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Original

Bond strength to bovine dentin of a composite core build-up material combined with four different bonding agents

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Abstract: Clearfil DC Bond (DC) is a new single-step, dual-cure bonding agent. In this study, the shear bond strengths of a core build-up composite to dentin used with four bonding systems [DC, Unifil Core Self-Etching Bond (UC), Clearfil SE Bond (SE) and Cleafil tri-S Bond (TS)] were measured. The bonding ability after 7 days of storage and *in vitro* durability following 20,000 thermocycles were also evaluated. The bond strength of DC did not differ significantly from those of other bonding systems after 24 hours of storage. Another dual-cure bonding system, UC, showed a significant reduction of bond strength after 7 days of storage. On the other hand, the bond strength of TS, a light-cured bonding system with a similar composition to DC, was reduced significantly following 20,000 thermocycles. SE, a two-step light-cure bonding system in the same series as DC, provided superior bond strength under all conditions. Although DC showed a slightly lower bond strength than SE, there was no significant difference between DC and SE under all conditions. Consequently, DC may be a useful and effective bonding system for multiple composite resin restorations. (J. Oral Sci. 50, 329-333, 2008)

Keywords: dual-cure; single-step; self-etching; bond strength; bonding system.

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Introduction

Various bonding systems with improved bonding characteristics that are easy to use have been developed. Recently, self-etching adhesive systems have been widely used for bonding to dentin substrates because of their superior bond strength and reduced technique sensitivity (1-3). These systems simultaneously demineralize and penetrate resin monomer into dentin without the rinsing and drying step (4,5). Currently, single-step self-etching systems, which combine the three steps of etching, priming and bonding in a single application, with no need to re-wet the dentin surface or re-expand the shrunken collagen network, have been proposed as suitable agents for dentin bonding (6).

Clearfil DC Bond (DC; Kuraray Medical Inc., Tokyo, Japan) is a new self-etching, two-bottle/single-step, dual-cure bonding agent. According to the manufacturer's instructions, the clinical indications include core build-ups, direct restorations, and cavity sealing as a pretreatment for indirect restorations. In this study, the shear bond strengths of a core build-up composite to dentin using four bonding systems, including DC, were measured. The bonding ability after 7 days of storage and *in vitro* durability following 20,000 thermocycles were evaluated.

Materials and Methods

Preparation of bonded specimens

A total of 96 bovine mandibular incisors were used as dentin specimens. The facial surface was ground with a rotary cutting instrument to expose the coronal dentin surface, which was then embedded in an aluminum mold

(13 mm in diameter) using self-polymerizing acrylic resin. The exposed dentin was ground until flat using 800-grit silicon carbide (SiC) abrasive paper under running water. A piece of tape with a 3.0-mm-diameter hole was then positioned on the surface of the dentin to define the bonding area. The bonding systems used in this study are shown in Table 1. These comprised two dual-cure adhesives [DC and Unifil Core Self-Etching Bond (UC)] and two light-cured adhesives [Clearfil SE Bond (SE) and Cleafil tri-S Bond (TS)]. Of these, DC, UC and TS are single-step systems and SE is a two-step system. The specimens were randomly divided into four groups of bonding systems with 24 specimens each. Bonding procedures were performed in each group according to the manufacturers' instructions presented in Table 2. Light-irradiation was performed with the same light source (Optilux 501, Kerr Corp., Danbury, CT, USA). The power density of the light source was adjusted to between 400 to 500 mW/cm^2, as measured with a dental radiometer (Optilux Radiometer, Kerr Corp.). After completion of the bonding procedures, a cylindrical plastic mold with an internal diameter of 5.0 mm and height 3.0 mm was placed to surround the bonding area. Dual-cure composite resin core material (Clearfil DC core Automix) was then filled into the mold and light exposure was performed for 40 s.

Bond strength test

Thirty minutes after light-curing, all specimens were stored in water at 37°C. The specimens in each bonding system were randomly divided into three subgroups of eight specimens each. One-third of the specimens were stored in water for 24 h. Another one-third were stored in water for 7 days. The remaining one-third were stored in water for 24 h and subsequently thermocycled between 5°C and 55°C in water baths for 20,000 cycles with a dwell time of 1 min per bath (Thermal Shock Tester TTS-1 LM, Thomas Kagaku Co. Ltd., Tokyo, Japan). Each specimen was then embedded in a steel mold and seated in an ISO/TR 11405 shear testing jig. Shear bond strengths were measured with a mechanical testing machine (Type

