

Influence of Power Density on Polymerization Behavior and Bond Strengths of Dual-cured Resin Direct Core Foundation Systems

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Clinical Relevance

The dentin bond strength and polymerization behavior of the dual-cured core foundation resins tested in this study were affected by the power densities of the curing unit. When using dual-cured core foundation systems, practitioners should consider power densities for getting optimal bond strength.

SUMMARY

This study examined the influence of power density on dentin bond strength and polymerization behavior of dual-cured direct core foundation resin systems.

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Two commercially available dual-cured direct core foundation resin systems, Clearfil DC Core Automix with Clearfil DC Bond and UniFil Core with Self-Etching Bond, were studied. Bovine mandibular incisors were mounted in autopolymerizing resin and the facial dentin surfaces were ground wet on 600-grit SiC paper. Dentin surfaces were treated according to manufacturer's recommendations. The resin pastes were condensed into the mold and cured with the power densities of 0 (no irradiation), 100, 200, 400 and 600 mW/cm². Ten specimens per group were stored in 37°C water for 24 hours, then shear tested at a crosshead speed of 1.0 mm/minute in a universal testing machine. An ultrasonic measurement device was used to measure the ultrasonic velocities through the core foundation resins. The power densities selected were 0 (no irradiation), 200, and 600 mW/cm², and ultrasonic velocity was calculated. ANOVA and Tukey HSD tests were performed at a level of 0.05.

The highest bond strengths were obtained when the resin pastes were cured with the highest power density for both core foundation systems (16.8 ± 1.9 MPa for Clearfil DC Core Automix, 15.6 ± 2.9 MPa for UniFil Core). When

polymerized with the power densities under 200 mW/cm², significantly lower bond strengths were observed compared to those obtained with the power density of 600 mW/cm². As the core foundation resins hardened, the sonic velocities increased and this tendency differed among the power density of the curing unit. When the sonic velocities at three minutes after the start of measurements were compared, there were no significant differences among different irradiation modes for UniFil Core, while a significant decrease in sonic velocity was obtained when the resin paste was chemically polymerized compared with dual-polymerization for Clearfil DC Core Automix.

The data suggests that the dentin bond strengths and polymerization behavior of the dual-cured, direct core foundation systems are still affected by the power density of the curing unit. With a careful choice of the core foundation systems and power density of the curing unit, the benefit of using resin composites to endodontically-treated teeth might be acceptable.

INTRODUCTION

Improvement of physical properties of resin composite and bonding ability of adhesive systems enable non-vital teeth to be restored by replacing only the tooth structure lost in endodontic treatment with direct core foundation resins.¹⁻⁵ Selecting the appropriate adhesive system in combination with a resin core composite for this purpose is important to the success of the treatment,⁶⁻⁷ since there is a high correlation between the dentin bond strength and mechanical properties of polymerized resin.⁸ One of the critical requirements for dentin bonding is that the restorative systems must achieve complete polymerization after placement, so that the bond strength will be optimized in clinical situations. It has been reported that the shear bond strengths of resin composites to flat dentin were significantly influenced by the mechanical properties of resin composites.⁹

In some cases, a dual-cured material might be indicated, since polymerization will occur by chemical as well as light activation. The rationale was to have a material with extended working time and be capable of reaching a high conversion ratio. However, it has been reported that the dual-cured resins have poorer mechanical properties when the polymerization reaction is limited to chemical-cure mode alone.¹⁰⁻¹² Thus, light irradiation seems to be important for dual-cured materials to achieve good mechanical properties and adhesion to tooth structure. It has been indicated that the regional bond strength of a light-cured resin composite to root canal dentin would be affected by the power density, which decreases due to the depth of the

post cavity, because polymerization reaction of light-cured material is dependent upon the energy of irradiated light.¹³ The transmission of light through the resin composite, which is controlled by absorption and scattering by filler particle, plays important roles in achieving the high conversion rate of monomers. Since there is little information about how the power density of a curing unit affects the dentin bond strength of dual-cured direct core foundation systems, research into the influence of power density on the dentin bonding ability and polymerization characteristics of dual-cured resin core foundation systems is needed.

