Bond strength of resin composites to cavity floor and cavity wall dentin

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Purpose: To evaluate the effect of dentin location and dentinal tubule orientation on resin composite bond strength to dentin of the cavity floor and cavity wall using various adhesive systems.
Materials and Methods: Box-form cavities were prepared on human molars. Each specimen was restored with one of three adhesives Clearfil SE Bond, Single Bond, or Clearfil tri-S Bond followed by filling or buildup using Z100 resin composite. After light-curing at $600 \ mW/cm^2$ for 40 s, the specimen was cut perpendicular to the bonded surface parallel to the floor or wall to obtain beams. The microtensile bond strength to the cavity floor or wall specimens was determined. Data were analyzed using the Bonferroni test.
Results: Single Bond and Clearfil tri-S Bond showed significantly lower bond strength to the cavity floor compared with that of the cavity wall ($p < 0.05$). However, there was no significant difference in bond strength between the cavity floor and wall using Clearfil SE Bond ($p > 0.05$). Clearfil SE Bond showed significantly higher bond strength to the cavity floor than that of Single Bond and Clearfil tri-S Bond ($p > 0.05$).
Conclusion: Single Bond and Clearfil tri-s Bond bond strength to the cavity floor dentin was lower than to the cavity wall dentin. However, there was no significant difference in bond strength between the cavity floor and cavity wall using Clearfil SE Bond.

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Key Words: bond strength, dentin location, dentinal tubule orientation, resin composite

Introduction

Resin composite polymerization leads to volumetric shrinkage, and light-cured composites develop higher stresses in the cured material because the polymerization reaction occurs faster than in self-cured composites [1]. Therefore, the maximum interfacial stress generated at the cavity wall is two-fold greater in light-cured composite restorations than in self-cured composite restorations [2]. This stress has been shown to lead to greater gap formation between the resin and cavity surfaces than self-cured resin composite restorative materials.

Many reports of the measurement of resin composite bond strength to superficial flat dentin surfaces have shown that dentin bond strength decreases during bonding to deep dentin [4-8]. It has been reported that the resin composite bond strength was two-fold greater in the more superficial dentinal layers when compared with deeper portions [5]. Resin composite bond strength registered on dentin close to the pulp has also consistently been only 30%-40% of the strength found on peripheral dentin [5]. Remaining dentin thickness has an important influence on the reduction in bond strength of dentin bonding systems. Moreover, varying bond strengths to dentin, particularly deep dentin, have been reported with different adhesive systems [7,8].

In clinical application, most bonding substrates are the three-dimensional dentin walls of Class I-V cavities. The microtensile bond strength (μTBS) of resin composite that is bonded to a box-like Class I dentin cavity floor has been shown to be affected by cavity configuration (C-factor) and depth [7,9,10]. Furthermore, the resin composite bond and adaptation to the cavity wall is influenced by the dentinal tubule location and orientation [11].

Hybrid layer formation is considered essential for creating a strong bond between resin and dentin [12].

However, the thickness of the hybrid layer is less important when the resin composite is bonded to a dentin substrate that is perpendicular to flat dentin, and the bond strength between the resin and dentin is independent of the thickness of the hybrid layer [8,13]. Thus, we thought that it would be interesting to evaluate the bonding performance of adhesive systems with different curing modes to box-formed cavity walls and floors.

The purpose of this study was to evaluate the effects of dentin location, dentinal tubule orientation on resin composite bond strength to box-formed cavity floors and walls using various adhesive systems.

Materials and Methods

Specimen preparation

The materials, components, manufacturers, batch numbers, and bonding procedures used in this study are listed in Table 1. Eighteen intact, erupted, non-carious third molars that were frozen immediately after extraction were used in this study. These molars were collected in accordance with protocol No. 725, as approved by the appropriate institutional review board.

Table 1 Study materials

Material/Manufacturer	Components[a]	Batch No.	Bonding instruction[b]
Clearfil SE Bond (SE) (Kuraray Noritake Dental Co., Ltd., Tokyo, Japan)	Primer: HEMA, dimethacrylates, photoinitiator, water	00539A	a (20 s), b, c, d, e (10 s)
	Bond: MDP, HEMA, Bis-GMA· dimethacrylates, photoinitiator, microfiller	00760A	
Single Bond (SB) (3M ESPE, St. Paul, MN, USA)	Uni-etch: 35% phosphoric acid	4JA	f (15 s), g, h, i, e (10 s)
	Bond: Bis- GMA, HEMA, dimethacrylates, methacrylates, pendent polyalkenoic acid copolymer, photoinitiator, ethanol, water	3KF	
Clearfil tri-S Bond (TS) (Kuraray Noritake Dental Co., Ltd.)	Bond: MDP, HEMA, Bis-GMA, photoinitiator, water, ethanol	00083A	c (5 s) d, e
Z100 (3M ESPE)	Bis-GMA, TEGDMA, dimethacrylate polymer, zirconia / silica filler, photo initiator, Filler load: 84.5 wt %	4NJ	e (40 s)

