

Dentin Bond Strengths of Three Adhesive/Composite Core Systems using Different Curing Units

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This study evaluated the tensile bond strengths of three adhesive/composite core materials to bovine dentin using three different curing units. Bovine dentin surfaces were ground with 600-grit SiC paper. Bonding area was demarcated with a vinyl tape (4-mm-diameter hole). Three adhesive/composite core systems — S6054 (experimental), UniFil Core, and Clearfil DC Core Automix — were used with three curing units — Curing Light XL3000 (quartz-tungsten-halogen), Hyper Lightel (high-power quartz-tungsten-halogen), and LEDemetron1 (blue light-emitting diode) — according to manufacturers' instructions. After 24 hours of storage in water at 37°C, tensile bond strengths were measured at a crosshead speed of 2 mm/min. Results were statistically analyzed with one-way ANOVA and Tukey's HSD test ($p < 0.05$). Highest tensile bond strength was obtained using Clearfil DC Core Automix with Hyper Lightel.

Keywords: Tensile bond strength, Composite core, Curing units

INTRODUCTION

Recently, owing to widespread acceptance and adoption of minimal intervention dentistry, composite core systems are concomitantly widely used in clinics to preserve sound tooth structure and prevent root fracture of non-vital teeth. Since dentin bonding systems have been shown to reinforce the remaining tooth structure¹⁻⁵, non-vital teeth can be built up without removing undercuts in post holes. Further, in contrast with metal posts, the elastic modulus of a composite core material is similar to that of dentin^{6,7}. This favorable property is therefore a boost to saving the remaining tooth structure from root fracture⁸.

Selecting a composite core system, comprising a composite core material and an adhesive system, is an important step toward the clinical success of restorations in endodontically treated teeth. Recent self-etching primer adhesive systems have demonstrated good bonding to root canal dentin^{9,10} and pulpal floor dentin^{11,12} as well as to coronal dentin. However, several clinical factors affect the bond strength to endodontically treated teeth, such as root dentin region (apical, middle, or coronal)^{13,14} and root dentin condition (normal *versus* sclerotic dentin)^{15,16}. Besides, the chemical irrigants and medicaments used during root canal treatment have been shown to further reduce bond strength to dentin^{17,18}.

As for the polymerization of adhesive/composite systems, several curing modes are currently available: light curing, chemical curing, and dual curing. On light polymerization, it has two advantages over

chemical polymerization: extended working time and polymerization on demand. On the other hand, light polymerization is not sufficient in deeper regions, such as the bottom of a post cavity, because of limited light energy transmission through the material. As a result, dual curing emerges as the most popular curing mode in composite core foundation systems, which are able to polymerize chemically in regions without light transmission. Nonetheless, light polymerization of composite resins is essential to achieving good bonding even for dual-cure composite core systems. It has been reported that the dentin bond strength of a dual-cure adhesive system without light curing was significantly lower than that with light exposure¹⁹. Although a chemically cured adhesive is beneficial for deep cavities in non-vital teeth, no successful composite core systems with a solely chemically cured adhesive have been reported to-date¹⁹.

Further on light curing, the quartz-tungsten-halogen (QTH) lamp has been the most popular light curing unit (LCU) in the clinic — although it is time-consuming. In addition, it has been reported that the QTH LCU is associated with degradation of the bulb and light reflector, broken filters, breakdown of optical fibers, and tip damage²⁰, which can lead to a reduction in light output over time. Besides QTH LCU, various types of LCUs are also currently available — namely plasma arc curing unit (PAC), light amplification by stimulated emission of radiation (Laser), and light-emitting diode (LED). Each LCU has a different intensity, spectral output, and curing

mode²¹).

To achieve a successful composite core build-up, it is desirable — if not mandatory — to attain a good match between material formulation and LCU characteristics. Therefore, the purpose of this study was to evaluate the dentin bond strengths of three adhesive/composite core systems using three different curing strategies. The hypothesis of this study was that the tensile bond strength to dentin would be affected by the composite core system and curing strategy.