Table 1 Materials used in this study

Material	**Trade name (Manufacturer)**		**Composition**	**Lot number**
Bonding system	Clearfil DC Bond (Kuraray Medical Inc., Tokyo Japan)	liquid A	HEMA, bis-GMA, MDP, colloidal silica, dibenzoyl peroxide, dl-camphorquinone, initiators	DCAT1
		liquid B	ethanol, water, initiators, accelerators	DCAT1
	Unifil Core Self-Etching Bond (GC Corp., Tokyo Japan)	liquid A	4-MET, dimethacrylate, silica, ethanol, water, catalyst	501051
		liquid B	ethanol, catalyst	501051
	Clearfil SE Bond (Kuraray Medical Inc., Tokyo Japan)	primer	HEMA, MDP, hydrophilic aliphatic dimethacrylate, dl-camphorquinone, water, accelerators, dyes	00552A
		bond	HEMA, bis-GMA, MDP, hydrophobic aliphatic dimethacrylate, colloidal silica, dl-camphorquinone, initiators, accelerators	00778A
	Clearfil Tri-S Bond (Kuraray Medical Inc., Tokyo Japan)		HEMA, bis-GMA, MDP, colloidal silica, ethanol, water, dl-camphorquinone, initiators, accelerators	00009A
Composite	Clearfil DC Core Automix (Kuraray Medical Inc., Tokyo Japan)	catalyst	bis-GMA, TEGDMA, silanated colloidal silica, dl-camphorquinone, initiators, accelerators	0015AC
		universal	TEGDMA, hydrophobic aromatic dimethacrylate, silanated colloidal silica, dl-camphorquinone, initiators, accelerators	

HEMA: 2-hydroxyethyl methacrylate; bis-GMA: 2,2-bis[4-(2-hydroxy-3-methacryloxypropoxy)phenyl] propane; MDP: 10-methacryloyloxydecyl dihydrogen phosphate; 4-MET: 4-methacryloyloxyethyl trimellitate, TEGDMA: Triethyleneglycol dimethacrylate

5567, Instron Corp., Canton, MA, USA) at a cross-head speed of 1.0 mm/min. The shear bond strength was calculated by dividing the force at which bond failure occurred by the bonding area.

Statistical analysis

For multiple comparisons, homogeneity of variance was assessed by the Levene test. Tukey HSD test was performed for comparison of bonding systems. Mann-Whitney U test was used for comparison of bond strength between the 24-hour storage group and 7-day storage group or between the 24-hour storage group and the 20,000-thermocycle group. Statistical analysis was carried out with SPSS software Version 14.0 for Windows (Chicago, IL, USA) at a significance level of $P < 0.05$.

Results

The data for shear bond strengths (MPa) and statistical analysis in the 24-hour storage group are shown in Table 3. The strength of DC was 12.0 ± 2.6 (mean ± SD) MPa. SE exhibited the greatest bond strength (mean ± SD: 14.0 ± 3.2 MPa), but there were no significant differences between DC, SE and TS.

The results for the 7-day storage group are shown in Table 4. The bond strength of UC (mean ± SD: 5.6 ± 2.7 MPa) was significantly lower than that of the other systems. Comparison of the bond strengths between the 24-hour storage group and 7-day storage group was performed to evaluate the effect of water storage. In the 7-day storage group, the strengths of DC, SE and TS were not affected significantly in comparison with those in the 24-hour storage group. However, the bond strength of UC was reduced significantly in comparison with that at 24 h.

Table 2 Application procedures of bonding systems

Bonding system	Procedure
DC	Mix bond liquid A and B, apply bond mixture, leave for 20 s, air dry, light cure for 20 s
UC	Mix bond liquid A and B, apply bond mixture, leave for 30 s, air dry, light cure for 10 s
SE	Apply primer, leave for 20 s, air dry, apply bond, gently air blow, light cure for 10 s
TS	Apply bond, leave for 20 s, air dry, light cure for 10 s

Table 3 Shear bond strength after 24-hour storage and statistical category

Bonding system	**24-hour storage**	
	Mean ± S.D. (MPa)	**Statistical category**
DC	12.0 ± 2.6	a, b
UC	9.1 ± 3.6	a
SE	14.0 ± 3.2	b
TS	10.7 ± 2.0	a, b

S.D.: standard deviation; statistical category: Identical letters indicate that values are not statistically different ($P > 0.05$) with Tukey HSD comparison.

Table 4 Shear bond strength after 7-day storage and statistical category

Bonding system	**7-day storage**	
	Mean ± S.D. (MPa)	**Statistical category**
DC	11.2 ± 1.6	d
UC	5.6 ± 2.7	c
SE	12.4 ± 2.0	d
TS	12.2 ± 2.3	d

S.D.: standard deviation; statistical category: Identical letters indicate that values are not statistically different ($P > 0.05$) with Tukey HSD comparison.