[a]Abbreviations: HEMA, 2-hydroxyethylmethacrylate; MDP, 10-methacryloyloxydecyl dihydrogen phosphate; Bis-GMA, bisphenyl-glycidyl-methacrylate
[b]Procedures: (a) apply primer; (b) dry with gently air-blowing; (c) apply adhesive; (d) gently air blow (e) light-cure; (f) acid-etch; (g) rinse with water; (h) blot-dry; (i) apply 2 coats of adhesive

The occlusal enamel (Fig. 1A) was ground away using a model trimmer under running water to expose a flat dentin surface, which was then wet-ground with #600 SiC paper. Box-form cavities (3 mm wide × 5 mm long × 2 mm deep) were prepared on the flat dentin surfaces using a diamond point (#211, ISO #110 014; Shofu Inc., Kyoto, Japan) with copious water spray and were finished with a carbide steel bur (#600, ISO #071 012; Dentech Co., Tokyo, Japan) (Fig. 1B). Each specimen was restored with one of three adhesives: Clearfil SE Bond (Kuraray Noritake Dental, Co., Ltd., Tokyo, Japan), Single Bond (3M ESPE, St. Paul, MN, USA), and Clearfil tri-S Bond (Kuraray Noritake Dental, Co., Ltd.). A Z100 resin composite (Shade A3; 3M ESPE) was then used to fill in the cavities (Fig. 1B). The resin composite was light-cured at 600 mW/cm^2 for 40 s using an experimental quartz-tungsten halogen light-curing unit (GC Corp., Tokyo, Japan) that was connected to a slide

regulator and had a control system for lamp voltage and an adjustable light intensity, with a light tip (diameter, 7 mm). The light intensity on the surface of the specimens was measured using a curing radiometer (Model 100; Demetron Research Co., Danbury, CT, USA).
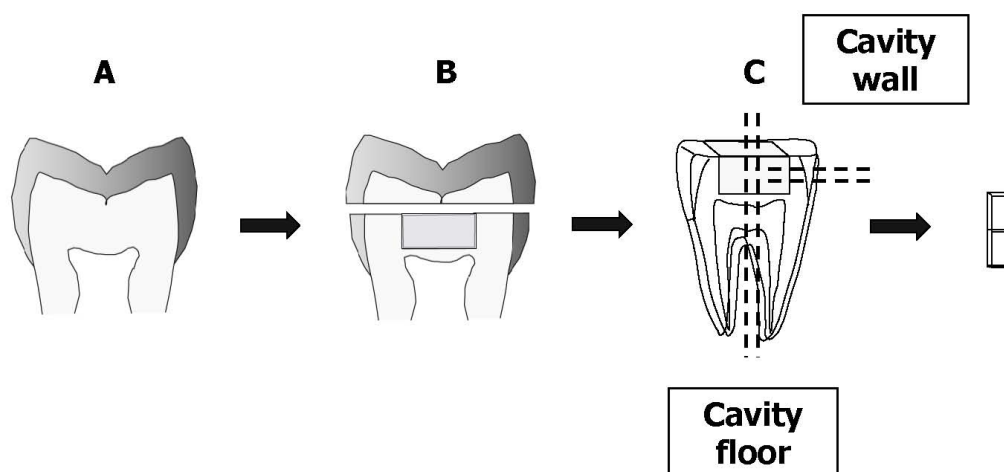

Fig. 1 Preparation of a cavity bonding substrate

Tensile bond strength measurement

The specimens were stored in water maintained at 37°C in the dark for 24 h. Then, the restored floor/wall specimens were sectioned perpendicular to the bonded surfaces using a diamond saw (Isomet, Buehler Co., Lake Bluff, IL, USA) under copious water lubrication (Figs. 1C). Each slab was cut into beams with a bonded area of approximately 0.9 mm^2 using a diamond saw under copious water lubrication. The trimmed specimens were mounted on a µTBS jig (KDA, Tokyo, Japan) with cyanoacrylate adhesive (Model Repair II Blue; Dentsply-Sankin Co., Ohtawara, Japan) and stressed to failure under tension at 1 mm/min in a universal testing machine (EZ test; Shimadzu, Kyoto, Japan). Each specimen was then inspected using a scanning electron microscope (SEM) to determine the mode of fracture. The bond strength to floor and wall dentin were statistically analyzed using the Fisher's PLSD test at a significance level of 5%.