MATERIALS AND METHODS

Materials used in this study

The three composite core systems used in this study were: S6054 (experimental, Dentsply-Sankin, Tokyo, Japan), UniFil Core (GC, Tokyo, Japan), and Clearfil DC Core Automix (Kuraray Medical, Tokyo, Japan). Table 1 lists their basic formulations and bonding procedures. The three LCUs used were: Curing Light XL3000, (QTH, 650 mW/cm², 3M ESPE, St. Paul, MN, USA), Hyper Lightel (high-power QTH,

Table 1 Composite core systems used in this study

Material	Manufacturer	Lot.No	Adhesive			Composite	
			Curing Mode	Component	Directions	Curing Mode	Component
S6054 (Experimental)	Dentsply-Sankin, Tokyo, Japan	—	Chemical	Primer, 4-MET, HEMA, ethanol, Water	Apply 20sec, dry	Light	UDMA, 2.6E, TEGDMA, CQ, SiO ₂ , fluoroaluminosilicate glass
				Bond; 4-MET, HEMA, UDMA, dimethacrylate, chemical initiator, ethanol	Apply A + B		
UniFii Core	GC, Tokyo, Japan	0410041	Dual	Liquid A: 4-MET, silicon dioxide, dimethacrylate resin, initiator, ethanol	Apply, A + B 5sec, leave for 30s, dry, light cure	Dual	UDMA, dimethacry- late, Fluoroaluminio- silicate glass, photo/ chemical initiators
				Liquid B: initiator, ethanol			
Clearfil DC Core Automix	Kuraray Medical, Tokyo, Japan	011115	Dual	LB primer, HEMA, MDP, photoinitiator, water	Apply A + B, 30sec dry,	Dual	Bis-GMA, TEGDMA, dimethacrylate, filler, photo/chemi- cal initiators
				LB bond; HEMA, MDP, Bis-GMA, filler, photo/ chemical initiator	Apply A + B, dry, light cure		

4-MET: 4-methacryloyloxyethyl trimellitate; HEMA: 2-hydroxyethyl methacrylate; UDMA: urethane dimethacrylate
MDP: 10-methacryloyloxy methyl methacrylate; Bis-GMA: bisphenol-A diglycidylmethacrylate
2.6E: 2,2-bis(4-methacryloyloxyphenyl)propane; TEGDMA: triethylene glycol dimethacrylate

Table 2 Curing strategies of adhesive/composite systems evaluated in this study

Light curing unit	Light source	Light intensity (mW/cm ²)	Curing time(second)					
			S6054		UniFill Core		Clearfil DC Core Automix	
			Adhesive (Chemical)	Composite (Light)	Adhesive (Dual)	Composite (Dual)	Adhesive (Dual)	Composite (Dual)
Curing Light XL3000 (3M-ESPE, St. Paul, MN, USA)	Quartz-tung- sten halogen	650	0	20	10	10	20	20
Hyper Lightel (Ushio Electric, Tokyo, Japan)	Quartz-tung- sten halogen	1300	0	10	5	5	10	10
LEDemetron1 (Sybron Dental Speciali- ties/Kerr, west Collins, Orange, CA, USA)	Blue light emitting diode	1660	0	5	5	5	5	5

1300 mW/cm², Ushio Electric, Tokyo, Japan), and LEDemetron1 (LED, 1660 mW/cm², Sybron Dental Specialties/Kerr, West Collins, Orange, CA, USA). Table 2 shows the irradiation time of each adhesive and composite system according to the manufacturers' instructions.

Wavelengths of the light curing units were measured using a spectroradiometer (USR-40V-01, Ushio Electric, Tokyo, Japan). Each light curing unit was warmed up for 30 seconds before commencement of each test. The end of the light guide was placed in contact with the center of the measuring window of the curing radiometer at right angles. Each reading of the maximum power output was acquired within five seconds after light passed through the measuring window.

