Mechanical Properties of Dual-Cured Resin Luting Agents for Ceramic Restoration

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Purpose: **The aim of the present study was to evaluate mechanical properties, including surface hardness, flexural strength, and flexural modulus, of two dual-cured resin luting agents [Clearfil Esthetic Cement (CEC) and Variolink II (VLII)] irradiated through four thicknesses of leucite ceramics (0, 1, 2, and 3 mm) and to evaluate their shear bond strength to zirconia ceramic (Cercon) using each ceramic primer.**

Materials and Methods: **Knoop hardness was measured on a thin layer of resin luting agent on the ceramic surface. Three-point bending tests were performed after 24 hours of storage at 37°C. Two differently shaped zirconia ceramic specimens with or without sandblasting with alumina were treated with each primer. The specimens were then cemented together with each resin luting agent. Half of the specimens were stored in water at 37°C for 24 hours and the other half were thermocycled 5000 times.**

Results: **VLII revealed statistically higher Knoop hardness and flexural modulus than CEC for each thickness of ceramic. No significant differences in flexural strength were observed between VLII and CEC for each ceramic spacer. Reduction of the mechanical properties with increase of ceramic thickness varied for each property; however, these properties were similar in the two materials. Blasting with alumina was significantly effective for increasing shear bond strength of both resin luting agents before and after thermal cycling. The use of Clearfil Ceramic Primer showed the highest shear bond strength and maintained bond durability after 5000 thermocycles.**

Conclusion: **Mechanical properties of CEC dual-cured resin luting agent appear adequate for ceramic restorations.**

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INDEX WORDS: dual-cured resin luting agent, zirconia ceramic restoration, surface hardness, flexural strength, bond strength

IT IS well known that patient demand for esthetic and metal-free restorations has increased, and that excellent resin bonding systems (combination of silane coupling agent and resin luting agent) have been developed.[1-5] Regarding feldspathic[6] or silica-based glass ceramics[7,8] and CAD/CAM ceramics,[9-11] hydrofluoric acid etching followed by application of a ceramic primer containing a silane coupling agent is a common and clinically successful procedure. In recent years, new, high-strength ceramics, such as glass-infiltrated[12-14] and CAD/CAM-fabricated densely sintered high-purity alumina[15,16] ceramics and zirconia ceramics, have become more common in restorative dentistry. Dental applications of zirconia materials involve all-ceramic cores and post systems[17,18] and coping for complete coverage of all-ceramic crowns and fixed partial dentures.[19-21] Neither etching with hydrofluoric acid nor a silane coupling agent for silica-based ceramics or glass can reliably improve bond strength between zirconia ceramics with no silica content and resin cements, because of the high resistance of acids.[22] Therefore, other bonding techniques such as tribochemical silica-coating using the Rocatec system[23-27] and special small hand-held fire lighters containing a mixture of butane gas and a silane called PyrosilPen[28] are required to strongly lute zirconia ceramics using a resin bonding system. An experimental primer mixture of phosphoric acid ester monomer and zirconate coupling agent significantly improved the bond strength between

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zirconia ceramic stabilized by yttrium oxide and exists as yttria-tetragonal zirconia polycrystals (Y-TZP) at room temperature and dual-cured resin cement.[29]

Apart from bonding systems for ceramics, the clinical success of ceramic restorations is heavily dependent on the cementation procedure. Resin luting agents should be easy to handle, lack complicated pretreatment steps, and have good mechanical properties, favorable esthetics, and strong adhesion to both tooth structure and ceramics. Dual-cured luting agents are widely used for cementing ceramic restorations in clinics, because they provide these desirable properties. Brands of dual-cured resin luting agents vary in mechanical characteristics such as (1) microleakage of ceramic inlays influenced by their viscosities,[30] (2) tensile strength to copy-milled ceramics influenced by light source direction,[31] and (3) surface hardness cured through machinable ceramics.[32] Adequate polymerization is a crucial factor in obtaining the optimal physical properties of a resin luting agent and a clinically satisfying initial management of the restoration, such as finishing and occlusal adjustment. Inadequate polymerization diminishes the physical properties, affecting mechanical characteristics such as hardness and flexural strength. To ensure adequate polymerization of a resin cement layer that is not readily accessible to the curing light due to the thickness of the ceramic restoration,[32] it is important for dual-cured resin luting agents to be capable of achieving a sufficient degree of hardening with light-curing. Limited information is available regarding the mechanical characteristics of dual-cured resin bonding systems with one-bottle ceramic primer containing silane and phosphoric acid ester monomer for most types of ceramics, including zirconia, and not only for silica-based ceramics. Therefore, we evaluated flexural strength and surface hardness of two dual-cured luting agents polymerized through different thicknesses of machinable ceramics, which simulates the clinical situation, and evaluated their shear bond strength to commercially available zirconia ceramic using each bonding system.