SEM observation of fractured surfaces

After the tensile bond test, each fractured dentin specimen was fixed in 10% neutral buffered formalin [14]. The dentin and composite-paired specimens were then trimmed and placed on SEM stubs, coated with gold-sputter, and observed using an SEM (JSM-5310LV; JEOL, Akishima, Japan) to microscopically assess the patterns of failure. The fractured surfaces were classified into one of four groups: interfacial failure; mixed failure; cohesive failure within the resin (adhesive layer or composite); and cohesive failure within the dentin.

Results

The tensile bond strength results are summarized in Table 2. Single Bond and Clearfil tri-S Bond showed significantly lower bond strength to the cavity floor than to the cavity wall ($p < 0.05$). However, there was no significant difference in bond strength with Clearfil SE Bond between the cavity floor and wall ($p > 0.05$). Overall, bond strength was significantly lower for Clearfil tri-S Bond than Clearfil SE Bond ($p < 0.05$).

Clearfil SE Bond showed significantly higher bond strength to the cavity floor compared with that of Single Bond and Clearfil tri-S Bond ($p > 0.05$). Single Bond showed significantly higher bond strength to the cavity

floor compared with that of Clearfil tri-S Bond ($p > 0.05$). Clearfil SE Bond and Single Bond showed significantly higher bond strength to the cavity wall compared with that of Clearfil tri-S Bond ($p > 0.05$). However, there was no significant difference in bond strength between Clearfil SE Bond and Single Bond to the cavity wall ($p > 0.05$).

**Table 2** Mean tensile bond strength of the bonding system to the cavity floor and cavity wall dentin

| Type of substrate / Material	Tensile bond strength (MPa)		
	Clearfil SE Bond	Single Bond	Clearfil tri-S Bond
Cavity wall	50.1 (6.1) [A]	46.1 (6.6) [a, B]	30.0 (2.7) [a, A, B]
Cavity floor	50.1 (2.9) [A]	29.7 (2.8) [a, A]	20.6 (1.8) [a, A]

Same lower-case superscript letters indicate significant differences in the strength of the bonding substrates ($p < 0.05$).
Same upper-case superscript letters indicate significant differences in the strength of the bonding systems ($p < 0.05$).

Table 3 Failure mode

		Interfacial failure	Mixed failure	Cohesive failure in resin	Cohesive failure in dentin
SE	Cavity wall	1	4	1	0
	Cavity floor	1	5	0	0
SB	Cavity wall	6	0	0	0
	Cavity floor	6	0	0	0
TS	Cavity wall	3	1	2	0
	Cavity floor	4	1	0	1
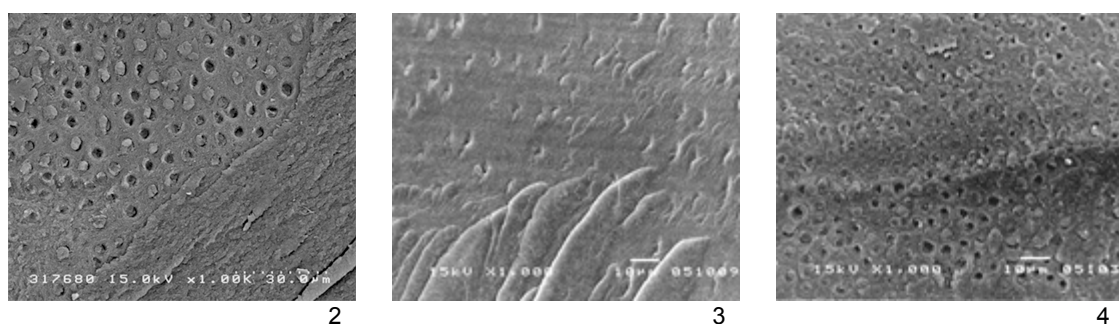
Fig. 2 Dentin side of fractured specimen to cavity floor by Clearfil SE Bond. Failure mode indicated mixed failure.
Fig. 3 Dentin side of fractured specimen to cavity wall by Clearfil SE Bond. Failure mode indicated mixed failure.
Fig. 4 Dentin side of fractured specimen to cavity floor by Single Bond. Failure mode indicated interfacial failure at the top of the hybrid layer.
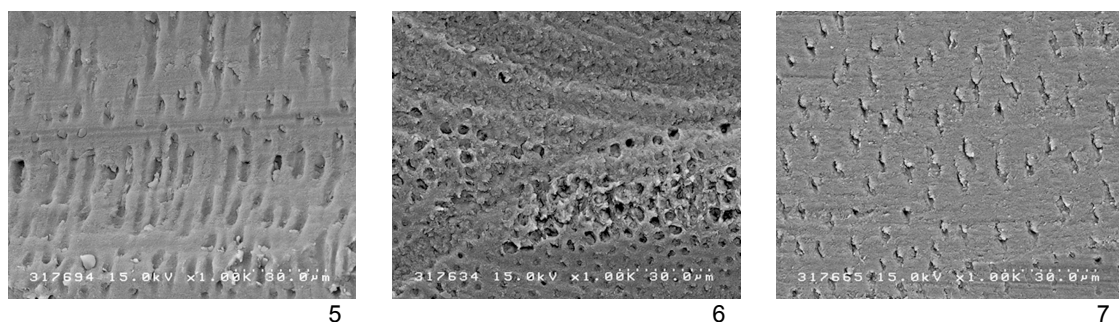
Fig. 5 Dentin side of fractured specimen to cavity wall by Single Bond. Failure mode indicated interfacial failure at the top of the hybrid layer.
Fig. 6 Dentin side of fractured specimen to cavity floor by Clearfil tri-S Bond. Failure mode indicated interfacial failure.
Fig. 7 Dentin side of fractured specimen to cavity wall by Clearfil tri-S Bond. Failure mode indicated interfacial failure.