Specimen preparation

Specimen preparation is illustrated in Fig. 1. Freshly extracted bovine teeth were used as test substrates. Preparation of tooth surfaces was carried out by first preparing a flat surface in superficial dentin with a model trimmer under copious water lavage. Then, the dentin surface was wet-ground with 600-grit silicon carbide paper to create a uniform smear layer and surface roughness. The area for bonding was demarcated by affixing a piece of vinyl masking tape (0.15 mm thick) with a 4-mm-diameter hole. Following this, the dentin surface was bonded using one of the three bonding systems and composites with three different light curing strategies according to the manufacturers' instructions listed in Tables 1 and 2. Before light curing the composite core, each composite was placed on the bonding resin, covered with a plastic matrix strip, and pressed flat with a glass slide. A stainless steel rod (8 mm in diameter and 25 mm in height) was then cemented to the surface of the cured composite using a resin cement (Panavia F, Kuraray Medical, Tokyo, Japan).

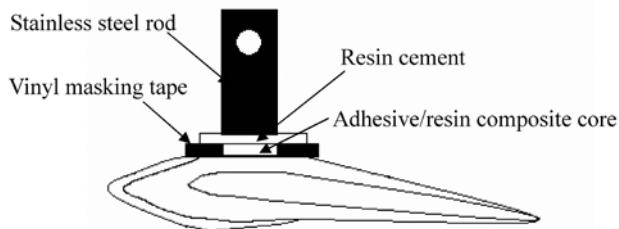


Fig. 1 Specimen for tensile bond strength test.

Tensile bond strength test

After the specimens were stored in water at 37°C for one day, tensile bond strengths were measured using a universal testing machine (Autograph AG-500B, Shimadzu, Kyoto, Japan) at a crosshead speed of 2 mm/min.

The number of specimens was 10 for each group. Results were statistically analyzed with one-way ANOVA and Tukey's HSD test ($p < 0.05$).

Failure mode analysis

Fractured specimen surfaces after debonding were observed by visual inspection. Several representative examples were then selected from each group for failure mode observation by a SEM (5310LV, JEOL, Tokyo, Japan). Fracture modes were classified into three categories as follows: A—Adhesive failure; B—Mixed failure including adhesive failure and cohesive failure in dentin; C—cohesive failure in dentin.

RESULTS

Table 3 shows the tensile bond strengths of the three composite core systems to dentin. Statistical analysis results of the dentin bond strengths are summarized in Table 4. In S6054 and UniFil Core systems, there were no significant differences in bond strength among the three different curing strategies. However, for each curing strategy, the mean tensile bond strength of S6054 was significantly higher than that of UniFil Core. As for the combination of Clearfil DC Core Automix with Hyper Lightel, it provided the highest dentin bond strength for each curing strategy among all the groups ($p < 0.05$).

Figure 2 shows the representative SEM images of the fracture modes after tensile bond testing. Table 5 then summarizes the failure modes of each group. For S6054, half of the failure modes were adhesive failure (mode A in Fig. 2a), while the other half were mixed failure including adhesive failure and cohesive failure in dentin (mode B in Fig. 2b). For UniFil Core, 80% of the failures were adhesive failure for each curing strategy. For Clearfil DC Core Automix, mixed failure (mode B) and cohesive failure

Table 3 Tensile bond strengths to bovine dentin (MPa)

	S6054	Unifil Core	Clearfil DC Core Automix
Curing Light XL3000	11.1 ± 1.2	8.0 ± 1.5	13.9 ± 2.3
Hyper Lightel	12.4 ± 3.1	9.3 ± 1.8	18.2 ± 2.4
LEDemetron1	13.7 ± 3.1	9.2 ± 1.9	14.9 ± 2.2

mean ± SD (n = 10)

Table 4 Summary of the statistical analysis of the bond strengths to bovine dentin using ANOVA supplemented with Tukey's HSD test