Measurement of the polymerization process of resin composites is challenging, because monomer conversion develops quickly after light irradiation. Ultrasonic imaging is a non-invasive technique that offers considerable potential for diagnosis, as well as being a valuable tool for research. Ultrasonic devices are used to detect demineralization of tooth substrates¹⁴⁻¹⁷ and to measure the dentin thickness between a tooth surface and the pulp chamber.¹⁸⁻²¹ Because the speed of sonic is sensitive to the viscoelastic properties of materials,²² ultrasonic devices can be used to monitor the polymerization behavior of core foundation resins.

This study examined the influence of the power density of a curing unit on the bond strengths of dual-cured direct core foundation systems to bovine dentin. The null hypothesis was that the dentin bond strengths and polymerization behavior of dual-cured resin core foundation systems were not affected by the power density of the curing unit.

METHODS AND MATERIALS

Core Foundation System

Two dual-cured resin core foundation systems, Clearfil DC Core Automix (DC, Kuraray Medical Inc, Tokyo, Japan) and UniFil Core (UC, GC Corp, Tokyo, Japan), in combination with the manufacturer's corresponding adhesives, were used (Table 1). A visible-light curing unit (Optilux 400; Demetron/Kerr, Danbury, CT, USA) was connected to a variable-voltage transformer (Type S-130-10; Yamabishi Electric Co, Tokyo, Japan) in order to adjust the power density 100, 200, 400 or 600 mW/cm²; these values were determined using a dental curing radiometer (Model 100; Demetron/Kerr). The curing unit was put on the jig so that the distance between the light tip end and the specimen surface was maintained as unchanged during the experiments.

Dentin Bond Strength

A total of 100 mandibular incisors from two-to-three year old cattle were used as a substitute for human teeth.²³⁻²⁵ After removing the roots with a low-speed diamond saw (Isomet 1000; Buehler Ltd, Lake Bluff, IL, USA), pulps were removed and the pulp chamber of each tooth was filled with cotton to avoid penetration

Core Foundation System (Manufacturer)	Adhesive System (Lot #)	Resin Paste (Lot #)
Clearfil DC Core Automix (Kuraray Medical Inc, Tokyo, Japan)	Clearfil DC Bond (A: 00134A, B: 00020A) HEMA, Bis-GMA, MDP, filler, CQ	Clearfil DC Core Automix (000320A) Bis-GMA, TEGDMA dimethacrylate, filler photo/chemical initiator
UniFil Core (GC Corp, Tokyo, Japan)	Self-Etching Bond (A: 0710241, B: 0711011) 4-MET, ethanol, water methacrylate monomer photo/chemical initiator	UniFil Core (0710241) UDMA, dimethacrylate fluoroaluminosilicate glass photo/chemical initiator

Abbreviations; HEMA: 2-hydroxyethyl methacrylate, Bis-GMA: 2, 2bis[4-(2-hydroxy-3-methacryloyloxypropoxy)]phenyl, MDP: 10-methacryloxydecyl di-hydrogen phosphate, 4-MET: 4-methacryloyloxyethyl trimellitate, CQ; di-Camphorquinone

er's recommendations. For DC, equal amounts of Clearfil DC Bond (Kuraray Medical Inc) A and B liquids were mixed for five seconds and applied on the dentin for 20 seconds. After blowing strongly with air for five seconds, the adhesive was

