Evaluating the Bonding Performance of a Novel Dual-curing Composite Cement to Zirconia

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Purpose: To investigate the bond strength and durability of a novel dual-curing composite cement to zirconia under different curing conditions.

Materials and Methods: Zirconia plates of different thickness (0.5, 1, and 2 mm) were bonded with either a novel dual-curing composite cement (Panavia V5, PV5, Kuraray Noritake) or a traditional one (RelyX Ultimate, RUL, 3M Oral Care; Multilink Automix, MLA, Ivoclar Vivadent), in light-, self-, or dual-curing mode. Bonded specimens were subjected to shear bond strength (SBS) tests after 24 h of water storage or after artificially aging by 20,000 thermal cycles plus 150 days of water storage. The degree of conversion (DC) of the composite cements under different curing conditions was measured by Fourier transform infrared (FTIR) spectroscopy. The irradiance and translucency of the zirconia plates of different thickness were also investigated.

Results: The irradiance and translucency of zirconia decreased significantly with increasing thickness ($p = 0.00$). Both before and after aging, SBS of PV5 in self-curing mode was significantly higher than that of RUL ($p = 0.07$ before aging and 0.02 after aging) and MLA ($p = 0.00$ both before and after aging). However, for the three composite cements, light- and dual curing yielding the same SBSs for a constant Y-TZP thickness ($p > 0.05$). The FTIR analysis showed that, for all three dual-curing composite cements examined in this study, the mean DC values obtained in dual-curing mode were lower than those achieved in light-curing mode ($p = 0.00$ for PV5, RUL, and MLA). For RUL and MLA, lower mean DC values were obtained in self-curing than dual-curing mode ($p = 0.00$ for both RUL and MLA), while the DC values of PV5 showed no significant difference between self-curing and dual-curing mode ($p = 0.33$).

Conclusion: When the photoactivation time is 60 s and the thickness of the zirconia restoration is less than 2 mm, it is safe to use the two traditional dual-curing composite cements RUL and MLA and PV5 for bonding zirconia. However, when the light exposure time is insufficient, PV5 provides improved bond strength and durability to zirconia.

Keywords: dual-cure, curing mode, zirconia, thickness, shear bond strength, degree of conversion.

J Adhes Dent 2021; 23: 569–578.
doi: 10.3290/j.jad.b2288181

Submitted for publication: 08.12.20; accepted for publication: 22.07.21

The long-term stability of all-ceramic restorations depends on the reliability of the bond.[12] Composite cements were initially employed for luting all-ceramic restorations because of their advantageous color matching, high retentive strength,[22] resistance to wear,[28] and low solubility.[14] However, the bonding quality of the composite cement/dentin and composite cement/ceramic interfaces is seriously affected by the conversion degree of the composite cement.[6]

Zirconia is one of the most popular all-ceramic materials used for inlays, crowns, post-core crowns, and fixed dentures. Its poor transparency promoted the widespread use of dual-cure systems for composite cements, involving both

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a photoinitiator and a self-cure initiator.[26] Although the minimum thickness of some zirconia restorations is required to be 0.5 mm at the axial wall,[4,32] in the case of inlays or full crowns, the recommended occlusal thickness of zirconia restorations is usually 2 mm.[21,32] In addition, the thickness of a zirconia fixed partial prosthesis may be much larger than 2 mm under certain clinical options, such as endocrowns.[30,31] Sufficient polymerization of the dual-cure composite cement beneath the zirconia ceramic is of great importance for achieving effective bonding.[3,9] Previous studies found that the polymerization of most dual-cure composite cements depends on photoactivation, because self-curing alone fails to ensure a sufficient degree of polymerization.[25,33] Insufficient light exposure negatively affects the physicochemical properties of dual-cure composite cements, which weakens the bonding at the cement/ceramic and cement/tooth interfaces, even after light exposure.[27,29] The blue light intensity reaching the cement beneath the restoration is one of the critical factors for optimizing the properties of dual-cure composite cements.[18] Unfortunately, previous studies revealed that the light intensity reaching the underlying dual-cure composite cement decreases with increasing thickness of zirconia, which in turn leads to bonding failure.[19,31]

In the attempt to overcome incomplete curing of the underlying dual-cure composite cement, such as that beneath an excessively thick zirconia layer, a novel amine-free, self-cure redox polymerization catalyst was developed and added to the primer system of a dual-cure composite cement, Panavia V5 (Kuraray Noritake; Tokyo, Japan); its manufacturer claims that the unique “touch and cure” feature of the novel curing system endows the Panavia V5 cement with a dentin bond strength equivalent to that of the gold standard light-cure bonding agent (Clearfil SE Bond, Kuraray Noritake) in self-cure mode.

With the novel amine-free polymerization catalyst, the self-curing interaction between the primer (containing the co-initiators) and the composite cement (containing the initiator) can initiate conversion and improve the polymerization activity when the two components contact each other, even without light curing.[26] Previous studies showed that Panavia V5 had significantly stronger dentin bonding than other common dual-cure composite cements used in either dual- or self-cure modes.[18,26] The excellent mechanical properties of Panavia V5 have also been confirmed by in vitro experiments.[18,24] All the above studies suggest that Panavia V5 may be a good candidate for luting zirconia restorations, especially restorations with an very thick ceramic layer.

In this study, we evaluated the shear bond strength (SBS) and durability of Panavia V5 bonded to zirconia substrates of different thicknesses under light-, dual-, and self-curing conditions. We also investigated the transparency and light transmission of zirconia layers of different thickness, as well as the degree of conversion (DC) of the underlying resin cement layer. The null hypothesis tested in the study was that the curing conditions would not affect the polymerization of Panavia V5 or its bonding performance to zirconia.

