecane dicarboxylic acid; and BPO: bezoyl peroxide

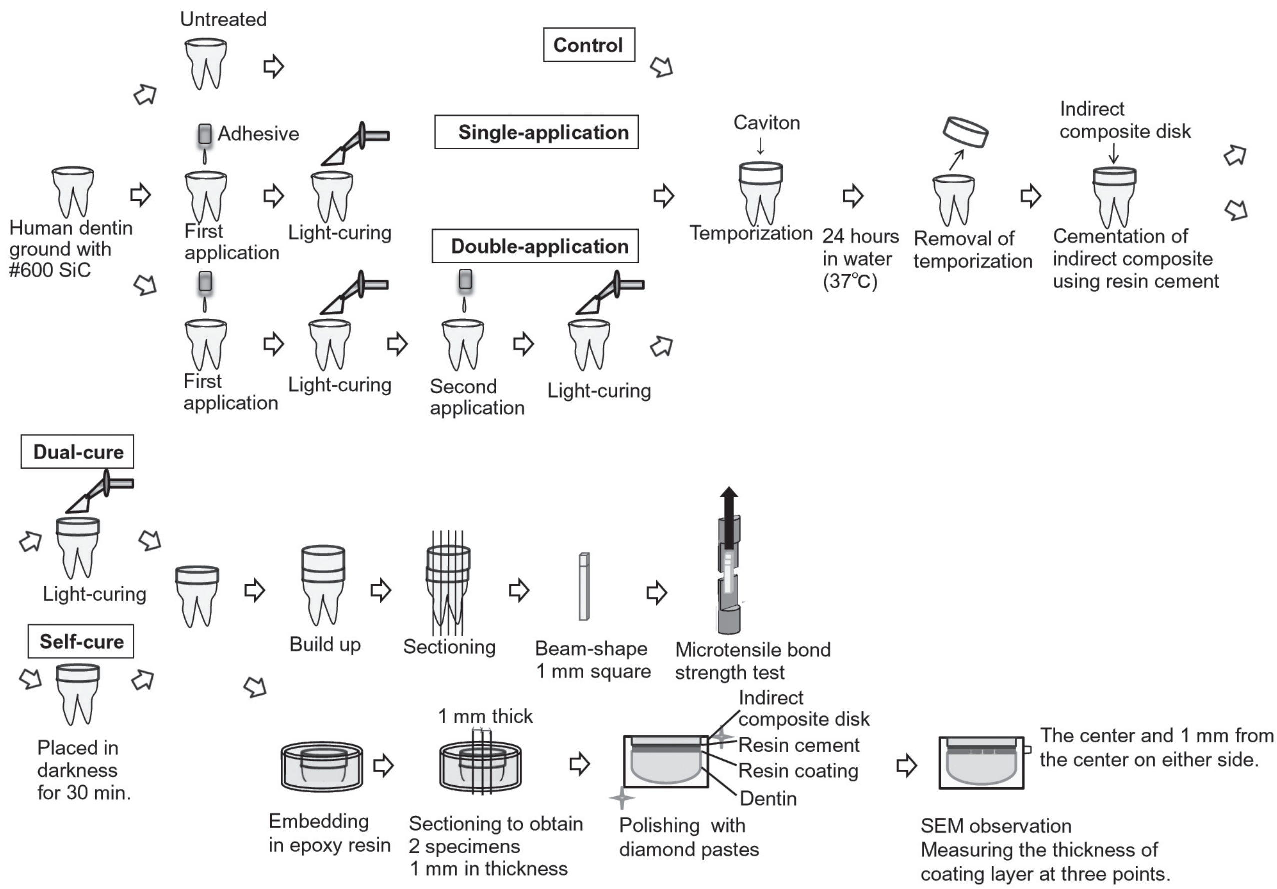

Fig. 1 Sample preparation for microtensile bond strength test and SEM observation of adhesive interface

diameter) were fabricated from a resin composite (Pearleste Shade DA2; Tokuyama Dental). The fabrication process entailed light curing for 1 minute using a halogen light curing unit (Optilux 501) and for 5 minutes using a laboratory light curing unit (Alpha Light II; J. Morita, Tokyo, Japan), and then using a heat curing unit at 100°C for 15 minutes in an oven (KL-100; Kuraray Medical, Tokyo, Japan). The surfaces of the resin composite were air-abraded using 70 µm Al_2O_3 particles (Hi Aluminas; Shofu, Kyoto, Japan) at 0.15 MPa at a distance of 10 mm from the surface for 5 seconds and cleaned ultrasonically for 2 minutes in distilled water. Then they were cleaned with 38% phosphoric acid (Palfique Etching Agent) for 20 seconds, rinsed with water and air-dried. A silane coupling agent (Tokuso Ceramic Primer; Tokuyama Dental) was applied to the surface and air-dried.