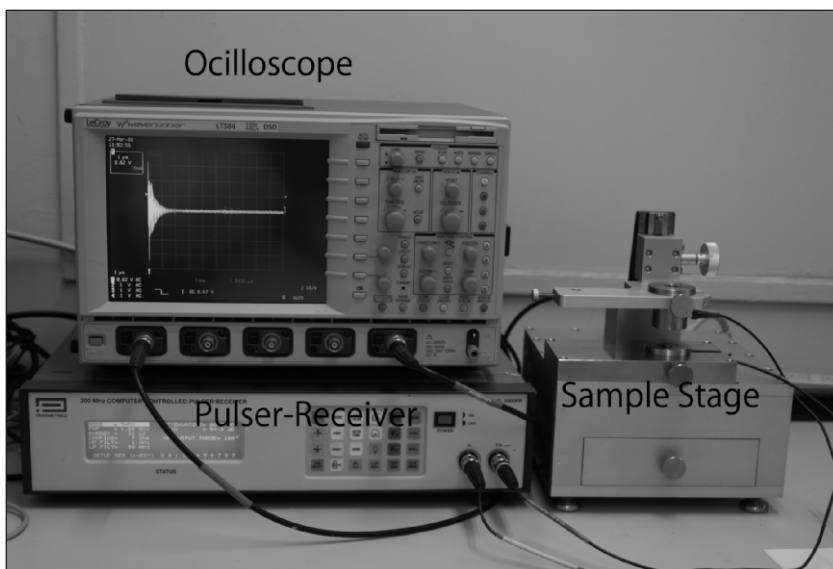


Figure 1. Ultrasonic measurement system used in the current study.

of the embedding medium. The labial surfaces of the bovine incisors were ground with wet 240-grit SiC paper to produce a flat dentin surface (Ecomet 4; Buehler Ltd). Each tooth was then mounted in self-curing acrylic resin (Trey Resin II; Shofu Inc, Kyoto, Japan) to expose the flattened area and placed in tap water to reduce the temperature rise from the exothermic polymerization reaction of the acrylic resin. The final finish of the dentin surface was accomplished by grinding on wet 600-grit SiC paper. After ultrasonic cleaning (Q140W/T; L & R Manufacturing, Kearny, NJ, USA) in distilled water for one minute to remove debris, the surfaces were washed with tap water and dried with oil-free compressed air.

Adhesive tape (NW-20; Nichiban Co, Tokyo Japan) was used to isolate the area of the tooth for bonding and a Teflon (Sanplatec Corp, 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 manufactur-

light polymerized for 20 seconds with the power density of 600 mW/cm². The auto-mixed paste was directly inserted into the mold on the dentin surface followed by light polymerization for 40 seconds with power densities of 0 (no irradiation), 100, 200, 400 or 600 mW/cm². For UC, one drop of each Self-Etching Bond (GC Corp) component was mixed for five seconds and applied on the dentin surface for 30 seconds. After mild air blowing for five seconds, the adhesive was light polymerized for 10 seconds with the power density of 600 mW/cm². Equal amounts of resin pastes were mixed for 10 seconds, and the mixed resin paste was inserted into the mold on the dentin surface followed by light irradiation for 30 seconds with power densities of 0 (no irradiation), 100, 200, 400 or 600 mW/cm².

The mold and adhesive tape were removed from the specimen 10 minutes after light polymerization, then all the specimens were stored in 37°C water for 24 hours. The specimens in each group were tested in shear mode using a knife-edge testing apparatus in a screw-driven universal testing machine (Type 4204; Instron Corp, 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 (SZH-131, Olympus Ltd, Tokyo, Japan) at a magnification of 10x to determine the location of the bond failure. The bonded area on the tooth was divided into eight equal segments and the percentage that was free of adhesive or restorative material was estimated for each segment.²⁴ The types of failures were determined based on the predominant percentage of substrate-free material as: adhesive failure, cohesive failure in resin composite and cohesive failure in dentin.

	Power Density (mW/cm ²)				
	0	100	200	400	600
DC	9.4 (3.8) ^a	9.3 (3.1) ^a	10.3 (3.3) ^a	15.2 (2.1) ^b	16.8 (1.9) ^b
Failure mode	0/0/10	0/0/10	0/0/10	2/3/5	3/3/4
UC	10.0 (2.4) ^c	10.4 (2.8) ^c	11.3 (2.0) ^{c,d}	14.1 (1.7) ^{d,e}	15.6 (2.9) ^e
Failure mode	0/0/10	0/0/10	1/0/9	2/1/7	2/2/6

*N=10, Values in parenthesis indicate standard deviations.
Values with the same letter in each core foundation system are not significantly different ($p>0.05$).
Failure mode: Cohesive failure in resin/Cohesive failure in dentin/Adhesive failure.*