MATERIALS AND METHODS

Irradiance Measurements

Machinable Y-TZP blocks (Cerec Zirconia, CZ, Dentsply Sirona; Bensheim, Germany) were sectioned into plates of three different thicknesses. All specimens were wet-ground with 600-grit silicon carbide abrasive paper, followed by complete sintering according to the manufacturer’s instructions (Table 1), to produce yttria partially stabilized tetragonal zirconia (Y-TZP) plates with side lengths of 10 mm x 10 mm and thicknesses of 0.5, 1, and 2 mm. The number of obtained 0.5-, 1-, and 2-mm-thick Y-TZP plates was 180, 120, and 120, respectively.

The irradiance (mW/cm^2) at the light guide tip of an LED light-curing unit (EliparTM S10, 3M Oral Care; St Paul, MN, USA) was measured by a curing radiometer (Check Marc, BlueLight Analytics; Halifax, Nova Scotia, Canada).

The light transmission values of the light-curing unit through the 0.5-, 1-, and 2-mm-thick Y-TZP plates (n = 5) were measured by placing the plate on the aperture of the curing radiometer.

Translucency

Ten Y-TZP plates were tested for each thickness value. The translucency parameters (TPs) of these plates were determined by calculating the color difference between readings against black (B) and white (W) backgrounds for the same specimen. Ten data points were obtained for each group, and the CIELAB parameters (L*, a*, and b*) were estimated using a dental colorimeter (ShadeEye; Shofu, Japan). The TPs were calculated according to the following equation:[10]

$$\sqrt{(L*_B - L*_w)^2 + (a*_B - a*_w)^2 + (b*_B - b*_w)^2}$$

where B and W denote color values on a black and white background, respectively, and L*, a*, and b*represent lightness, red-green axis, and yellow-blue axis, respectively.

SBS Tests

Three dual-curing composite cements Panavia V5 (PV5, Kuraray Noritake; Tokyo, Japan), RelyX Ultimate (RUL, 3M Oral Care), and Multilink Automix (MLA, Ivoclar Vivadent; Schaan, Liechtenstein) were examined in this study.

The 420 Y-TZP plates prepared in this study were subjected to air-particle abrasion at 0.25 MPa for 20 s, using 50-µm alumina particles from a distance of 10 mm (JNBP-2, Jianian Futong Medical Equipment; Tianjin, China). Then, the plates were ultrasonically cleaned in anhydrous ethanol and distilled water in an ultrasonic bath for 5 min and dried with an oil-free air spray.

A total of 420 pre-polymerized composite cylinders were made from a light-curing composite (Filtek Z250, 3M Oral Care) using nylon molds with an inner diameter of 3 mm and a height of 2 mm.

To prepare bonding specimens, the Y-TZP plates were divided into nine groups according to the Y-TZP thickness

Table 1 Materials used in this study

Material/ trade name	Type	Composition	Manufacturer [lot no.]	Application procedure
Cerec Zirconia (CZ, A2)	3Y-TZP	$ZrO_2 + HfO_2 + Y_2O_3 \geq 99.0\%$ $4.5\% < Y_2O_3 \leq 6\%$ $HfO_2 \leq 5\%$, $Al_2O_3 \leq 0.07\%$ Other oxides ≤ 1.1%	Dentsply Sirona; Bensheim Germany [3314000013]	Sintering according to manufacturer's instructions: heating rate 15°C/min, holding temperature 1510°C, holding time 2 h, cooling rate 30°C/min.
Panavia V5 (PV5, Universal)	Dual-curing composite cement	Paste A: bis-GMA, TEG-DMA, hydrophobic aromatic dimethacrylate, hydrophilic aliphatic dimethacrylate initiators, accelerators, silanated barium glass filler, silanated fluoroaluminosilicate glass filler, colloidal silica Paste B: bis-GMA, hydrophobic aromatic dimethacrylate, hydrophilic aliphatic dimethacrylate, silanated barium glass filler, silanated aluminum oxide filler, accelerators, dl-camphorquinone, pigments	Kuraray Noritake; Tokyo, Japan [4h0076]	Apply by auto-mix syringe on 3Y-TZP surface followed by light-, self-, or dual-curing.
RelyX Ultimate (RUL, A1)	Dual-curing composite cement	Base paste: methacrylate monomers containing phosphoric acid groups, methacrylate monomers, silanated fillers, alkaline fillers. Catalyst paste: methacrylate monomers	3M Oral Care; St Paul, MN, USA [4801907]	Apply by auto-mix syringe on Y-TZP surface followed by light-, self-, or dual-curing.
Multilink Automix (MLA, yellow)	Dual-curing composite cement	Base paste: bis-GMA, HEMA, 2-dimethylaminoethyl methacrylate. Catalyst paste: ethyoxylated bisphenol A dimethacrylate, UDMA, HEMA	Ivoclar Vivadent; Schaan, Liechtenstein [W34404]	Apply by auto-mix syringe on Y-TZP surface followed by light-, self-, or dual-curing.
Single Bond Universal (SBU)	Universal adhesive	Vitrebond copolymer, MDP, silane	3M Oral Care [91024B]	Apply and leave on Y-TZP for 20 s and air-dry gently for 5 s, then light-cure for 10 s.
Filtek Z250	Resin composite	Bis-GMA, UDMA, bis-EMA, TEG-DMA, zirconia/silica	3M Oral Care [NA35876]	Fill composite into mold cavities (2 mm in height and 3 mm in inner diameter) and keep in place with plastic foil and a glass plate on each side, then light cure for 60 s on both sides.