Following this, the surfaces of all the specimens were applied mixed primer 1A and 1B of Bistite II for 30 seconds, air-dried, applied primer 2 of Bistite II for 20 seconds and air-dried. The indirect composite disks were then bonded to the surface using cement paste of Bistite II with one of two curing modes; dual-cure and self-cure. For the dual-cure mode, each specimen was light-cured by a visible light-curing unit (Optilux 501) for 60 seconds from an occlusal direction. For the self-cure mode, each specimen was placed in darkness for 30 minutes in order to polymerize the cement by chemical activation only.

Microtensile bond strengths test

The bonded specimens were stored in water at 37°C for 24 hours. A direct composite resin (Clearfil AP-X Shade A2; Kuraray Medical) was built up incrementally to create a flat crown with a height of approximately 4 mm for the microtensile bond strength (µTBS) test. Each tooth was cross-sectioned longitudinally with a low-speed diamond saw (Isomet; Buehler, Lake Bluff, IL, USA) to obtain beam-shape specimens with an approximate surface area of 1×1 mm^2. Only the four central dentin sticks were used in order to eliminate substrate regional variability. The dimension of each beam was checked with a digital caliper (Mitutoyo CD-15C; Mitutoyo, Kawasaki, Japan) before the µTBS test.

Following this, each specimen was attached to a customized microtensile jig with cyanoacrylate adhesive (Zapit; Dental Ventures of America, Corona, CA, USA) and placed in a testing apparatus (EZ-test; Shimadzu,

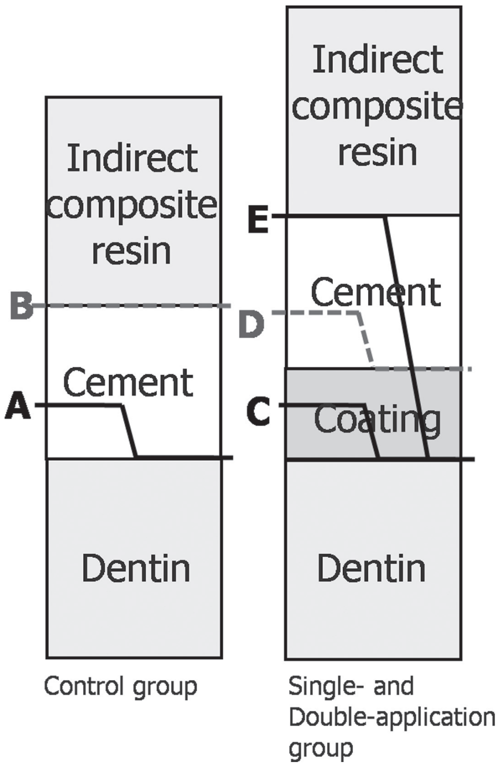

Fig. 2 Illustration of each failure mode

Kyoto, Japan) for the μTBS test at a crosshead speed of 1 mm/min. Twelve specimens were obtained from each group. The μTBSs were statistically analyzed by two-way ANOVA with Bonferroni's correction (p=0.05).

Failure modes

After debonding, the fractured specimens were gold sputter-coated and observed with a SEM (JSM-5310LV, JEOL, Tokyo, Japan) under ×100 magnification. The failure modes were classified into the following six categories as illustrated in Fig. 2;

Type A: Complete or partial adhesive failure at the interface between resin cement and dentin;

Type B: Failure at the interface indirect resin composite and resin cement;

Type C: Cohesive failure in coating or failure at the interface between resin coating and dentin;

Type D: Cohesive failure in resin cement or failure at the interface between resin cement and resin coating;

Type E: Cohesive failure in resin cement and resin coating or failure at the interface between indirect resin composite and resin cement.