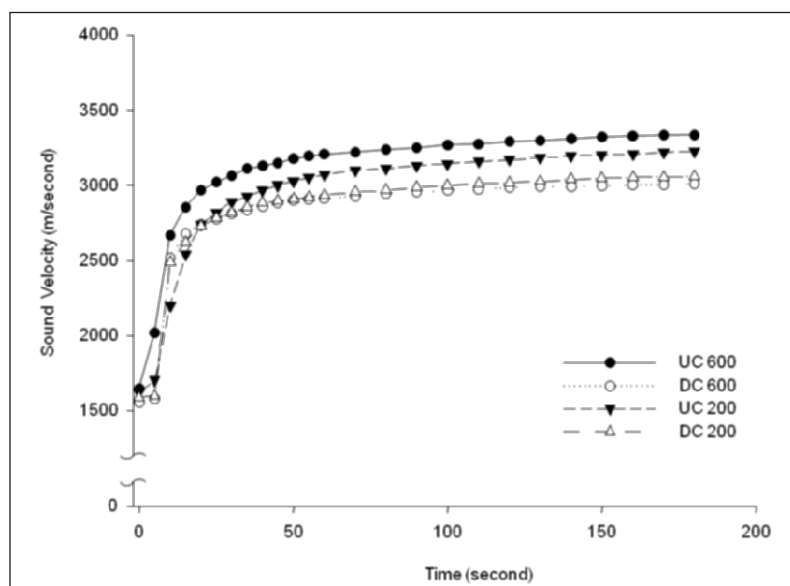


Figure 2. The influence of power densities on changes in ultrasound velocities in core foundation resins as a function of time.

Ultrasonic Velocity

The ultrasonic equipment employed in this study comprised a pulser-receiver (5900PR; Panametrics, Waltham, MA, USA), transducers (V155 and V156; Panametrics) and an oscilloscope (Waverunner LT584, LeCroy, Tokyo, Japan) as shown in Figure 1.²⁶ The transducer equipment was calibrated each time it was used by employing a standard calibration procedure on calibration blocks. The transducer was oriented perpendicular to the contact surface of each specimen.

Ultrasonic velocities were determined by measuring the time taken for an ultrasonic pulse to transverse the specimen. Ultrasonic waves were propagated from the transducer to the specimen, and time taken for the pulse to travel from transducer through the specimen to the receiver was determined from the waveform. The time delay and measured path length (specimen thickness) were then used to calculate the ultrasonic velocity in the specimen.

Core foundation resin composites were mixed according to the manufacturer's instruction and inserted into

the transparent mold (2 mm in thickness). Then the specimen was put on the sample stage and light irradiated (40 seconds for DC, 30 seconds for UC) with power densities of 0 (no irradiation), 200 and 600 mW/cm². The transit time through the specimen disk was divided by the specimen thickness, in order to account for the sonic velocity within the material. The measurements were repeated three times for each irradiation condition.

Statistical Analysis

For purposes of analysis, each resin core system was treated as an independent experiment to compare the results from the different power densities. The bond strength data for each resin system was subjected to a one-way ANOVA on the five groups from each resin core system. Multiple comparisons were then conducted using Tukey's HSD test at a level of 0.05. All statistical analyses were performed using a commercial statistical software package (Sigma Stat; Version 3.1, SPSS Inc, Chicago, IL, USA).

RESULTS

The results of influence of power density of curing unit on the shear bond strengths are shown in Table 2. For both core foundation systems, the highest bond strengths were obtained when the highest power density was employed, and statistically significant differences were found compared to values obtained with the lower power densities (below 200mW/cm²).

The results of failure modes recorded for each group are listed in Table 2. For both core foundation systems, the predominant failure mode was adhesive failure between dentin and adhesive resin, if the resin pastes were light irradiated with lower power densities. When the resin pastes were light irradiated with higher power density, failure mode changed to cohesive failure.