The failure mode results are summarized in Table 3. Most of the Clearfil SE Bond specimens showed mixed failure of the cavity floor (Fig. 2) and walls (Fig. 3). All of the Single Bond specimens showed interfacial failure at the top of the hybrid layer of the cavity floor (Fig. 4) and walls (Fig. 5). Most of the Clearfil tri-S Bond specimens showed interfacial failure of the cavity floor (Fig. 6). Most of the Clearfil tri-S Bond specimens showed interfacial failure (Fig. 7) and cohesive failure in resin of the cavity walls.

Discussion

In descending order, bond strength of the materials to the cavity floor was as follows: Clearfil SE Bond > Single Bond > Clearfil tri-S Bond. The Single Bond adhesive system uses a phosphoric acid etching agent. In the case of an etching agent, the bonding material may not fully infiltrate the collagen fibril network of the demineralized dentin. Failure of the resin to adequately penetrate the collagen network in deeply etched dentin will produce a porous zone at the hybrid layer base, resulting in a weak porous hybrid layer zone that is susceptible to degradation of the resin-dentin bond. Conversely, the self-etching primer system appears to allow the bonding resin to completely penetrate the demineralized dentin. Thus, the self-etching primer system provides a high quality resin-impregnated layer that contributes to a strong bond between the bonding system and tooth wall. The hybrid layer of Single Bond is about 2-6 times thicker than that of Clearfil SE Bond [15]. The quality of a hybrid layer, rather than quantity, is considered more important for obtaining a good resin-dentin bond [16,17]. These findings are in agreement with an earlier study showing that the bond strength between resin and dentin was independent of hybrid layer thickness [8,13].

There was no significant difference in bonding to the cavity wall between Clearfil SE Bond and Single Bond. However, Clearfil tri-S Bond showed significantly weaker bond strength to the cavity wall than that of Clearfil SE Bond and Single Bond. Failure mode of Clearfil SE Bond showed an almost mixed failure of bonding to the cavity wall. Most of Clearfil tri-S Bond specimens showed interfacial failure and cohesive failure in the bonding layer. One-step self-etching systems are more hydrophilic and water absorbent than two-step self-etching systems [18]. Evaporating water from the one-step adhesives is difficult, and even if evaporation is successful, water rapidly diffuses back from the bonded dentin into the adhesive resin [19], resulting in water sorption plasticized polymers and increases solubility, and decreases modulus of elasticity [18] and mechanical properties of the polymer [20]. Therefore, the one-step self-etching system Clearfil tri-S Bond showed cohesive failure in the bonding layer and lower bond strength.

Single Bond and Clearfil tri-S Bond showed significantly lower bond strength of the cavity floor than that of the cavity wall. Bond strength was significantly higher with parallel tubules than with perpendicular tubules. The intertubular dentin has smaller dimensions in the floor than in the walls because the dentinal tubules are oriented almost parallel to wall dentin. The hybrid layer is reportedly thinner in areas with parallel tubules [11], confirming that resin-dentin bond strength is not affected by hybrid layer thickness, which supports the findings of previous studies [8,13].

There was no significant difference in bond strength between the cavity floor and wall for Clearfil SE Bond. This was believed to be because of the effect of a difference in adhesive layer thickness. Clearfil SE Bond contains microfillers in the bonding resin, and the thickness of the adhesive resin layer has been shown to range from 40 to 200 μm [21]. Conversely, Single Bond produces a thin film of unfilled adhesive at 30-40 μm [22]. Thus, the thick adhesive resin layer of Clearfil SE Bond was likely to absorb some of the shrinkage stress that

occurred during light curing of the resin composites [23].

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