Bis-GMA: bisphenol A diglycidylether methacrylate; TEG-DMA: triethylene glycol dimethacrylate; UDMA: urethane dimethacrylate; MDP: methacryloyloxydecyl dihydrogen phosphate; HEMA: 2-hydroxyethyl methacrylate.

and composite cements used (PV5-0.5, -1, -2; RUL-0.5, -1, -2; MLA-0.5, -1, -2).

The bonding surface of each Y-TZP plate was treated with Single Bond Universal adhesive (SBU, 3M Oral Care) for 20 s, gently air dried for 5 s, and then light cured for 10 s. A piece of tape with a circular hole 3 mm in diameter was placed on the treated Y-TZP surface to define the bonding area. The pre-polymerized composite cylinders were then cemented under a constant load of 5 N to create bonding specimens. The materials used in this study are described in detail in Table 1.

The 0.5-mm Y-TZP group was then divided into three subgroups (n = 20) according to the curing mode of the composite cement (light-, dual-, or self-curing). The 1- and 2-mm Y-TZP groups were divided into two subgroups (n = 20) corresponding to light- or dual-curing of the composite cements. The corresponding procedures for each group were as follows:

- Light-curing subgroup (LC): the bonding specimen was light cured for 60 s from the zirconia surface direction and then wrapped in tin foil to avoid light exposure.
- Dual-curing subgroup (DC): the bonding specimen was light cured for 20 s from the zirconia direction and then wrapped in tin foil to avoid light exposure.
- Self-curing subgroup (SC): the bonding specimen was placed in the dark for 30 min to polymerize the cement by self-cure activation only, followed by wrapping in tin foil to avoid light exposure.

The bonded specimens in each group were stored in water at 37°C for 24 h, or artificially aged by 20,000 thermal cycles (30-s immersion in 5 and 55°C baths) followed by 150 days of water storage at 37°C. The samples were then subjected to SBS tests using a universal testing machine (Instron Model 3365, ElectroPuls; Canton, MA, USA) with a crosshead speed of 1.0 mm/min. The maximum load (N) was recorded when the bonded specimen was loaded to failure, and the SBS (MPa) was calculated according to the following formula: SBS (MPa) = maximum load (N) / bonding interface area (mm^2).

The SBSs in MPa were recorded for all groups. After examining the normal distribution and homogeneity of

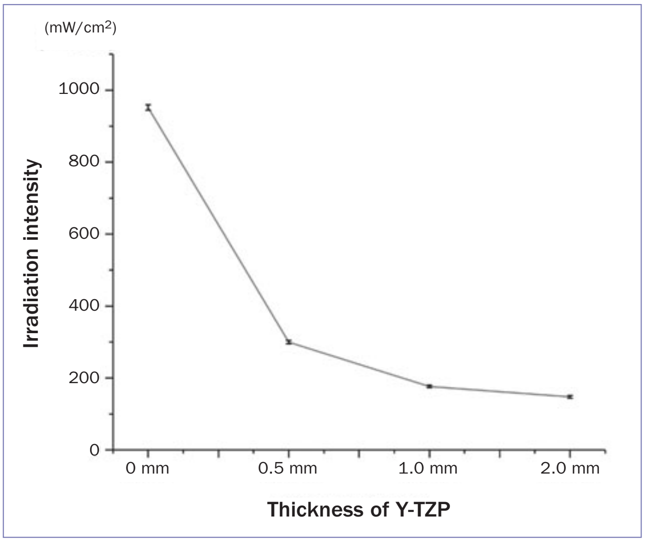

Fig 1 Irradiance of Y-TZP with different thickness.

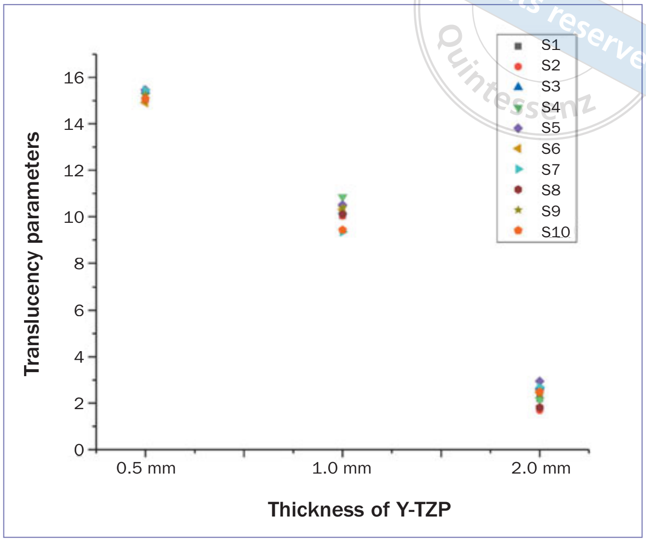

Fig 2 Translucency parameter of Y-TZP with different thickness. Different icons labeled S1 to S10 represent different samples of Y-TZP of the same thickness.

these values, two-way ANOVA and Tukey's post-hoc honestly significant difference (HSD) tests were performed to evaluate the effects of different Y-TZP thickness and curing modes on the bonding properties of the composite cements to zirconia, using SPSS 22.0 software (IBM SPSS; Armonk, NY, USA). Statistical significance was set at $\alpha = 0.05$.