Adhesive		S6054			UniFil Core			Clearfil DC Core Automix		
Adhesive	LCU	Curing Light XL3000	Hyper Lightel	LED emetron1	Curing Light XL3000	Hyper Lightel	LED emetron1	Curing Light XL3000	Hyper Lightel	LED emetron1
S6054	Curing Light XL3000									
	Hyper Lightel	n.s								
	LEDemetron1	n.s	n.s							
UniFil Core	Curing Light XL3000	n.s	*	*						
	Hyper Lightel	n.s	n.s	*	n.s					
	LEDemetron1	n.s	n.s	*	n.s	n.s				
Clearfil DC Core Automix	Curing Light XL3000	n.s	n.s	n.s	*	*	*			
	Hyper Lightel	*	*	*	*	*	*	*		
	LEDemetron1	*	n.s	n.s	*	*	*	n.s	*	

ns: no significant differences; *: p<0.05 LCU; Light curing unit

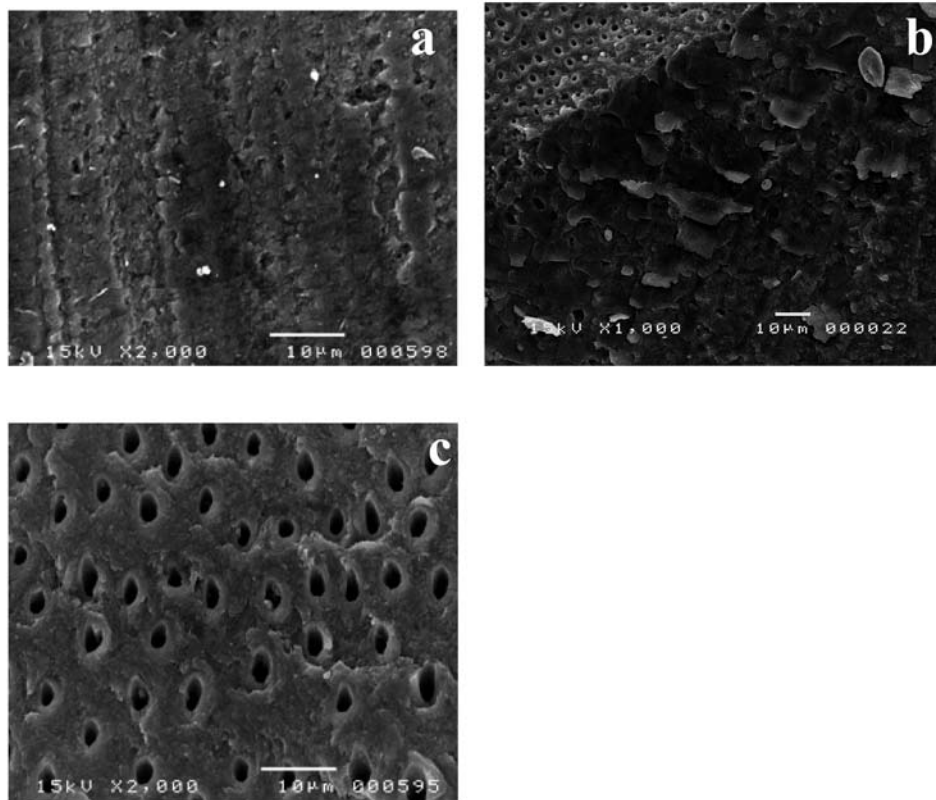


Fig. 2 SEM photographs illustrating typical fractured surfaces after debonding: (a) Adhesive failure at the interface. Note that scratch marks created with 600-grit SiC paper were observed; (b) Mixed failure including adhesive failure and cohesive failure in dentin; (c) Cohesive failure in dentin.