Materials and Methods

Dual-cured Resin Luting Agents with Ceramic Bonding System

Two dual-cured resin luting agents [Clearfil Esthetic Cement (CEC, Kuraray Medical Inc., Kurashiki, Japan) and Variolink II HV (VLII, Ivoclar/Vivadent, Schaan, Liechtenstein)] were prepared. Two one-bottle ceramic primers [Clearfil Ceramic Primer (CCP, SCP-100) and Monobond S (MBS), respectively] were components of each bonding system. Descriptions of these materials are summarized in Table 1.

Preparation of Specimens for Knoop Hardness and Test Procedures

Three thicknesses of machinable ceramic plates (10 × 8 mm squares with 1.05, 2.05, and 3.05 mm thicknesses) were prepared from CAD/CAM blocks (GN-I, shade A3, GC Corp., Tokyo, Japan) using a low-speed

Table 1. Resin Bonding Systems (Combination of Ceramic Primer and Resin Luting Agent)

Product	*Abbreviation*	*Component*
Resin Luting Agent		
Clearfil Esthetic Cement (universal)	CEC	*Monomer:* Bis-GMA, dimethacrylate monomers *Filler*: 70 wt% hybrid, 2.0 μm SiO_2, Ba-glass, colloidal silica
Variolink II (A3, high viscosity)	VLII	*Monomer:* Bis-GMA, UDMA, TEGDMA *Filler:* 75.3 wt% hybrid, 0.7 μm Ba-glass, YTF-glass, Ba-Al-F-Si-glass
Ceramic Primer		
Clearfil Ceramic Primer (single-liquid)	CCP	Ethanol MDP MPTS
Monobond S (single-liquid)	MBS	52% ethanol 47% water 1% MPTS

Bis-GMA = bis-phenol-A-diglycidylmethacrylate; TEGDMA = triethyleneglycol dimethacrylate; UDMA = urethane dimethacrylate; MDP = 10-methacryloxydecyl dihydrogen phosphate; MPTS = 3-methacryloxypropyl trimethoxysilane; Ba = barium; YTF = ytterbium; Al = aluminum; F = fluorine; Si = silicon.

cutting saw (Isomet, Buehler Ltd., Lake Bluff, IL). Prefabricated ceramic material was mainly composed of SiO_2, K_2O, and Al_2O_3, and the main precipitated crystal was leucite $K_2O{\cdot}Al_2O_3{\cdot}4SiO_2$. The ceramic plates were sanded to a flat surface by hand grinding on wet 320-, 400-, 600-, and 800-grit silicon carbide paper and cleaned ultrasonically in distilled water for 5 minutes. The final thickness of each ceramic plate was 1.0, 2.0, or 3.0 mm.

The preparation of test specimens for Knoop hardness and the procedure for measurements of Knoop hardness were previously described.[32] A piece of adhesive polyethylene tape with a circular hole 5 mm in diameter was positioned on the surface of each thickness of machinable ceramic plate to control the cement layer, which had a thickness of approximately 50 μm. A small amount of product was placed on each thickness of ceramic surface within the circle. The ceramic plate with resin cement paste was placed on a clear micro cover glass (thickness 0.15 mm, Matsunami Glass Ind., Ltd., Tokyo, Japan) over a zirconia ceramic block (thickness 2 mm) to obtain a flat surface. A thin layer of resin cement was sandwiched between each thickness of ceramic plate with adhesive polyethylene tape and glass.

The dual-cured resin cement material was polymerized through each thickness of machinable ceramic using a halogen visible-light-curing unit (Candelux VL-5, J Morita Mfg., Corp., Kyoto, Japan) with 800 mW/cm^2 intensity and an 11-mm tip, at irradiation times of 40 seconds. After curing, the adhesive tape was carefully removed from the ceramic surface. Other specimens were made directly with visible-light irradiation on the clear glass for 40 seconds (not through machinable ceramic, 0 mm thickness) to establish a controlled hardness for each resin material. Each group contained five specimens.