After the SBS tests, the interfaces of all fractured specimens were examined under a stereomicroscope (C-DSS230, Nikon; Tokyo, Japan), and the fracture mode was determined according to the following classification:

- Adhesive failure (A): fracture sites were entirely located between the composite cement and the zirconia surface, with no residual composite cement on the surface of Y-TZP.
- Cohesive failure (C): fractures occurred exclusively within the resin composite/composite cement.
- Mixed failure (M): partial fractures occurred in the resin composite/composite cement, with partial exposure of the Y-TZP plate surface.

DC Measurements

The DC of composite cements treated under different curing conditions was measured by a FTIR spectrometer coupled with an attenuated total reflectance (ATR) accessory (Smart iTR Nicolet iS10, Thermo Scientific; Waltham, MA, USA).

The samples were divided into groups according to their Y-TZP thickness, curing mode, and type of composite cements used in the SBS test. Three specimens were tested for each group. Observation times of 24 h and 1 week were selected for each group.

FTIR spectra in the 4000–400 cm^{-1} region were recorded from 32 scans at a resolution of 4 cm^{-1} in ATR mode. The DC of the composite cements was determined from the aliphatic C=C peak at 1638 cm^{-1}, using the aromatic C=C peak at 1608 cm^{-1} for internal calibration. In particular, the DC was calculated using the following formula:

$$DC\% = (1 - R_{cured}/R_{uncured}) \times 100\%$$

where R_{cured} and $R_{uncured}$ are the ratios of the peak areas at 1638 cm^{-1} and 1608 cm^{-1} before and after curing of the composite cement, respectively.

Two-way ANOVA was used to evaluate the effects of different Y-TZP thicknesses and curing modes on the DC of each group, using SPSS 22.0, with $\alpha = 0.05$ indicating statistical significance.

RESULTS

Irradiance Measurements

The results of the irradiance measurements are presented in Fig 1. The irradiance at the light guide tip of the curing light was 951.90 ± 7.26 mW/cm^2. The irradiance of the 0.5, 1, and 2 mm-thick Y-TZP plates were 299.80 ± 4.87 mW/cm^2 ($p = 0.00$), 176.60 ± 3.60 mW/cm^2 ($p = 0.00$), and 147.60 ± 3.69 mW/cm^2 ($p = 0.00$), respectively.

Translucency

Figure 2 shows a scatter diagram of the TPs of the three Y-TZP plates of different thickness. TP of the 0.5-, 1-, and 2-mm-thick Y-TZP plates was 15.23 ± 0.20, 10.12 ± 0.46, and 2.35 ± 0.40, respectively.

One-way ANOVA revealed significant differences between TP measured with different Y-TZP thicknesses, and Tukey's post-hoc test showed that the TPs decreased significantly with increasing thickness.

Table 2 Shear bond strengths (MPa) and standard deviations of all groups

			Thickness					
			0.5 mm		1.0 mm		2.0 mm	
Group	Cement	Curing mode	24-h water storage	20,000 thermal cycles + 150-day water storage	24-h water storage	20,000 thermal cycles + 150-day water storage	24-h water storage	20,000 thermal cycles + 150-day water storage
PV5-LC (-0.5, -1, -2)	Panavia V5	Light curing	18.11 ± 2.67^a	13.21 ± 1.88^A	17.70 ± 2.07^a	13.42 ± 2.25^A	18.04 ± 1.43^a	12.90 ± 2.80^A
PV5-DC (-0.5, -1, -2)		Dual curing	16.65 ± 1.26^b	13.18 ± 2.17^A	16.68 ± 2.56^b	13.36 ± 0.74^A	16.39 ± 1.90^b	13.27 ± 2.54^A
PV5-SC (-0.5, -1, -2)		Self curing	16.70 ± 1.89^b	10.17 ± 1.89^B	/	/	/	/
RUL-LC (-0.5, -1, -2)	RelyX Ultimate	Light curing	17.85 ± 2.30^a	9.85 ± 2.00^B	17.69 ± 2.51^a	9.68 ± 2.28^B	18.90 ± 4.26^a	9.40 ± 1.28^B
RUL-DC (-0.5, -1, -2)		Dual curing	16.49 ± 2.73^b	10.31 ± 2.79^B	16.30 ± 1.68^b	10.62 ± 1.77^B	16.42 ± 2.44^b	9.71 ± 1.47^B
RUL-SC (-0.5, -1, -2)		Self curing	13.73 ± 2.63	7.41 ± 1.56^C	/	/	/	/
MLA-LC (-0.5, -1, -2)	Multilink Automix	Light curing	18.21 ± 3.28^a	13.44 ± 2.22^A	18.04 ± 2.85^a	13.39 ± 1.72^A	18.95 ± 2.34^a	13.34 ± 0.69^A
MLA-DC (-0.5, -1, -2)		Dual curing	16.7 ± 1.48^b	13.44 ± 1.58^A	16.54 ± 2.06^b	13.12 ± 1.88^A	16.82 ± 1.84^b	13.37 ± 2.31^A
MLA-SC (-0.5, -1, -2)		Self curing	7.63 ± 1.23^d	1.01 ± 1.20^D	/	/	/	/

All values are reported as mean ± SD. Before aging, values marked with the same superscript lowercase letter are not statistically significantly different ($p > 0.05$). After aging, mean values marked with the same superscript capital letter are not statistically significantly different ($p > 0.05$).

