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Influence of adhesive polymerization mode on dentin bond strength of direct core foundation systems

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Abstract: This study examined the influence of various adhesive systems on dentin bond strength of direct core foundation resins. Two commercially available direct core foundation resin systems and 2 adhesive polymerization modes were used. Facial bovine dentin surfaces were wet ground on 600-grit SiC paper. Dentin surfaces were treated according to the manufacturers' instructions and were light polymerized (control). Chemical- and light-polymerized adhesive systems were used separately. The resin paste was condensed into a mold and bonded to the dentin surface. Ten specimens per test group were stored in water at 37˚C for 24 hours, and a shear test was conducted at a crosshead speed of 1.0 mm/minute using a universal testing machine. Analysis of variance (ANOVA) and Duncan's multiple comparison test were performed (α = 0.05). Dual polymerization of resin pastes revealed higher bond strength with the combination of light-polymerized adhesive (22.8-24.3 MPa), but significantly lower bond strength with the combination of a chemical-polymerized adhesive (4.2-5.7 MPa). The present data suggests that dentin bond strengths in direct core foundation systems can be influenced by the combination of adhesive and resin paste. (J. Oral Sci. 46, 185-189, 2004)

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Introduction

Adhesion between tooth substrates and restorative materials enables minimally invasive restorative techniques (1-3). The goal of these techniques is to preserve tooth structure as opposed to cavity preparation for mechanical retention. The use of newly developed adhesive systems also enables non-vital teeth to be restored by only replacing tooth structure lost due to endodontic treatment with direct core foundation resins (4-6).

Because of the numerous treatment alternatives available, there is much confusion associated with choosing the most suitable material or combination of materials for a given procedure. Selecting the appropriate adhesive system and core foundation resin for restoring endodontically treated teeth is a determining factor for the success of treatment (7-10). It has been reported that chemical-polymerized resins might be suitable for core foundations (11), but controlled placement into a narrow canal is difficult within the limited working time. The major advantage of light-polymerized materials is their ease of use. As a result of their set-on-command capability, working time is not a clinical limitation. However, for regions distant from the light source, adequate polymerization of the resin paste cannot be achieved (12,13). In order to combine the advantages of chemical- and light-polymerized materials, dual-polymerized core foundation resins have been developed (14).

The combination of an adhesive system and a resin paste recommended by the manufacturer is thought to be

an important factor in obtaining good clinical results. Clinically, different types of adhesive system can be used in combination with a number of core foundation resins. Because there is little information regarding how polymerization strategy affects dentin bond strength in direct core foundation systems (8), research into the influence of polymerization mode on dentin bonding utilizing these materials is required (15,16). The purpose of this study was to examine the influence of different adhesive polymerization modes on dentin bond strength of dual-polymerized core foundation resins. The null hypothesis was that there would be no difference in the ability to obtain proper dentin adhesion irrespective of the adhesive polymerization mode employed.

Materials and Methods

Two commercial resin core foundation systems (Clearfil DC Core and UniFil Core) in combination with the corresponding dual polymerized adhesive systems were used (Table 1). In addition, 2 commercially available adhesive systems were used; the light-polymerized adhesive system Clearfil Mega Bond (known as Clearfil SE Bond outside of Japan, Kuraray Medical, Tokyo, Japan) and the chemical-polymerized adhesive system Clearfil New Bond (Kuraray Medical) were used (Table 1).

A polymerization unit (Optilux 501, Demetron/Kerr, Danbry, CT, USA), with light intensity above 600 mW/cm^2 as measured with a dental radiometer (Model 100; Demetron/Kerr), was used for all specimens.