SEM observation of adhesive interface

Specimen preparation is illustrated in Fig. 1. Each specimen was bonded in the same manner as described for μTBS test. The specimens were cut off 2 mm below the cemento-enamel junction, perpendicular to the long axis of the tooth with a low-speed diamond saw (Isomet). Then they were embedded in an epoxy resin (Epoxicure; Buehler). After curing the epoxy resin, each specimen was sectioned perpendicularly to the adhesive interface at 1 mm intervals to yield two specimens using a low-speed diamond saw (Isomet). Each specimen was subsequently polished with silicon carbide papers #600, #800, #1000, #1200, and #1500 under running water, and then finished with abrasive disks and diamond pastes (DP-Paste; Struers, Ballerup, Denmark) of 6, 3, 1, and 0.25 μm particle size. The specimens were cleaned ultrasonically in distilled water for 2 minutes at each step and dried at room temperature for 24 hours, and then subjected to argon-ion-beam etching (EIS-1E; Elionix, Tokyo, Japan) for 6 minutes at 0.2 mA and 1 kV to disclose the interfacial structure, gold sputter-coated and finally observed with an SEM. The thickness of the coating layers created by single- and double-applications was also measured at the same time. The mean value of the thickness of coating layer for each group was measured at three points; the center of the bonding interface and at a distance of 1 mm from the center on either side (Fig. 1). The mean value of the thickness of coating layers were calculated for each group (n=6).

RESULTS

Microtensile bond strengths

The μTBSs of the resin cement to dentin are summarized in Table 2. Two-way ANOVA indicated that dentin bond strength was influenced by the combination of number of application times (F=11.354, p<0.0001) and curing modes of resin cement (F=29.276, p<0.0001). The μTBS of the double-application groups were statistically higher than those of the single-application group and control groups in both dual-cure and self-cure modes. However, there were no statistical differences in μTBSs between the single-application and the control groups in both dual-cure and self-cure modes. Dual-cure mode provided statistically higher μTBSs than self-cure mode except for the single-application group. The highest μTBS was obtained by the double-application group using the dual-cure mode.

Failure modes

The failure modes distribution is summarized in Fig. 3. For the dual-cure mode, complete or partial adhesive failure at the interface between resin cement and dentin (Type A) was mainly observed in the control group, while cohesive failure in resin cement (Type D and Type E) was mainly observed in the single- and double-application groups.

In the case of the self-cure mode, both complete or partial adhesive failure at the interface (Type A) and failure at the interface between indirect composite resin and resin cement (Type B) were observed in the control group. Cohesive failure in resin cement, including failure at the interface between resin cement and resin coating material (Type D) was observed in single- and double-application groups.

SEM observations of adhesive interface

SEM observations of resin cement-dentin interface with and without the resin coating in the dual-cure mode are shown in Fig. 4. The thickness of the coating layer is summarized in Table 3. Good adaptation was found at the interface in all the groups. The thickness of the hybrid layer of the control group was approximately 2 to 4 µm (Figs. 4a and b). However, the hybrid layer was hardly detected even under ×5000 magnification in the resin coating groups (Figs. 4c to f). In the resin coating groups, the double-application group (Fig. 4e) created a thicker coating layer on the dentin surface compared to the single-application group (Fig. 4c). Regardless of the curing modes, a single-application and a double-application created approximately 8-12 µm and 14-20 µm thick layers on the dentin surface, respectively.

DISCUSSION

All-in-one adhesive systems contain a high concentration of solvents, such as acetone and alcohol, which facilitate wetting and spreading of the solvated comonomers[19]. However, residual solvents in the adhesives interfere with the polymerization of

Table 2 Mictotensile bond strength of resin cement to dentin (MPa)

	Control	Single-application	Double-application
Dual-cure	13.3±3.0^{A,a}	13.4±5.1^{A,c}	18.8±4.4^{B,e}
Self-cure	8.6±2.7^{C,b}	9.8±3.1^{C,d}	12.7±3.8^{D,f}

Mean±SD, *n*=12
Within the same row, means with the same large superscript letter are not statistically different (p>0.05)
Within the same column, means with the same small superscript are not statistically different (p>0.05)

Table 3 Thickness of the coating layer (µm)