Five measurements of hardness in the layer of resin luting agent on the ceramic surface were recorded at a post-irradiation time of 24 hours from each specimen using a microhardness tester (MVK-E, Akashi Co., Ltd., Tokyo, Japan). A Knoop diamond indenter was applied under a load of 50 g for 30 seconds, and the length of the indentation's long diagonal was measured after the applied load was removed. The specimens were stored dry in a light-proof container at 25°C except for during measurements.

Preparation of Specimens for Bending Tests

Rectangular cross-sectional area specimens with a 25-mm length, 2-mm width, and 2-mm height were obtained using a Teflon split mold (thickness 2.0 mm) according to ISO 4049.[33] Equal amounts of base and catalyst pastes of resin luting agent were mixed according to the manufacturers' directions and inserted into the mold placed on a micro cover glass. Each of the three thicknesses of machinable ceramic plates was placed between the micro cover glass and the tip of the halogen visible-light-curing unit. Photo-activation was performed only on the upper surface of the specimen, and the luting agent was irradiated through ceramics divided by three sections for 40 seconds each to polymerize the full length of the specimens. Other specimens were made directly with visible-light irradiation on the clear glass (not through machinable ceramic, 0 mm thickness) to establish the controlled properties for each luting agent material. Each group contained seven specimens.

According to ISO 4049, specimens of photo-activated materials should be irradiated by placing the tip of the light source at the center of the specimen and activating for the recommended exposure time. This procedure should be continued for the entire length of the specimen and repeated on the other side of the specimen; however, in this study only one side of the specimens was irradiated, simulating the clinical situation.

Bending Tests Procedure

The specimens prepared were allowed to stand for 30 minutes at room temperature, and then stored in distilled water at 37°C for 24 hours. The flexural strength was then measured with a universal testing machine (DCS-500, Shimadzu Corp., Kyoto, Japan) at a crosshead speed of 1.0 mm/min. Flexural strength testing was performed in a 3-point bending mode with a span length of 20 mm. Flexural modulus values were also calculated from the normal linear portion of the force-deflection curve.

The means and standard deviations for the Knoop hardness, flexural strength, and flexural modulus were computed and were compared using two-way ANOVA and Student-Newman-Keuls tests with the type of resin luting agent and the ceramic thickness as independent factors at a significance level of 0.05. The Pearson's correlation coefficient and corresponding level of significance were calculated to analyze a possible correlation between each property.

Preparation of Specimens for Shear Bond Tests

Two different-sized zirconia disks (diameters of 10 mm and 8 mm, and a thickness of 2.5 mm) of Y-TZP with Cercon (DeguDent GmbH, Hanau, Germany) were fabricated according to the manufacturers' directions. Half the ceramic specimens were air-abraded with 50 μm alumina particles at an air pressure of 0.4 MPa (Air-Jet, Morita Corp., Osaka, Japan) for 15 seconds at a distance of 10 mm, and then ultrasonically cleaned in distilled water for 5 minutes (sand blasted [SB]). A piece of polyethylene tape with a circular hole 4 mm in diameter was positioned on the surface of the

10 mm diameter × 2.5 mm thick zirconia ceramic specimen to control the area of the bond. On two sizes of zirconia ceramic specimen surfaces sanded or unsanded, each ceramic primer was applied according to manufacturers' directions, air-dried for 5 seconds, and bonded together with each resin luting agent. A sample holder secured the bonded specimens in a rigid position during bonding and controlled the cement film thickness to approximately 50 μm. Excess cement was removed before complete hardening of the resin luting agent. The dual-cured resin luting agent was irradiated from four directions for 20 seconds, for a total exposure time of 80 seconds using the visible-light-curing unit. The specimens were allowed to stand for 30 minutes at room temperature. The specimens were assigned randomly to one of the four test groups: CCP/CEC, SB + CCP/CEC, MBS/VLII, and SB + MBS/VLII, and divided into two subgroups of seven specimens each. One of the two subgroups was stored in distilled water at 37°C for 24 hours. The remaining subgroup was stored in distilled water at 37°C for 24 hours and followed by 5000 thermocycles between water baths (Rika-Kogyo, Hachioji, Japan) held at 4°C and 60°C with a dwell time of 1 minute in each bath. Thermal cycling was performed to evaluate the durability of the bond.