A total of 100 mandibular incisors from 2-3 year old cattle stored frozen at -20˚C for up to 2 weeks after extraction were used as substitutes for human teeth. After removing the roots with a low-speed diamond saw (Buehler, Lake Bluff, IL, USA), pulps were removed, and the pulp chamber of each tooth was filled with cotton in order to avoid penetration of embedding medium. The labial surfaces of the bovine incisors were ground with wet 240-grit SiC paper to produce a flat dentin surface. Each tooth was then mounted in auto polymerizing acrylic resin (Tray Resin II, Shofu, Kyoto, Japan) to expose the flattened area and was placed in tap water to reduce the temperature rise from the exothermic polymerization of the acrylic resin. The final finish of the dentin surface was accomplished by grinding with wet 600-grit SiC paper. After ultrasonic cleaning in distilled water for 1 minute to remove debris on the dentin surface, the specimens were washed with tap water and dried with oil-free compressed air.

Adhesive tape (NW-20, Nichiban, Tokyo Japan) was used to define the tooth area for bonding and a Teflon (Sanplatec, Osaka, Japan) mold, 2.0 mm high and 4.0 mm in diameter, was used to form and hold the materials to the tooth surface. The dentin surfaces were treated according to each manufacturer's recommendations. For the Clearfil DC Core system, the dentin surfaces were primed (ED Primer II) for 30 seconds and were then air dried with a gentle stream of air. Equal amounts of Clearfil Photo Bond liquids were mixed and applied on the primed dentin surface. After gentle air blowing, the adhesive was irradiated for 10 seconds. The Clearfil DC Core pastes were mixed for 20 seconds, and the mixed resin was inserted into the mold on the dentin surface followed by irradiation for 40 seconds. For the UniFil Core system, one drop of each Self-Etching Bond component was mixed and applied to the dentin surface for 30 seconds. After air blowing for 10 seconds, the adhesive was irradiated for 10 seconds. Equal amounts of the UniFil Core pastes were mixed for 10 seconds, and the mixed resin paste was inserted into the mold onto the dentin surface followed by irradiation for 30 seconds.

For the chemical-polymerized adhesive system Clearfil New Bond, dentin surfaces were etched for 30 seconds with 35% phosphoric acid followed by washing with distilled water for 30 seconds. A thin coat of mixed adhesive was applied to the dentin followed by application of each of the core pastes polymerized by either chemical or light activation. For the light-polymerized adhesive system Clearfil Mega Bond, a self-etching primer was applied for 20 seconds. One coat of adhesive resin was applied and irradiated for 10 seconds followed by each resin core foundation paste, which was polymerized either chemically or by light activation.

The mold and adhesive tape were removed from the specimens 1 hour after resin paste placement, and all specimens were stored in water at 37˚C for 24 hours. Specimens in each group were tested in shear mode using a knife-edge testing apparatus in a universal testing machine (Type 4204, Instron, Canton, MA, USA) at a crosshead speed of 1.0 mm/minute. Shear bond strengths in MPa were calculated from the peak load at failure divided by the bond surface area. After testing, the specimens were examined under an optical microscope (MW-10F, Tokyokinzoku, Tokyo, Japan) at a magnification of ×10 in order to determine the location of bond failure. The test area on the tooth was divided into 8 segments according to the point count method, and the percentage that was free of adhesive or restorative material was estimated. The type of failure was determined based on the predominant percentage of substrate-free material as: adhesive failure, cohesive failure in resin composite, cohesive failure in bond agent, and cohesive failure in enamel.

The results were analyzed by calculating the mean and standard deviation of values for each group. Bond strength

data for each group of specimens was subjected to analysis of variance (ANOVA) in order to determine any significant differences within the data with respect to polymerization method, and Duncan's multiple comparison was performed (α=0.05). Statistical analysis was performed using statistical software (Sigma Stat, Version 2.03, SPSS, Chicago, IL, USA).

Results

The influence of various types of adhesive on dentin bond strength of Clearfil DC Core and UniFil Core are shown in Tables 2 and 3, respectively. With the use of chemical-polymerized adhesive resin, bond strengths decreased significantly when compared with those of controls, in which the dual polymerized resin pastes were irradiated. Dual polymerization of resin pastes resulted in decreasing bond strength for the chemical-polymerized adhesive system, but significantly lower bond strength was observed for the light-polymerized adhesive system. When the dual-polymerized resin pastes were not irradiated, significantly higher bond strengths were obtained when compared with those obtained under the dual-polymerized mode.