The results obtained after subjecting the specimens to 20,000 thermocycles are shown in Table 5. The bond strengths of UC and TS were lower than those of DC (mean ± SD: 10.3 ± 3.1 MPa) and SE (mean ± SD: 14.2 ± 3.2 MPa). Although there was a significant difference in bond strengths between SE and TS, no significant difference was detected between those of DC and SE or those of DC and TS. Evaluation of durability was performed by comparing the bond strengths in the 24-hour storage group and the 20,000-thermocycle group. The bond strengths of DC and SE were not changed significantly, whereas those of UC and TS were significantly reduced.

Discussion

The prerequisite for esthetic restorations and for preservation of healthy tooth structure has lead to an improvement of adhesive materials. DC is a newly developed, self-etching, two-bottle/single-step, dual-cure bonding system. The two-bottle modification is to separate the initiation system into two components. Consequently, this system is applicable in deep areas such as post cavities. In this study, the bonding abilities of four bonding systems, including DC, were measured and compared. SE is a two-step, light-cured bonding system, whereas TS is a single-step, light-cured bonding system. These systems were developed by the same manufacturer and have similar compositions. The self-etching adhesives consist of a mixture of self-etching adhesive monomers [e.g., 2-hydroxyethyl methacrylate (HEMA)-phosphate, 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP)], cross-linking monomers [e.g., 2,2-bis [4-(2-hydroxy-3-methacryloxypropoxy) phenyl] propane (bis-GMA)] and additional monofunctional co-monomers (e.g. HEMA). On the other hand, UC is also a self-etching, two-bottle/single-step, dual-cure bonding system produced by another manufacturer, in which 4-methacryloxyethyl trimellitate (4-MET) is utilized as the adhesive monomer.

Clearfil DC Core Automix is a dual-cure, two-component core build-up material supplied in an automix delivery system. The paste can be squeezed out from the dispenser syringe into the cavity. Foxton et al. reported that light exposure of dual-cure adhesive resulted in significantly higher bonding strength to root canal dentin than chemical cure alone (7). Additionally, Aksornmuang et al. reported that the bond strength of SE at the apical region was lower than at the coronal region (8). In this study, we focused on evaluation of bond strength when the adhesives were directly light-cured.

In the 24-hour storage group, the bond strength of DC did not demonstrate any significant difference from those of UC, SE or TS. Storage in water for 7 days did not affect the bond strength of any system except for UC. These observations suggested that the bond strength of UC was easily weakened by water exposure. In the 20,000-thermocycle test, the reduction of the bonding strength of UC and TS was significant. Although there was a significant difference of bond strengths between SE and TS, no significant difference was detected in those of DC and SE or those of DC and TS. Accordingly, it is inferred that the behavior of DC is intermediate between SE and TS.

Single-step systems are formulated to be more acidic and hydrophilic than two-step self-etch adhesives (1,9), and water is essential in single-step self-etching systems to adequately ionize the acidic monomers, dissolve the smear layer, and demineralize the dentin (10). Incorporation of high concentrations of acidic monomers may lead to water sorption, resulting in a decline in the marginal integrity of the adhesives. It has been reported that one-step adhesives are often associated with lower bond strengths than two-step or multi-step bonding agents (11,12). The strength reduction of TS after 20,000 thermocycles in this study was in agreement with these previous reports.

The potential benefit of additional chemical interaction between the functional monomer and residual hydroxyapatite has been reported (13). That study

Table 5 Shear bond strength after 20,000-thermocycle and statistical category

Bonding system	24-hour storage + 20,000-thermocycle	
	Mean ± S.D. (MPa)	Statistical category
DC	10.3 ± 3.1	f, g
UC	4.2 ± 2.9	e
SE	14.2 ± 3.2	g
TS	7.2 ± 3.0	e, f

S.D.: standard deviation; statistical category: Identical letters indicate that values are not statistically different ($P > 0.05$) with Tukey HSD comparison.

demonstrated that 10-MDP interacted strongly with hydroxyapatite, and that its calcium salt was hydrolytically stable. Furthermore it was concluded that the bonding potential of 4-MET was substantially lower than that of 10-MDP (13). The lower strength and durability of UC with 4-MET in this study was also in agreement with that report.

Within the limitations of this study, it can be concluded that the new dual-cure bonding system, Clearfil DC Bond, has greater shear bond strength and better durability than other dual-cure bonding systems. Furthermore, the performance of Clearfil DC Bond is as good as that of the two-step, light-cured bonding system, SE. Consequently, this bonding agent seems to be effective not only for core build-up, but also for direct restoration.

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