SBS Tests

The results of the SBS tests for all groups before and after aging are shown in Table 2. Two-way ANOVA revealed that the SBSs before aging were affected by the curing mode ($p = 0.03$ for PV5, $p < 0.001$ for RUL and MLA), but not by the Y-TZP thickness ($p = 0.95$ for PV5, 0.73 for RUL, and 0.70 for MLA). Moreover, no significant correlations emerged between Y-TZP thickness and curing mode ($p = 0.88$ for PV5, 0.77 for RUL, and 0.87 for MLA). In the case of RUL and MLA, Tukey's post-hoc test revealed significant differences between light-curing and dual-curing groups ($p = 0.045$ for RUL and 0.014 for MLA), between light-curing and self-curing groups ($p = 0.00$ for both RUL and MLA), as well as between dual-curing and self-curing groups ($p = 0.026$ for RUL and 0.00 for MLA). However, for PV5, RUL, and MLA under the same curing conditions, no significant differences in SBS were found between groups with different Y-TZP thicknesses ($p > 0.05$).

SBS of PV5 in self-curing mode was the highest; in particular, this value was significantly higher than that of RUL ($p = 0.07$) and MLA ($p = 0.00$). However, no significant difference in SBS was found between PV5, RUL, and MLA in light-curing ($p = 0.97$) or dual-curing ($p = 0.44$) mode. The comparison of the 24-h SBS results showed that the self-curing MLA group had the lowest SBS of all groups, followed by the self-curing RUL group.

Aging resulted in a significant decrease in the SBS of all tested groups ($p < 0.05$). The SBS of the MLA-SC group was the lowest, followed by the RUL-SC and PV5-SC groups. The SBS of PV5 in self-curing mode (10.17 ± 1.89 MPa) was significantly higher than that of RUL ($p = 0.02$) and MLA ($p = 0.00$). There was no significant difference in bond strength among all PV5 groups under light-curing or dual-curing conditions ($p > 0.05$), and the same applied to the RUL and MLA groups ($p > 0.05$). Moreover, under light-curing or dual-curing conditions, no significant difference in SBS was observed between PV5 and MLA ($p > 0.05$), but both values were significantly higher than that of RUL ($p = 0.00$).

Fracture Mode Analysis

Before aging, mixed failure was the dominant fracture mechanism in all groups of the three cements examined in this study under light-curing or dual-curing conditions, regardless of the Y-TZP thickness. In self-curing mode, RUL mainly exhibited mixed failure, with only a few cohesive failure specimens; PV5 presented pure mixed failure modes, while the majority of fractured MLA specimens showed cohesive failure modes.

After thermocycling followed by water storage, 50% of the samples of the MLA-SC group exhibited spontaneous debonding, while none of the other groups except MLA-SC did so. All MLA-SC specimens presented cohesive failure, while mixed failure modes were still dominant in all other groups of the three cements.

Degree of Conversion Measurements

Figures 3 to 5 show the FTIR spectra of PV5, RUL, and MLA samples after 24 h and 1 week. The 24-h DC values of all

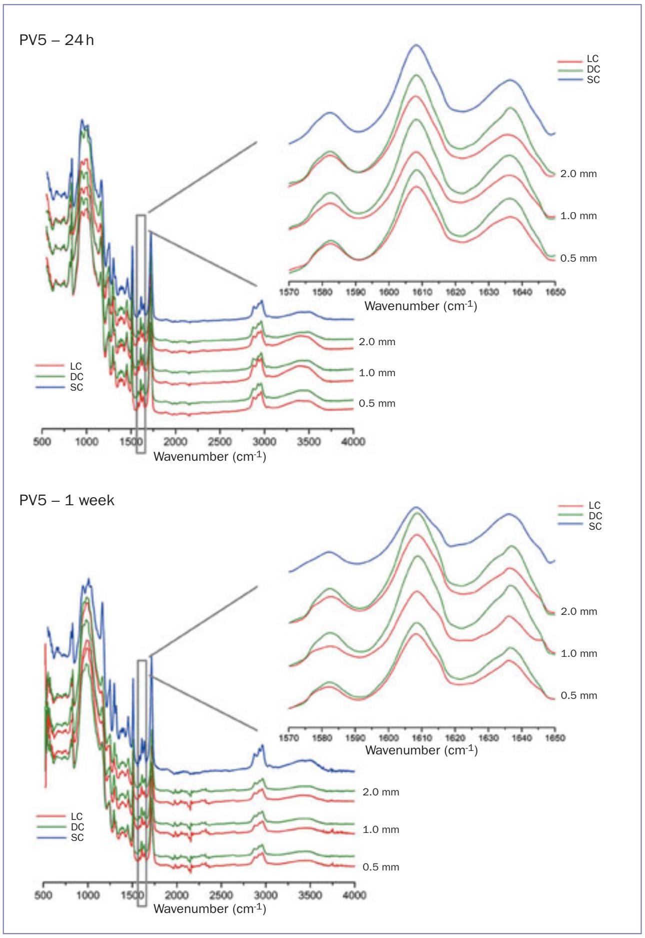

Fig 3 ATR-FTIR and the amplification of representative ATR-FTIR spectra from 1570 cm^{-1} to 1650 cm^{-1} of Panavia V5 (PV5) after 24 h and 1 week under different curing conditions. LC: light curing; DC: dual curing; SC: self-curing.