The predominant failure mode was adhesive failure between the dentin and adhesive resin when the chemical-polymerized adhesive system was used. When the light-polymerized bonding agent was used, the predominant failure mode was cohesive failure in dentin regardless of polymerization mode of the resin pastes.

Discussion

Large numbers of intact, extracted teeth are required for conducting bond strength tests, but it is difficult to obtain sufficient numbers of extracted human teeth in Japan. It has been reported that the adhesion to the superficial layer of dentin does not differ significantly between human and bovine dentin, and that the dentin bond strength decreases with dentin depth due to the lower density of dentinal tubules (17). Since differences in tubule diameter and number of lateral branches may have some effect on dentin bond strength (18,19), bovine superficial dentin was used as a substitute for human dentin in this study, as has been reported in previous studies (20,21). Bond strength values measured depend on the bonding system used, site on the tooth, and type of tooth structure (22-26). Care should be taken when drawing conclusions from bond strength data because there are numerous factors that affect bond values (27).

It has been reported that there is an inverse relationship

Table 1 Materials tested in this study

Core foundation systems used

Core foundation system (Manufacturer)	Adhesive system (Lot No.)	Resin paste (Lot No.)
Clearfil DC Core (Kuraray Medical, Tokyo, Japan)	ED Primer II (A: 00134A, B: 00020A) HEMA, MDP, 5-NMSA N, N-diethanol p-toluidin, water	DC Core (C: 0176, U: 0169) Bis-GMA, MDP, dimethacrylate, filler photo/chemical initiator
	Clearfil Photo Bond (C: 342, U: 444) Bis-GMA, HEMA, MDP N, N-diethanol p-toluidin photo/chemical initiator	
UniFil Core (GC Corp, Tokyo, Japan)	Self-Etching Bond (A: 0112251, B: 0012011) 4-MET, ethanol, water methacrylate monomer photo/chemical initiator	UniFil Core (0201291) UDMA, dimethacrylate fluoroaluminosilicate glass photo/chemical initiator

Additional adhesive systems used

Adhesive system (Manufacturer)	Etching/primer (Lot No.)	Bond agent (Lot No.)
Clearfil New Bond (Kuraray Medical, Tokyo, Japan)	Etchant (811) 40% phosphoric acid	Bond (U: 031, C: 921) Bis-GMA, HEMA, MDP N, N-diethanol p-toluidin chemical initiator
Clearfil Mega Bond (Kuraray Medical, Tokyo, Japan)	Primer (00135A) Water, ethanol, MDP, HEMA, N, N-diethanol p-toluidin	Bond (00088A) MDP, bis-GMA, HEMA, N, N-diethanol p-toluidin, micro filler, camphorquinone

HEMA: 2-hydroxyethyl methacrylate, MDP: 10-methacryloxydecyl dihydrogen phosphate, 5-NMSA: N-methacryloyl 5-aminosalicylic acid, Bis-GMA: 2, 2-bis[4-(2-hydroxy-3-methacryloyloxypropoxy)]phenyl propane, 4-MET: 4-methacryloyloxyethyl trimellitate

Table 2 Influence of adhesive system on dentin bond strength of Clearfil DC Core and results of statistical analysis

Adhesive system	Polymerization mode Adhesive	Resin paste	Mean bond strength MPa (SD)	Duncan group (α = 0.05)	Number of specimens in failure mode
Control	DP	DP	14.6 (2.1)	a	5 / 5 / 0 / 0
Clearfil New Bond	CP	CP	7.5 (1.2)	b	0 / 0 / 0 / 10
	CP	DP	5.7 (1.2)	c	0 / 0 / 0 / 10
Clearfil Mega Bond	LP	CP	16.0 (1.6)	a	2 / 3 / 5 / 0
	LP	DP	24.3 (1.9)	d	2 / 2 / 6 / 0