Table 5 Modes of failure pattern after tensile bond testing

	S6054	UniFil Core	Clearfil DC Core Automix
Curing Light XL3000	A(40%), B(60%)	A(80%), B(20%)	B(60%), C(40%)
Hyper Lightel	A(70%), B(30%)	A(80%), B(20%)	B(40%), C(60%)
LEDemetron1	A(60%), B(40%)	A(90%), B(10%)	B(70%), C(30%)

Fracture mode: A—adhesive failure; B—mixed failure including adhesive failure and cohesive failure in dentin; C—cohesive failure in dentin

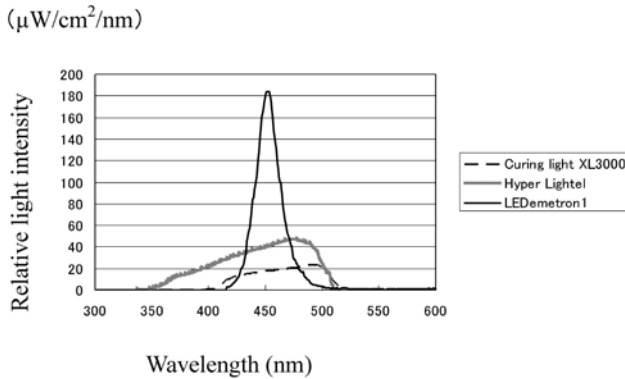


Fig. 3 Wavelength distributions of the light curing units

in dentin (mode C in Fig. 2c) were observed.

Figure 3 shows the wavelength distributions of the three light curing units used in this study. XL3000 emitted a broad output spectrum ranging from approximately 400 to 500 nm. Hyper Lightel had a wider output spectrum than Curing Lightel XL3000. In contrast, LEDemetron1 had a narrow output spectrum.

DISCUSSION

Bovine teeth are choice substrates for screening tests of dentin bonding systems because of their appropriate size and good availability. Further, previous studies have shown few differences in tensile bond strength between human and bovine teeth^{22,23}. Taken together, bovine teeth have proven to be a suitable substitute for human teeth in dentin or enamel bond strength testing²⁴.

Two QTH LCUs, one conventional (Curing Light XL3000) and one high-power (Hyper Lightel), and one high-power LED LCU (LEDemetron1) were used in this study. High-power LCUs help to enhance the speed of polymerization of light-activated adhesives and/or composites because of their higher

light intensity, therefore contributing to reduced chairtime²⁵. Compared to QTH LCUs, a LED LCU presents several advantages such as non-degradation of bulbs, straightforward handling, and longer lifetime^{26,27}. The irradiance spectrum of each LCU is shown in Fig. 3, revealing that Curing Light XL3000 emitted a broad output spectrum covering a wavelength range of 400–500 nm. Comparatively, Hyper Lightel covered a wider light spectrum than Curing Lightel XL3000. On the other hand, LEDemetron1 had the narrowest output spectrum with a LED emission peak at 455 nm, which could activate camphorquinone and initiate a free radical polymerization reaction.

Camphorquinone is commonly used as a photoinitiator for light-cured dental materials, with an effective wavelength range for activation at 410 to 500 nm and a peak intensity at about 470 nm²⁸. At this juncture, it must be mentioned that other photoinitiators with different absorption ranges — such as with an absorption maximum at 410 nm²⁹ — may also be included in the dental materials. However, such chemical compositions are usually not expressly stated by the manufacturers. In the context of the present study, the LED LCU — with the narrowest spectral output among the three LCUs used — might not have activated the photoinitiators with different absorption ranges, if such photoinitiator systems were included in the materials.

When polymerization was performed using the two QTH LCUs, irradiation times for the adhesive/composite core materials were as per the manufacturer's instructions for each material. However, when polymerization was performed using the LED LCU, the irradiation time for each material was as per the LCU manufacturer's recommendation, where photoirradiation for five seconds was sufficient to polymerize a composite with thickness less than 2 mm. In the current study, composite thickness was standardized at 0.15 mm with a vinyl masking tape, which was considered to be sufficient for polymerization by each LCU condition.