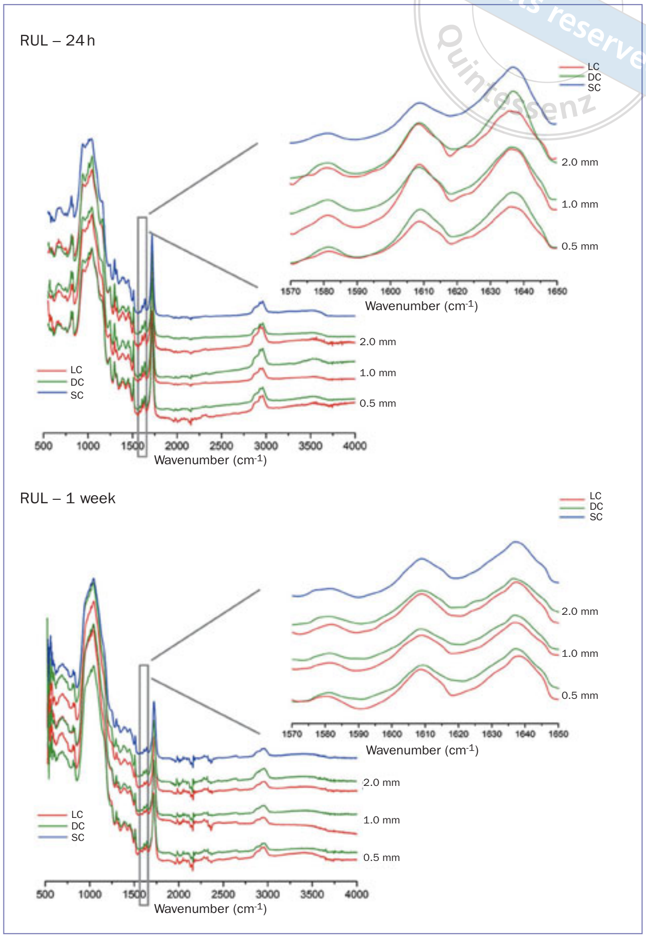

Fig 4 ATR-FTIR and the amplification of representative ATR-FTIR spectra from 1570 cm^{-1} to 1650 cm^{-1} of RelyX Ultimate (RUL) after 24 h and 1 week under different curing conditions. LC: light curing; DC: dual curing; SC: self-curing.

groups are shown in Table 3. For all three dual-cure composite cements investigated in this study, the mean DC in dual-curing mode was lower than that measured under light-curing conditions ($p = 0.00$ for PV5, RUL, and MLA). The mean DC measured for RUL and MLA under self-curing conditions was lower than that obtained in dual-curing mode ($p = 0.00$ for both RUL and MLA), while DC of PV5 obtained in self-curing and dual-curing mode showed no significant differences ($p = 0.33$). In addition, under the same curing conditions, no significant difference in DC of the same composite cement was observed between zirconia groups with different thickness ($p = 0.76$ for PV5, 0.97 for RUL, and 0.82 for MLA).

For all three dual-cure composite cements, the 1-week DC of all groups (Table 4) under self-curing conditions was significantly higher than the 24-h ones ($p = 0.00$), while no difference was observed between the 1-week and 24-h DC under light-curing or dual-curing conditions ($p > 0.05$).

DISCUSSION

In order to elucidate the relationship between Y-TZP thickness, light attenuation, and bonding strength, we tested the transparency of Y-TZP plates of different thicknesses based on the irradiance of the light source after passing through the Y-TZP plate. The zirconia used in the current study is a high-translucency material. However, the quantitative analysis of light attenuation showed that, compared with the 0-mm case, with increasing Y-TZP thickness, the irradiance from the light-curing unit reaching the composite cement under the Y-TZP plate decreased significantly. The irradiance measured through the 2-mm Y-TZP plate showed a decrease of more than 80%. This is consistent with the decreasing trend of the transparency of Y-TZP with increasing thickness of Y-TZP.[5] The sharp drop in irradiance under the 2-mm Y-TZP plate would severely affect the polymerization of dual-curing composite cements, which mostly depend on photoactivation. If the Y-TZP plate is thick enough to completely block light transmission, the polymerization of composite cements must be performed in self-curing mode to achieve bonding. In order to determine whether the novel PV5 product can provide sufficient bonding strength to Y-TZP under insufficient light conditions, as claimed by the manufacturer,[11] two dual-cure composite cements widely used for luting zirconia restorations were chosen as control; the three dual-composite cements were then evaluated under the following conditions: light-curing with sufficient light, dual-curing with insufficient light, and self-curing in complete darkness.

According to the statistical analysis of the SBS results, the curing mode affected the SBS of the RUL and MLA composite cements. The SBSs obtained with the three curing modes decreased in the order of light curing > dual curing > self-curing, which is consistent with the conclusions of previous studies.[8,31] The SBS of PV5 under light-curing conditions was also higher than those obtained in dual-curing and self-curing mode, although the SBSs of the latter two modes showed no statistical differences. Therefore, the null hypothesis that the curing conditions did not affect the bonding performance of PV5 to zirconia could be rejected.

According to the FTIR analysis, DC measured for RUL and MLA in self-curing mode was significantly lower than that obtained in dual-curing and light-curing mode. Moreover, lower DC was obtained in dual-curing than light-curing mode, which was in good agreement with the SBS results. These results indicate that the high SBS of the two traditional dual-cure composite cements in light-curing mode was due to their more effective polymerization, and also suggest that light irradiation, even at insufficient levels, was necessary to improve the degree of polymerization and bonding performance of the dual-cured composite cements.

In the absence of sufficient light or under self-curing conditions, the degree of conversion of the traditional dual-curing composite cements after 20-s photoactivation pass through the Y-TZP plate was found to be significantly lower than that measured under the sufficient 60-s photoactivation time,[7,8] with a significantly negative effect on the bonding performance.[8] It would be highly desirable to remove the dependence of resin-composite cement polymerization on light exposure. On the other hand, according to the observed fracture modes under dual-curing and light-curing conditions, mixed failure modes were dominant for PV5, RUL, and MLA. In the case of MLA under self-curing conditions, the proportion of cohesive failures was up to 90%, suggesting a low DC of MLA in self-curing mode. However, for the other two composite cements in self-curing mode, the fractured specimens mainly presented mixed failure modes.