CP: Chemical polymerization, LP: Light polymerization, DP: Dual polymerization, n = 10

Failure mode: Cohesive failure in resin / Cohesive failure in adhesive / Cohesive failure in dentin / Adhesive failure

Table 3 Influence of adhesive system on dentin bond strength of UniFil Core and results of statistical analysis

Adhesive system	Polymerization mode Adhesive	Resin paste	Mean bond strength MPa (SD)	Duncan group (α = 0.05)	Number of specimens in failure mode
Control	DP	DP	15.6 (2.2)	a	3 / 3 / 4 / 0
Clearfil New Bond	CP	CP	5.3 (1.2)	b	0 / 0 / 0 / 10
	CP	DP	4.2 (1.8)	c	0 / 0 / 0 / 10
Clearfil Mega Bond	LP	CP	15.3 (1.5)	a	2 / 2 / 6 / 0
	LP	DP	22.8 (2.0)	d	1 / 2 / 7 / 0

CP: Chemical polymerization, LP: Light polymerization, DP: Dual polymerization, n = 10

Failure mode: Cohesive failure in resin / Cohesive failure in adhesive / Cohesive failure in dentin / Adhesive failure

with regard to the use of different polymerization modes of adhesive system with an chenmical-polymerized core foundation resin paste (8). Also, some of the two-step total etching adhesives were found to be incompatible with chemical-polymerized resin composites (28). The decrease in dentin bond strength of chemical-polymerized resin composite is inversely proportional to the acidity of uncured adhesive remaining on the dentin surface (29). Unexpected debonding of chemical-polymerized core foundation resins has been also reported (8). These reported results were attributed to the acid-base reaction between acidic monomers in the oxygen inhibited layer (30,31) and tertiary amines in the chemical-polymerized resins, as this is responsible for the benzoyl peroxide polymerization process. From the results of this study, lower bond strengths occur when different combinations of polymerization mode were employed, and this is thought to be caused by adverse interactions between the nucleophilic tertiary amine and acidic functional monomers (15,16). Though the highest bond strengths were obtained through the use of the light-polymerized adhesive system, proper light irradiation of the adhesive resin is required for optimal bond strength. If clinical situations are considered in which light from the polymerization unit is attenuated, the use of light-polymerized adhesive systems as the resin core foundation has some limitations.

For dual-polymerized resin materials, a photosensitizer such as camphorquinone (CQ) and binary peroxide-amine catalytic components were commonly employed for chemical polymerization (32). CQ requires a co-initiator for effective polymerization to occur, and a tertiary amine photoreductant is used to interact with CQ in its activated triplet state to form an intermediate excited complex and reactive radicals for polymerization. Resin polymerization is affected by acidic moieties, as tertiary amines in the adhesive resins are neutralized by the acidic functional monomers and lose their activity as reducing agents in redox reactions (33,34), thus resulting in poor polymerization. To overcome this type of incompatibility, ternary redox catalysts, such as sodium salts of aromatic sulphinic acid, have been introduced (35). Although these alternative reducing agents enhance the polymerization of resin monomers in dual-polymerized resin systems by scavenging oxygen from the resin, combinations of polymerization modes between the adhesive and resin pastes still affect bond strength.

Within the limitations of this study, the authors must reject the null hypothesis that there is no difference between the different modes of adhesive systems with regard to their compatibility with dual-polymerized core foundation resins. The use of additional chemical analysis techniques should further improve understanding of adverse chemical interactions between adhesive systems and dual-polymerized resins (36-38). Further research is needed in order to elucidate suitable combinations of adhesive systems and direct core foundation resin pastes. Although, polymerization mode in dual-polymerized core foundation systems affects bonding ability, care should be taken not to infer clinical success from bond strength values alone.

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