Shear Testing Procedure

Each specimen was embedded in an acrylic resin mold and arranged in an ISO/TR 11405 shear-testing jig. Shear tests were performed, using a method previously described,[29] with the universal testing machine at a crosshead speed of 0.5 mm/min. The calculated shear bond strength was determined by dividing the force at which bond failure occurred by the bonding area. The means for each group were analyzed by two-way analysis of variance (ANOVA) with the shear bond strength as the dependent variable and the combinations of surface treatment and resin luting agent and storage conditions of specimens as independent factors. The Student-Newman-Keuls test with $p < 0.05$ was used to establish significance.

Results

Table 2 shows the mechanical properties of two dual-cured resin luting agents through different thicknesses of machinable ceramic. Variolink II revealed a statistically higher Knoop hardness and flexural modulus than Clearfil Esthetic Cement for each thickness of machinable ceramic. No significant differences in flexural strength were observed between VLII and CEC for each ceramic thickness. Reduction of the mechanical properties with increase of ceramic thickness varied for each property; however, these properties were similar between the two materials. Statistically significant correlations could be detected between hardness and flexural modulus in CEC (r = 0.973, $p = 0.0323$) and hardness and flexural strength in VLII (r = 0.964, $p = 0.0459$) with Pearson's correlation coefficient and respective p values (Table 3).

Table 4 shows the shear bond strengths of two resin luting agents to Cercon zirconia ceramic with or without sandblasting with alumina using each one-bottle ceramic primer (CCP

Table 2. Mechanical Properties of Two Dual-cured Resin Luting Agents Through Different Thicknesses of Machinable Ceramics

Property	*Machinable Ceramic Thickness (mm)*	*Resin Luting Agent*	
		Clearfil Esthetic Cement (CEC)	*Variolink II (VLII)*
Knoop hardness number mean ± SD (KHN)	0	37.3 ± 0.9^{b}	$^{*}40.5 \pm 0.5^{d}$
	1	$36.3 \pm 1.2^{a,b}$	$^{*}40.4 \pm 0.8^{d}$
	2	34.8 ± 1.6^{a}	$^{*}38.8 \pm 0.3^{c}$
	3	34.8 ± 0.9^{a}	$^{*}37.5 \pm 0.5^{b}$
Flexural strength mean ± SD (MPa)	0	167.9 ± 5.5^{c}	162.6 ± 20.1^{c}
	1	163.2 ± 7.2^{c}	161.8 ± 7.2^{c}
	2	$157.9 \pm 5.9^{b,c}$	$154.9 \pm 17.4^{b,c}$
	3	$145.0 \pm 10.8^{a,b}$	137.9 ± 10.1^{a}
Flexural modulus mean ± SD (GPa)	0	8.81 ± 0.44^{c}	10.82 ± 0.52^{e}
	1	7.85 ± 0.49^{b}	10.28 ± 0.45^{d}
	2	7.32 ± 0.60^{a}	10.01 ± 0.22^{d}
	3	7.16 ± 0.41^{a}	9.80 ± 0.31^{d}

Identical letters were not significantly different at each property by Student-Newman-Keuls test ($p > 0.05$).
*Reference no. 32.

Table 3. Pearson's Correlation Coefficient and Respective p Value Between Each Property

Material	*Knoop Hardness-Flexural Strength*	*Knoop Hardness-Flexural Modulus*	*Flexural Strength-Flexural Modulus*
Clearfil Esthetic Cement (CEC)	$0.841, p = 0.2204$	$0.973, p = 0.0323$	$0.845, p = 0.2150$
Variolink II (VLII)	$0.964, p = 0.0459$	$0.870, p = 0.1827$	$0.803, p = 0.2678$

or MBS) at 0 and 5000 thermocycles. SB was significantly effective for increasing shear bond strength of both resin luting agents compared to with and without sandblasting before and after thermal cycling. The use of CCP could maintain shear bond strength after 5000 thermocycles. There were significant differences between bond strengths before and after thermal cycling for the MBS/VLII and SB + MBS/VLII groups ($p < 0.05$).