Although the irradiance measured through the 2-mm Y-TZP plate showed a decrease of more than 80% in light output, the total energy output (ie, radiant energy [J/cm^2]) was calculated by multiplying the radiant exitance (mW/cm^2) by the total exposure time (s), according to the "total energy principle" or "exposure reciprocity law".[20] It has been reported that the total irradiated energy has a close relationship with the mechanical properties of photopolymerized dental materials.[18,23] Although the clinically recommended photoactivation time of composite cements is 20 s,[13,26] in this study the light curing time was set to 60 s,[1,2] in order to achieve sufficient light curing. The present FTIR and SBS results support the effectiveness of this light exposure setup, and also show that sufficient polymerization of composite cements can be achieved for various Y-TZP thicknesses (up 2 mm) with 60 s of photoactivation. Previous studies have reported that the effect of light attenuation on the degree of conversion of composite cements due to zirconia ceramic thickness can be compensated by extending the light curing duration.[15] This shows that, although irradiance decreases with increasing Y-TZP thickness, the total radiant energy received over a sufficient photoactivation time is enough for the two traditional composite cements to reach a sufficient degree of conversion. This can offset the negative effect of light attenuation caused by ceramics with a thickness up to 2 mm on the degree of polymerization of the cements.

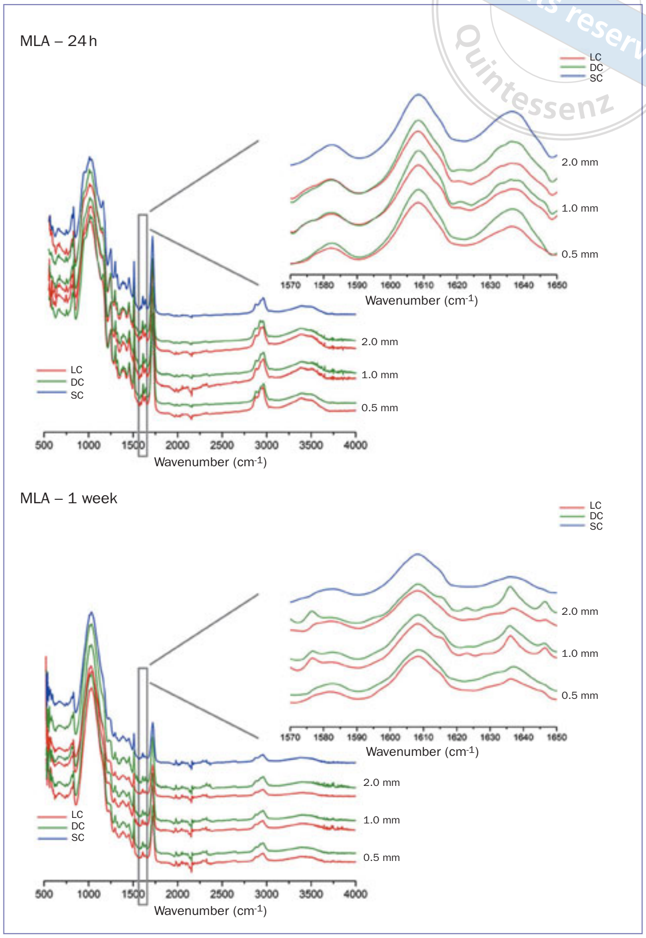

Fig 5 ATR-FTIR and the amplification of representative ATR-FTIR spectra from 1570 cm^{-1} to 1650 cm^{-1} of Multilink Automix (MLA) after 24 h and 1 week under different curing conditions. LC: light curing; DC: dual curing; SC: self-curing.

Table 3 Mean degree of conversion (%) after 24 h

	Curing condition						
	LC-0.5	LC-1.0	LC-2.0	DC-0.5	DC-1.0	DC-2.0	SC-0.5
PV5	51.217 ± 1.20^{aB}	50.54 7 ± 1.10^{aB}	51.24 7 ± 0.50^{aB}	42.757 ± 3.53^{bB}	42.38 7 ± 0.63 ^{bB}	43.49 7 ± 0.37^{bB}	40.907 ± 3.39^{bA}
RUL	50.117 ± 1.80^{aB}	51.52 7 ± 2.33^{aB}	51.63 7 ± 4.28^{aB}	43.747 ± 1.92^{bB}	43.517 ± 1.48^{bB}	41.807 ± 2.19^{bB}	26.057 ± 0.45^{cB}
MLA	59.117 ± 3.02^{aA}	58.437 ± 0.43^{aA}	58.027 ± 0.96^{aA}	50.187 ± 3.28^{bA}	50.307 ± 2.86^{bA}	49.577 ± 2.54^{bA}	43.447 ± 0.42^{cA}

All values are reported as mean ± SD. Within the same row, values marked with the same lowercase superscript letter are not statistically significantly different ($p > 0.05$). Within the same column, mean values marked with the same capital superscript letter are not statistically significantly significantly different ($p > 0.05$).