	Single-application	Double-application
Tokuyama Bond Force	9.8±1.3	16.6±2.6

Mean±SD, *n*=6

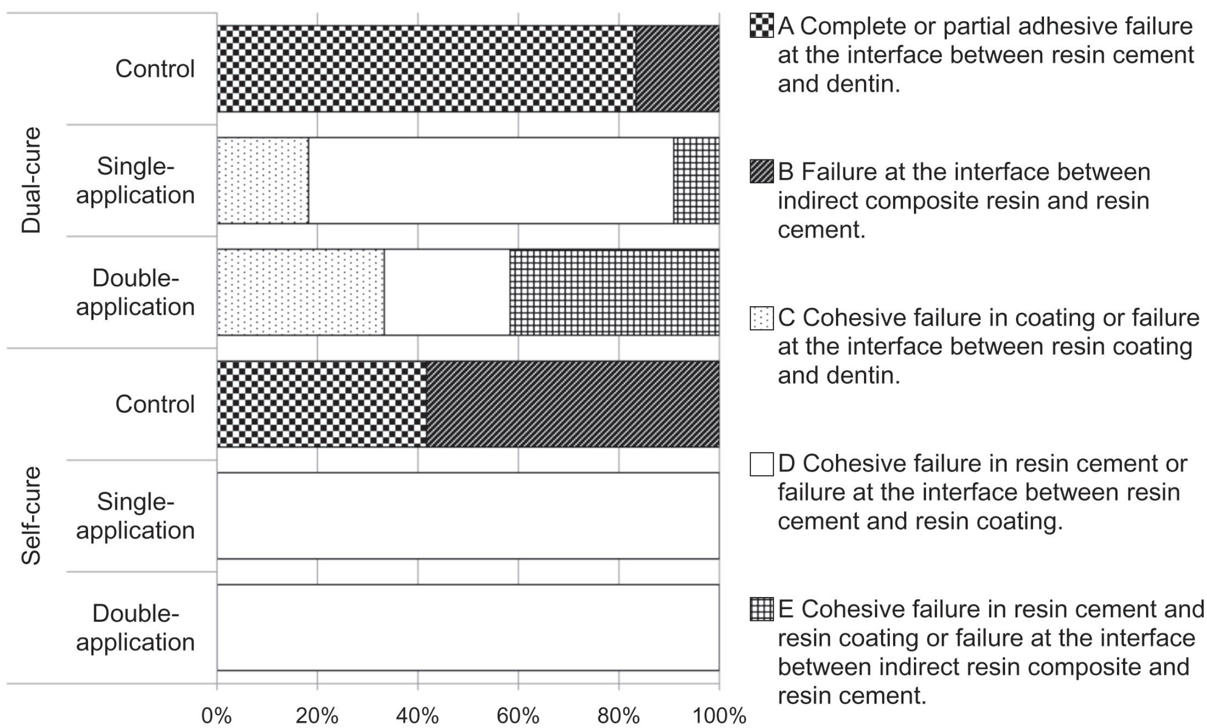

Fig. 3 The failure modes distributions

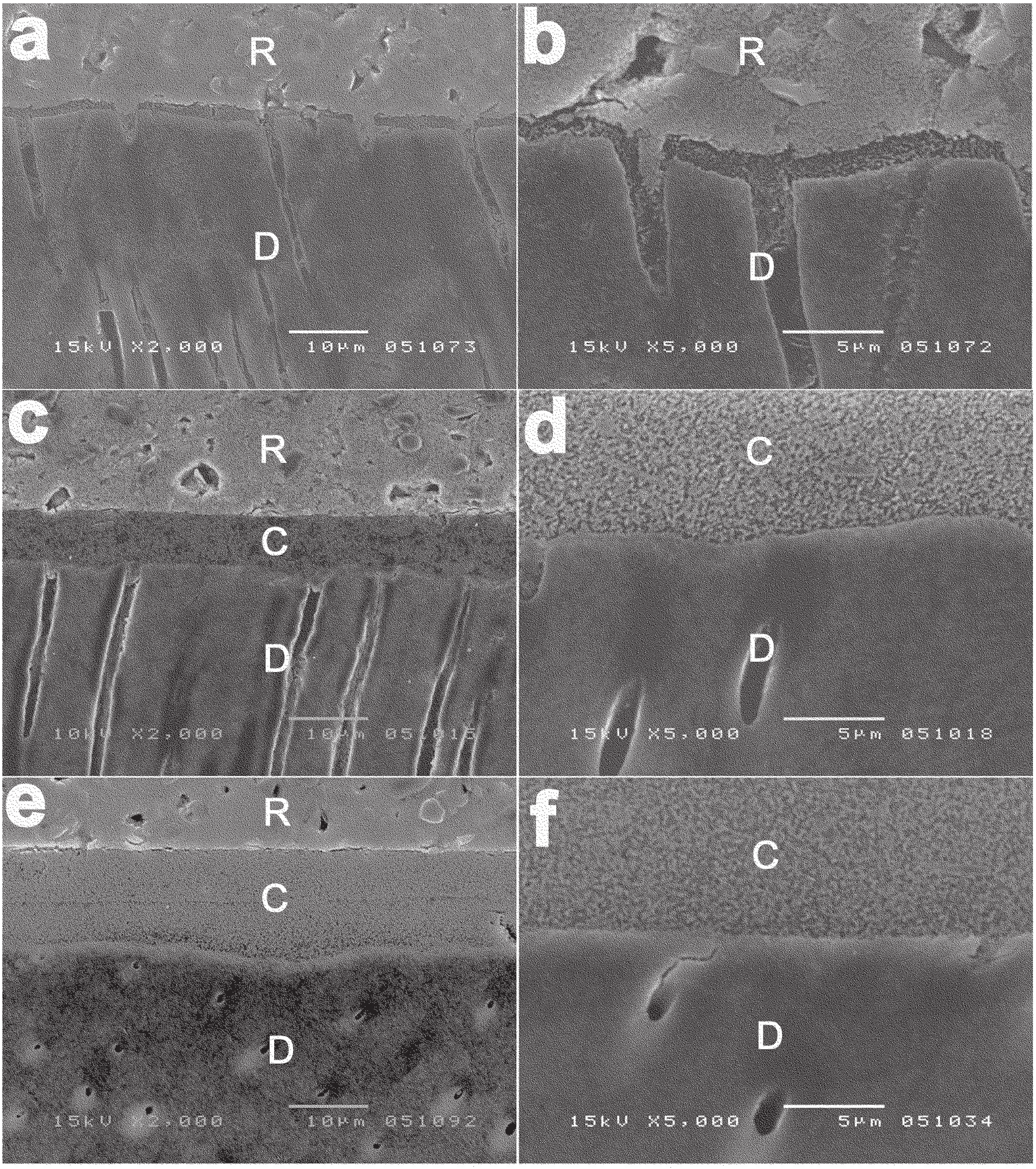

Fig. 4 SEM observations of resin cement-dentin interface with/without the resin coating with Tokuyama Bond Force using by dual-cure mode. (R: Bistite II, C: Tokuyama Bond Force, D: Dentin). (a) control group (×2000); (b) control group (×5000), the thickness of hybrid layer was approximately 2 to 4 μm; (c) resin coating with single-application of Tokuyama Bond Force (×2000), thicknesses of the resin coating layer were approximately 8 to 12 μm; (d) resin coating with single-application of Tokuyama Bond Force (×5000), the hybrid layer was hardly detected; (e) resin coating with double-application of Tokuyama Bond Force (×2000), thicknesses of the resin coating layer were approximately 14 to 20 μm; and (f) resin coating with double-application of Tokuyama Bond Force (×5000), the hybrid layer was hardly detected.

monomers and reduce the mechanical properties of the cured adhesive, leading to poor bonding performance[20]. Thus, they should be evaporated completely before light-curing. Since the solvent is evaporated by strong air-blowing, the thickness of the adhesive layer becomes an extremely thin film[19].

Tokuyama Bond Force is used as a thin coating material, which contains isopropyl alcohol as an organic solvent. The thickness of the coating layer created by a double-application of this adhesive was approximately 17 µm, which is approximately double of that created using the single-application. Therefore, the thickness of the resin coating with Tokuyama Bond Force is applicable to crown preparations, even with the double-applications.

In addition, the previous study demonstrated that a double-application of the adhesive significantly improved the bond strength to dentin, compared with the non-coated[5] and resin coating with single-application[19]. It was reported that the degree of polymerization and mechanical properties of bonding resin influence the bond strength to dentin[21]. Therefore, a double-application of the adhesive would improve the quality of the first applied adhesive layer in the resin coating[19]. Applying a second coating of the adhesive can eliminate the oxygen inhibited layer of the first coating, thus the uncured resin in the oxygen inhibited layer of the first adhesive layer is subsequently polymerized by diffusion of free radicals from the second adhesive layer[22].