Discussion

Dual-cured resin luting agents have been recommended for luting ceramic or resin composite restorations to compensate for the attenuation of the curing light and to allow complete polymerization of the resin luting agent even at the bottom of the cavity or at abutting teeth, where limited curing light reaches. Mechanical properties for resin luting agents have been evaluated in photoactivation through a 2.5-mm-thick ceramic[34] or a 2.0-mm composite[35] spacer, which was used to approximate the conditions of the experiment to those found in clinical practice. However, restorations with different thicknesses are clinically luted to the cavity or abutment teeth mostly using dual-cured resin luting agents. It is well known that the light intensity reaching the resin luting agent is greatly reduced when light is transmitted through a ceramic or composite restoration. The intensity decreases exponentially as a function of the restoration thickness. An intensity of 800 mW/cm^2 was reduced to approximately 310, 160, and 80 mW/cm^2 when light was transmitted through 1-, 2-, and 3-mm thick machinable ceramic spacers.[32] Therefore, longer exposure[36,37] or multiple directed exposures[31] are recommended to diminish the effects of the attenuation of the light that reaches the dual-cured resin luting agents.

Table 4. Shear Bond Strength (measurement in MPa, mean ± SD) of Resin Luting Agent to Zirconia Ceramic at 0 and 5000 Thermocycles

Group	*Thermocycle 0*	*Thermocycle 5000*
CCP/CEC	51.0 ± 3.0^{d}	51.5 ± 6.7^{d}
SB + CCP/CEC	82.2 ± 1.8^{f}	80.0 ± 7.2^{f}
MBS/VLII	27.2 ± 2.2^{b}	8.4 ± 2.2^{a}
SB + MBS/VLII	68.2 ± 6.0^{e}	34.0 ± 1.9^{c}

Identical letters were not significantly different by Student-Newman-Keuls test ($p > 0.05$).

The mechanical properties of resin luting agents are influenced by the type and composition of the matrix resin, type and content of the filler, and mode of polymerization. The filler particles incorporated in the matrix provide better mechanical properties than the matrix itself. A correlation between filler content and hardness has been reported.[38] VLII, which contains higher filler load than CEC, also showed higher hardness and flexural modulus than CEC, regardless of ceramic thickness; however, in this study, differences observed in hardness and flexural modulus values between VLII and CEC did not correspond to differences in flexural strength in all ceramic thickness spacers. VLII and CEC showed similar flexural strength for each ceramic spacer. After the threshold network to produce resin with high strength is formed, the strength becomes less dependent on the degree of polymerization.[39] Irradiation through porcelain significantly reduced the hardness of only light-cured composites.[36,40] With the dual-cured resin cement, our results were in agreement with other studies.[34] In contrast, no reduction of the mechanical properties irradiated through machinable ceramic was observed for Knoop hardness of both resin luting agents as a result of the irradiation through 0- or 1-mm-thick ceramic, flexural strength through 0-, 1-, or 2-mm thick ceramic, and flexural modulus through 2- or 3-mm thick ceramic. Strong performance of both resin luting agents in flexural strength was observed, considering the low light intensity that reached the cement up to the 2-mm thick ceramic. Therefore, dual-cured resin luting agents may be preferred even for clinically esthetic restorations.

Sandblasting is effective for improving bond strength of the two resin luting agents because of the increase of the adhesive area on the zirconia ceramic surface. The bond between silica-based ceramics to resin luting agents is well established, because etching with hydrofluoric acid and application of a silane coupling agent provides good bonding. A silane coupler has the property of increasing the wettability of the ceramic surface for the resin luting agent, thus improving the ability of the ceramic surface to adhere to the resin luting agent. In addition, this facilitates bonding between the silica in the ceramic and the matrix resin monomer in the resin luting agents; however, MBS containing silane coupler could not maintain bond strength between zirconia ceramic with no silica content and resin luting agents after thermal cycling. Hydroxyl groups in 10-methacryloyloxydecyl dihydrogen phosphate (MDP) monomer may react with hydroxyl groups on the zirconia ceramic surface by dehydration-condensation, as seen in the reaction between silane couplers and hydroxyl groups on the silica surface.[29] As a result, CEC showed significant shear bond strength after thermal cycling using CCP containing MDP monomer.

Conclusions

Under the conditions of this study, VLII revealed a higher Knoop hardness and flexural modulus than CEC for each thickness of ceramic. No significant differences in flexural strength were observed between VLII and CEC for each ceramic spacer. Reduction of the mechanical properties with increase of ceramic thickness varied for each property; however, these properties were similar between the two materials. The bond strength of resin bonding systems of one-bottle ceramic primer, including CCP, appears adequate for zirconia ceramic restorations.

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