Table 4 Mean degree of conversion (%) after 1 week

	Curing condition						
	LC-0.5	LC-1.0	LC-2.0	DC-0.5	DC-1.0	DC-2.0	SC-0.5
PV5	48.13±1.73^{aB}	48.39 ± 2.97^{aB}	48.00 ± 0.47^{aB}	39.74 ± 1.18^{bC}	39.76 ± 1.69^{bC}	39.10 ± 2.19^{bC}	69.20 ± 0.91^{cA}
RUL	51.64 ± 2.53^{aB}	51.04 ± 2.68^{aB}	52.60 ± 2.72^{aB}	45.39 ± 1.18^{bB}	46.32 ± 2.12^{abB}	48.82 ± 2.04^{abB}	46.33 ± 2.65^{abB}
MLA	62.06 ± 0.90^{aA}	60.72 ± 0.56^{aA}	63.39 ± 0.70^{aA}	52.9 ± 2.48^{bA}	54.90 ± 2.39^{bA}	52.63 ± 3.86^{bA}	61.80 ± 2.38^{aA}

All values are reported as mean ± SD. Within the same row, values marked with the same lowercase superscript letter are not statistically significantly different ($p > 0.05$). Within the same column, mean values marked with the same capital superscript letter are not statistically significantly different ($p > 0.05$).

A dual-cure initiator system is designed to ensure that the self-curing component of the cementing system (eg, the benzoyl peroxide (BPO)/tertiary amine pair) achieves optimal polymerization by self-curing reactions following exposure to light. The polymerization of dual-curing composite cements is catalyzed by a chemically activated (auto-polymerization) and a photoactivated (light-curing) initiator. The polymerization reaction starts by mixing a base with a catalyst paste, thus activating the chemical initiator. However, the chemical curing process is very slow.[16] The photoinitiator triggers an intense polymerization reaction immediately after the restoration is correctly placed and cement excess is removed.[16] In the present study, the irradiation time of the dual-curing mode was set to 20 s[13,20,26] to simulate the polymerization process under clinical conditions, ie, 20-s photoactivation due to the attenuation of light intensity caused by the restoration. According to the FTIR and SBS results, when the light intensity attenuation was within 80%, the three composite cements employed in this study could still achieve similar polymerization through the dual-curing initiator system, although the degree of conversion was lower than that achieved under light-curing conditions. This indicates that, for an irradiation time of 20 s, Y-TZP plates of thickness up to 2 mm cannot allow enough radiant energy to pass and activate the light-curing mode. Under these conditions, dual-curing polymerization is activated, achieving a lower degree of polymerization than that obtained in light-curing mode. Therefore, PV5 did not show any advantages over traditional composite cements in light-curing or dual-curing mode.

Y-TZP plates with different thicknesses obviously lead to different degrees of light attenuation; however, we did not find differences in the DC and bonding performance of the three dual-cure composite cements examined in this study. This applied to both dual-curing and light-curing modes, suggesting that the polymerization of the dual-curing composite cements in dual-curing mode was more affected by the presence than by the intensity of the light. In other words, as long as photoactivation is performed, the traditional dual-cure composite cements and the new PV5 material eventually achieved a stable degree of polymerization, although their DC value was lower than that obtained in light-curing mode with sufficient irradiation. However, unlike the two traditional composite cements, FTIR analysis showed that the degree of polymerization of PV5 in self-curing mode was not significantly different from that obtained in dual-curing mode. The SBS results were consistent with those of the DC; SBS of PV5 in self-curing and dual-curing mode was not significantly different. Moreover, SBS of PV5 in self-curing mode was more than twice that of MLA, although no such difference was found between the SBS of the three composite cements employed to study Y-TZP groups with the same thickness in the other two curing modes. This highlights the advantages of PV5 over traditional composite cements. The current SBS and FTIR results indicate that the degree to which the three types of cement are affected by the curing method reflects the order of dependence on light irradiation: PV5 < RUL < MLA. PV5 maintained good bonding to zirconia under self-curing conditions, suggesting that if the zirconia restoration is too thick to allow blue-light transmission (eg, as with endocrowns), the PV5 cement under the restoration can still achieve good bonding, which represents a considerable advantage over traditional dual-cure composite cements.

Thermal stress and hydrolysis induced by thermocycling between 5°C and 55°C can simulate the effect of the varying temperatures in the oral cavity. It was suggested that 10,000 cycles may represent 1 year of service. Previous studies by our group have found that 20,000 thermocycles followed by 3 months of water storage reduced the bond strength of composite cement to zirconia.[34] The results of the present study showed that 20,000 thermocycles followed by 150 days of water storage significantly reduced the bond strength of PV5, RUL, and MLA to zirconia. The bond strength reduction in each PV5 group was similar to that of MLA and lower than that of RUL, which is a positive result. Although the bonding strength of PV5 to zirconia under self-curing conditions also decreased significantly, it still met the minimum bonding requirements of 5 MPa.[17]

CONCLUSION

Regardless of the light-curing or dual-curing mode employed, when the irradiation time reached 60 s and the thickness of the zirconia restoration was up to 2 mm, the attenuation of light caused by the increased zirconia thickness did not affect the polymerization degree or bonding performance of traditional and PV5 dual-cure composite cements. This confirms that traditional dual-curing composite cement and PV5 can be safely employed for bonding zirconia restoration.

However, the two traditional dual-cure composite cements showed a strong light dependence, so that photoactivation was a required step to achieve an effective bond, whereas PV5 could achieve a stable bond in complete absence of light. This indicates that PV5 could achieve better bonding to zirconia than the two traditional composite cements under the same conditions. Therefore, this study confirmed the superior performance of PV5, even under insufficient light or dark conditions.

ACKNOWLEDGEMENTS

The authors thank Mr. Taoran Ma (Kuraray Noritake) for providing Panavia V5. This study was supported by the National Natural Science Foundation of China (grant 81970927), the Natural Science Foundation of Jiangsu Province of China (BK20191348), the Qing Lan Project and the Priority Academic Program Development of Jiangsu Higher Education Institutions (grant 2018-87).

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Clinical relevance: Panavia V5 provides improved bonding performance to zirconia when the light exposure time was insufficient, or even without light exposure.