It was reported that an all-in-one adhesive was applied to the root dentin surface as a root surface coating material in an attempt to prevent root dentin demineralization[23]. They reported that a double-application of the all-in-one adhesive, Hybrid Bond (Sun Medical), inhibited demineralization of dentin more effectively than a single-application. Interestingly, a double-application significantly increased the microhardness of the coating material, compared to the single-application[23]. This fact strongly suggests that a double-application of the adhesive improved polymerization of the first layer.

Previous studies have recommended a resin coating consisting of a combination of a DBA and a LVR, which significantly improved the bond strengths of resin cement to dentin compared to a single-application with a DBA[10]. They concluded that the combination of a DBA and a LVR could eliminate the oxygen inhibited layer of the first applied adhesive, since the oxygen inhibited layer is simultaneously polymerized by the diffusion of free radicals from the LVR. This explanation is also thought to account for the results of the present study.

The effect of the two curing modes, dual-cure and self-cure, on the bond strengths were also compared in the present study. Dual-cure resin cements are widely used in practice because they enable polymerization without the need for light exposure, which can be beneficial in the clinic. However, there may be an area of the restoration where the light cannot effectively reach. In addition, the opacity of restorative materials may also inhibit sufficient light energy from being transmitted to the resin cement[24]. Increasing the thickness of the composite restoration also attenuates light intensity through the restoration, affecting the bond strength of the dual-cure resin cement to dentin[18]. It was reported that the microhardness of dual-cure resin cement is affected by the curing mode[25]. A reduction in light energy results in poor mechanical properties and reduced bonding capacity of dual-cure resin cement to dentin[26,27].

Fracture mode analysis revealed that failure mode was influenced by application times and the curing mode. In the resin coating groups with the self-cure mode, the observed fracture mode was only Type D; cohesive failure in the resin cement and failure at the interface between resin cement and resin coating. This result suggested that only chemical cure of the resin cement could not enhance interfacial polymerization of dual-cure resin cement.

In the self-cure mode, the polymerization behavior at the interface between the resin coating layer and the chemical-cure resin cement may be adversely affected by their catalytic interaction[28-30]. Tokuyama Bond Force contains an acidic monomer, 3D-SR monomer to promote self-etching and monomer penetration properties[17]. However, it has been known that uncured acidic resin monomers which are present within the oxygen inhibition layers of the adhesive inactivate the aromatic tertiary amine derived from the chemical-cure composite, and reduce the quantity of free radicals diffusing through the oxygen inhibition layer of the adhesive[30]. Another possibility is due to presence of the phosphoric acid monomer contained in the primer applied prior to cementation. For the cementation to the resin coated surface, the procedures of primer application and gentle air-dry were carried out according to the manufacturer's recommendations. However, the phosphoric acid monomer in the primer may interfere the polymerization of the resin cement in the case of the self-cure mode.

The failure mode data indicate that the dentin surface was still covered by the resin coating in the resin-coated groups in both single- and double-application groups after testing. This fact is very important with regards to protection of the dentin and pulp in a clinical environment.

Adhesion of resin composite to dentin occurs as a result of hybrid layer formation[31], which is also true for resin cements[32,33]. The thickness of the hybrid layers produced by the primers of Bistite II in the control group was 2 to 4µm (Fig. 4b). The thicknesses of the hybrid layers in the resin-coated groups could not be determined in the present study (Figs. 4d and f). The reason for the difference in hybrid layer thickness is the pH value of primer. The weak acidity of Tokuyama Bond Force (pH 2.3) attributes to the less demineralization of the dentin surface compared to the primer of Bistite II (pH 1.7). However, a previous studies[34,35] suggested that there is no relationship between the thickness of the hybrid layer and the bond strength.

This *in vitro* study was performed as a screening test for selection of the materials for the resin coating. Therefore, further studies should be carried out to simulate the clinical situation such as dentinal fluid movement, cavity configuration for indirect restoration, and fatigue and thermal stress. Finally, clinical evaluation is necessary to establish the clinical application procedures for long-term success of the indirect restorations.

CONCLUSION

A resin coating consisting of a double-application of the all-in-one adhesive system significantly improved the bond strength of a resin cement to dentin. In addition, the dual-cure mode for curing of the resin cement enhanced the bond strengths to dentin.

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