THE JOURNAL OF PROSTHETIC DENTISTRY

RESEARCH AND EDUCATION

The effect of monowave and polywave light-polymerization units on the adhesion of resin cements to zirconia

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ABSTRACT

Statement of problem. Although newly developed photoinitiators can make up for the limitations of camphorquinone, their absorption spectra are different. However, little attention has been paid to the compatibility of available wavelength spectra of light-polymerization units (LPUs) and photoinitiators within resin cements.

Purpose. The purpose of this in vitro study was to evaluate the effect of monowave and polywave light-emitting diode (LED) units on the adhesion of dual-polymerizing self-adhesive resin cements to monolithic zirconia.

Material and methods. Monowave LPUs and polywave LPUs were chosen to polymerize 2 dual-polymerizing self-adhesive resin cements. Ninety-six zirconia disks were randomly divided into 4 groups as different combinations of LPUs and resin cements, namely ES-U200, BS-U200, ES-SC, and BS-SC. Resin cements were adhered to zirconia disks, and the microshear bond strength (μSBS) test was conducted after 24 hours of H_2O storage (24 h) and 10 000 thermocycles (10k/TC). Failure modes were examined by stereomicroscopy and scanning electron microscopy (SEM). The degree of conversion (DC) was tested both immediately and 24 hours later. In statistical analyses, 2-way ANOVA and the Tukey honestly significant difference post hoc test were performed for μSBS and DC results, and chi-square test was performed for failure mode analysis (α=.05 for all tests).

Results. The 2-way ANOVA demonstrated that different combinations of LPUs and resin cements, as well as different artificial aging levels, significantly influenced microshear bond strength values (P<.001). The interactions between 2 factors were also significant (P<.001). The BS-SC group possessed relatively high bond strength in both 24-hour and 10k/TC aging levels. For the same resin cement, no significant difference was found in the immediate DC (P=.405 for U200 and P=.708 for SC). At 24 hours, DC and BS-U200 values were significantly higher than ES-U200 values (P=.002), whereas BS-SC values were not significantly different from ES-SC values (P=.284).

Conclusions. Within the limitation of this in vitro study, the emission spectra of LED units significantly influenced the bond strengths, DC, and failure mode of dual-polymerizing self-adhesive resin cements to zirconia at both immediate and artificial aging levels. The LPU should provide light energy to match the absorption wavelengths of photoinitiators in the resin cements. (J Prosthet Dent 2019;121:549.e1-e7)

High-translucency zirconia may be a feasible alternative to lithium disilicate for anterior restorations.[1,2] However, the effective bonding of zirconia remains problematic on account of its silica-free composition.[3] Dual-polymerizing self-adhesive resin cements are now widely used in bonding zirconia restorations. The combination of chemical and light activation increases the degree of polymerization, and the self-adhesive mechanism offers a simplified solution with little postcementation sensitivity.[4,5] Nevertheless, bonding performance is highly dependent on the 1-step cementation and light polymerization.

Light polymerization involves the combined effects of photoinitiators within the composite resin and light-polymerization units (LPUs),[6] affecting both mechanical and functional properties.[7,8]

This study was supported by National Natural Science Foundation of China under grant #81500889.
The authors declare that they have no conflict of interests.
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Clinical Implications

Incompatibility of the emission spectra of light-polymerization units and the absorption spectra of photoinitiators in resin cements may lead to unsatisfactory immediate and delayed bond strengths. Polywave light-polymerization units are recommended because their emission spectra cover not only the absorption wavelengths of camphorquinone but also the newly developed photoinitiators.

Camphorquinone/tertiary amine (CQ/TA), which was introduced in the 1970, is a traditional and commonly used photoinitiator system in composite resins.[9,10] Light-emitting diode (LED) units have been widely used for light polymerization because of their high power intensity and low heat generation.[11] The main limitation of the CQ/TA system is the compromised color stability caused by amines.[12] Since the development of new photoinitiators such as Lucirin TPO and Ivocerin, photosensitivity and color stability have improved.[13,14] The absorption spectra of Lucirin TPO is 380 to 425 nm, whereas that of Ivocerin is 390 to 450 nm.[10] Meanwhile, traditional monowave LPUs have only 1 emission peak (usually at approximately 455 nm),[15,16] and discrepancies may exist between the emission spectra of traditional monowave LPUs and the absorption spectra of alternative photoinitiators. Therefore, polywave technology has been developed to produce a broad spectral emission that contains more than one emission peak and covers the absorption spectra of camphorquinone and alternative photoinitiators.[9,13,16-18] However, information regarding the effect of monowave and polywave LED units on the adhesion of zirconia is lacking.