The changes in the ultrasonic velocities within the core foundation resins as a function of time are shown in Figures 2 and 3. In the earliest stages of the setting process, most of the ultrasonic energy was absorbed by

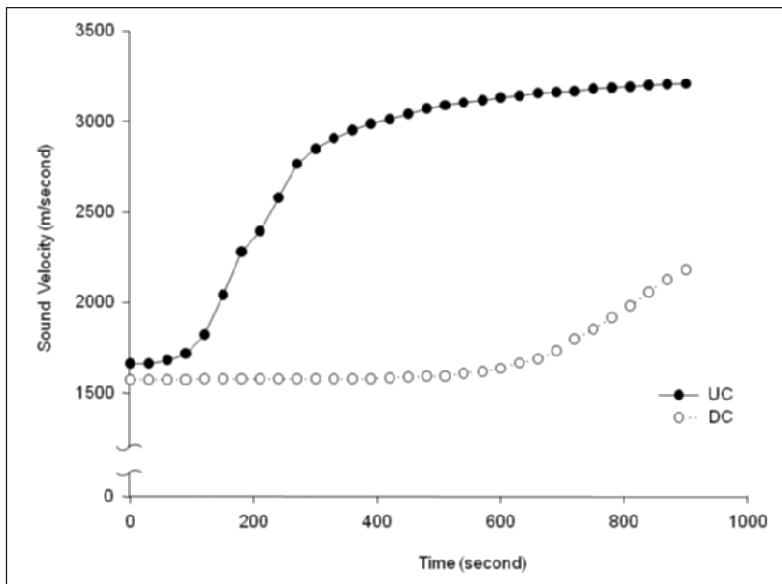


Figure 3. The influence of chemical polymerization (without irradiation) on changes in ultrasound velocities in core foundation resins as a function of time.

Table 3: Influence of Power Density of the Curing Unit on Longitudinally Sonic Velocities (in m/second) of Direct Resin Core Foundation Systems

	Power Density (mW/cm ²)		
	0	200**	600**
DC	2182 (41) ^a	3059 (31) ^b	3008 (49) ^b
UC	3210 (53) ^c	3227 (84) ^c	3338 (50) ^c

*N=3, Values in parenthesis indicate standard deviations.
 *: Sonic velocities at 180 seconds, **: Sonic velocities at 180 minutes.
 Values with the same letter in each core foundation system are not significantly different (p>0.05).*

the core foundation resins, and the echoes that passed through the resin composites were too weak to detect when the resin composites were chemically cured without light irradiation. As the core foundation resins hardened, the sonic velocities increased until they reached a plateau for UC. For DC, the sonic velocities gradually increased 10 minutes after the start of measurement. Although the sonic echoes could be detected from the beginning of the measurements, the rates of sonic velocity increase were relatively slow. When the resin pastes were light irradiated, a rapid increase in sonic velocities was investigated for all specimens regardless of power density employed (200 or 600 mW/cm²). After light irradiation, the sonic velocities reached a certain value and thereafter increased only slightly.

When the sonic velocities at three minutes after the start of measurements were compared, there were no significant differences among the different irradiation modes for UC, while a significant decrease in sonic velocity was obtained when the resin paste was chemically polymerized compared with dual-polymerization for DC (Table 3).

DISCUSSION

It is known that the power density reaching the material is drastically reduced when light is transmitted through a material. The power density decreased exponentially as a function of the material thickness.²⁷ So, dual-cured core foundation resins have been recommended to compensate for the attenuation of the curing light affected by the depth of the post cavity.²⁸ Based on the results of the current study, the null hypothesis that the bonding ability of dual-cured core foundation resins was not affected by power density of the curing unit was not confirmed. When the resin paste was dual-cured with higher power densities, higher values of dentin bond strengths were found. On the other hand, lower bond strengths obtained when the resin pastes were polymerized with the power densities under 200 mW/cm², including no irradiation, may suggest inferior physical properties due to poorly polymerized resin.⁹ This indicated that, with the same reason, the mechanical properties of the material at the coronal portion were better than those at the apical portion. Therefore, the properties of dual-cured core foundation resins may be different at different regions of the post cavity because of the reduction of light energy in the deeper regions of the post cavity, and this may also affect regional bond strengths.