In the S6054 system, the experimental adhesive was a chemically cured adhesive; however, the

composite core was light-activated. Chemical activation of adhesives is beneficial in the case of deep cavities without light transmission. However, there is little published information on dentin bonding that involves a chemically-cured adhesive and a light-cured composite core. In the present study, the S6054 system was not affected by the different curing strategies. Therefore, the current results suggested that in the bid to enhance dentin bond strength, a chemically-cured adhesive system could be a viable alternative for composite core materials.

Among the three composite core systems, the UniFil Core system ranked the lowest in dentin bond strength. Failure mode of the debonded specimens was mainly adhesive failure for each curing strategy. Theoretically, debonding of a bonded specimen occurs at the weakest point in the bonding interface³⁰. In other words, there might be some difficulty for this system to sufficiently remove the smear layer to form a hybrid layer with intact dentin. The adhesive, UniFil Core Self-etching Bond, was categorized as a single-step self-etching adhesive system which contained an acidic monomer of 4-MET, ethanol, and water. A self-etching primer adhesive system is easy to use, but technique-sensitive^{31,32}. It has been reported that phase separation took place in one-step self-etching adhesives, deteriorating dentin bonding performance^{33,34}.

Further, in the underlying self-etching adhesive layer, studies have suggested that adverse surface interactions occur between delayed-activated light-cured or chemically cured resin composites and uncured acidic resin monomers. This uncured monomer surface layer is formed during irradiation of the adhesive, due to the effect of atmospheric oxygen on the activation reaction of the adhesive resin^{10,31,35}. Consequently, adverse interactions can reduce the bond strength of adhesive systems to dental tissues³⁶.

In contrast, Clearfil DC Core Automix demonstrated the best dentin bonding performance among the three composite core systems. Cohesive failure in dentin was dominantly observed after debonding. The two-step self-etching primer adhesive system, Clearfil Liner Bond 2V, was composed of a two-bottle primer and adhesive, both of which were dual-activated. Interestingly, the highest bond strength was obtained by light curing with Hyper Lightel. It is noteworthy that LEDemetron1 also had sufficient light intensity, but a narrower wavelength range. It is common for light-cured dental materials to have camphorquinone as the photoinitiator, which is activated in the range of 410 to 500 nm with a peak intensity at about 470 nm²⁸. However, Yamauti *et al.*³⁷ reported that the two-step self-etching primer system, Clearfil SE Bond, had a photoinitiator which could not be activated with a plasma arc light curing

unit because of the latter's narrow wavelength distribution. On a separate note, it has also been reported that some adhesive and/or composite materials contain camphorquinone and other photoinitiators with different absorption ranges (such as absorption maximum at 410 nm)²⁹. Thus, it was not clear whether Clearfil Liner Bond 2V indeed had another photoinitiator which could have influenced the bond strength result.

Until recently, non-vital teeth were treated with a crown, core, and/or dowel, often leading to the sacrifice of sound tooth structure³⁸. A post space is prepared to gain mechanical retention of the core, meaning further removal of sound tooth substance. In the case of a post and core system, a greater degree of stress may be concentrated in the root^{9,39}. Put together, these factors may adversely affect the longevity of a non-adhesively restored tooth. If good retention can be obtained using a dentin bonding system, a post may not be necessary, thus preserving sound tooth structure. Moreover, in clinical situations, direct composite core restorations are the preferred treatment over indirect composite core restorations because they require minimal intervention and cavity preparation.

On one hand, compatibility between the emission spectrum of LCUs and the absorption spectrum of photoinitiators is a highly influential factor in dentin bond strength. On the other hand, it must be recognized that there are regions in sound tooth structure that are inaccessible to light transmission. A previous study reported that bond strength to post space dentin was lower than that to coronal dentin⁴⁰. Therefore, further research should be carried out to evaluate the regional bond strengths of these composite core systems.

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