A study²⁹ was performed to evaluate the regional ultimate tensile strength and Knoop hardness of various dual-cured core foundation resins and their regional bond strengths to root canal dentin using a photo-cured self-etching priming adhesive system, and their conclusions were that the ultimate tensile strength and microhardness of the core foundation resins varied depending on the type of material. The mechanical properties of the resins at the coronal region were found to be superior to those at the apical region of the post space; however, differences in the mechanical properties of the resin core foundation resins did not affect their adhesion to root canal dentin. From the results of polymerization behavior by means of ultrasonic measurement, the sonic velocity increased gradually when the resin pastes were chemically polymerized, and neither significant nor rapid increases were observed for DC. On the other hand, UC revealed rapid increases in the ultrasonic velocities after 120 seconds from the start of measurement and reached to plateau.

In the current study, non-destructive ultrasonic testing was applied to monitor the polymerization behaviors of core foundation resins, based on the relationship between the ultrasonic velocities and the elastic constants described by a micromechanical model.³⁰

Higher ultrasonic velocities indicate higher polymerization reaction of resins. Though the time required to reach the plateau was much longer than light irradiated specimens, there were no significant differences in sonic velocities recorded for UC. When the resin pastes were light irradiated with the power densities of 200 or 600 mW/cm², changes in ultrasonic velocities were similar for both core foundation resins. It has been indicated that lower bond strengths are obtained with the lower degree of conversion and resultant lower physical properties of dual-cured resins.³¹ This generalization fits to DC, but not to UC, which exhibited lower bond strength without inferior polymerization.

The adhesive systems tested are polymerized via a dual polymerized reaction and a photosensitizer, such as camphorquinone (CQ), is employed in conjunction with the binary peroxide-amine catalytic components commonly employed in chemical polymerized resins.³² CQ requires a coinitiator for an effective polymerization process to occur, and a tertiary amine photoreductant is employed. The tertiary amine interacts with an activated triplet state of CQ to form an intermediate excited complex followed by the production of reactive radicals for polymerization.

The adhesive systems used in this study are categorized as self-etching systems. These self-etching adhesives are aqueous mixtures of acidic functional monomers and other constituents. Since acidic components of these adhesives are primarily dicarboxylic acids, superficial dentin beneath the smear layer is simultaneously demineralized and infiltrated with resin monomers.³³ After infiltration into the etched/primed dentin, resin monomers must sufficiently polymerize to create a durable bond. However, self-etching adhesives contain acidic monomer and, consequently, a high concentration of uncured acidic monomers would be present on the superficial layer of cured adhesives.

It is possible that the polymerization ability of the core-foundation resin is affected by the acidic moieties, since tertiary amines in the resin paste might be neutralized by the acidic functional monomers in the adhesive. The adhesive functional monomers affect the polymerization of BPO/amine or even CQ/amine catalysts, resulting in poor polymerization leading to poor interaction within the resin paste.³⁴ To improve polymerization in the presence of acidic monomers, accelerators, such as aromatic sulfinic acid sodium salts, were incorporated, together with initiator systems.³⁵ Incompatibility between the adhesives and the core-foundation resin does not occur under higher power density conditions. However, improper polymerization occurs when the dual-cured resin paste polymerizes primarily within a chemical reaction, which probably

leads to adverse interaction between the nucleophilic tertiary amine and acidic functional monomers.³⁶

Although the most reliable conclusions about the performance of bonding systems in the oral environment must be derived from long-term clinical trials, *in vitro* studies provide initial information. The use of additional chemical analysis techniques should further improve understanding of adverse chemical interactions between self-etch adhesives and dual-polymerized resins. Further research is needed in order to elucidate suitable combinations of adhesives and direct core foundation resin pastes. Although polymerization mode in dual-polymerized core foundation systems affect bonding ability, care should be taken not to infer clinical success from bond strength values alone.

CONCLUSIONS

For the dual-cured resin direct core foundation systems studied, dual polymerization with higher power densities resulted in higher bond strengths. If the core resin was polymerized with the power densities under 200 mW/cm², significant reductions in bond strengths were obtained. The data suggests that the dentin bond strengths and polymerization behavior of the dual-cured, direct core foundation systems are still affected by the power density of the curing unit.

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