The purpose of this in vitro study was to evaluate the effect of monowave and polywave LED units on the bonding effectiveness of dual-polymerizing self-adhesive resin cements to monolithic zirconia. The null hypotheses tested were that the monowave and polywave LPUs would not affect the immediate bond strengths, the long-term bonding durability, and the degree of conversion of resin cements.

MATERIAL AND METHODS

Ninety-six zirconia disks (Zenostar; Wieland), shade A3, each with a diameter of 10 mm and a thickness of 1 mm, were densely sintered according to the manufacturer's instructions. Power analysis was run by using the G-power software (G-power version 3.1; Heinrich-Heine-Universität Düsseldorf) to determine the sample size. The disks were abraded with 110-μm Al_2O_3 at 0.25 MPa for 15 seconds at 10 mm perpendicular to the surface and cleaned ultrasonically in 95% ethanol for 10 minutes.[19]

Table 1. Datasheet of resin cements tested

Resin Cement	Composition	Lot Number/ Manufacturer
RelyX U200 (U200)	Base paste: methacrylate monomers containing phosphoric acid groups, methacrylate monomers, silanated fillers, initiator components, stabilizers, rheological additives Catalyst paste: methacrylate monomers, alkaline fillers, silanated fillers, initiator components, stabilizers, pigments, rheological additives	619159/3M ESPE
SpeedCEM (SC)	Base paste: dimethacrylates, glass filler, silicon dioxide, initiators, stabilizers, pigments Catalyst paste: dimethacrylates, ytterbium trifluoride, copolymer, silicon dioxide, adhesive monomer (10-MDP), initiators, stabilizers, pigments	V08561/Ivoclar Vivadent AG

Table 2. Datasheet of light-polymerization units used

Light-Polymerization Units	Type	Overall Light Intensity (mW/cm^2)	Emission Spectrum (nm)	Spectral Peak (nm)	Manufacturer
Elipar S10 (ES)	Monowave	950	430-480	~455	3M ESPE
Bluephase Style (BS)	Polywave	970	385-515	~410; ~470	Ivoclar Vivadent AG

LPU, light-polymerization unit. Overall light intensity of 2 chosen LPUs measured by dental radiometer (Bluephase Meter II; Ivoclar Vivadent AG).

Two types of transparent, shaded, dual-polymerizing self-adhesive resin cements were selected (RelyX U200; 3M ESPE and SpeedCEM; Ivoclar Vivadent AG) (Table 1). RelyX U200 was assumed to contain camphorquinone (absorption wavelength peak at approximately 470 nm), and SpeedCEM was assumed to contain camphorquinone and alternative photoinitiators (absorption wavelength peak at approximately 400 nm). The monowave LPU (Elipar S10; 3M ESPE) and polywave LPU (Bluephase Style; Ivoclar Vivadent AG) were selected (Table 2). Four groups (n=24) containing different combinations of LPUs and resin cements were formed and correspondingly named ES-U200, BS-U200, ES-SC, and BS-SC. Randomization was achieved by generating random numbers in a software program (Microsoft Excel 2016; Microsoft Corp).

The bonding area was standardized by using Teflon tubes with 1-mm inner diameter as molds. A cylinder-shaped resin cement of diameter 1 mm and height 4 mm was formed on each disk (Fig. 1). The specimens were stored in double distilled water in darkness at 37 °C for 24 hours. Within each group, the specimens were divided into 2 subgroups (n=12): microshear bond strength (μSBS) test after 24 hours of H_2O storage (24 h) and others aged for 10 000 thermocycles (10k/TC).[20] The μSBS test was conducted with a tungsten wire at a crosshead speed of 1 mm/min in a universal testing machine (ElectroForce 3220; Bose) (Fig. 1).[21]

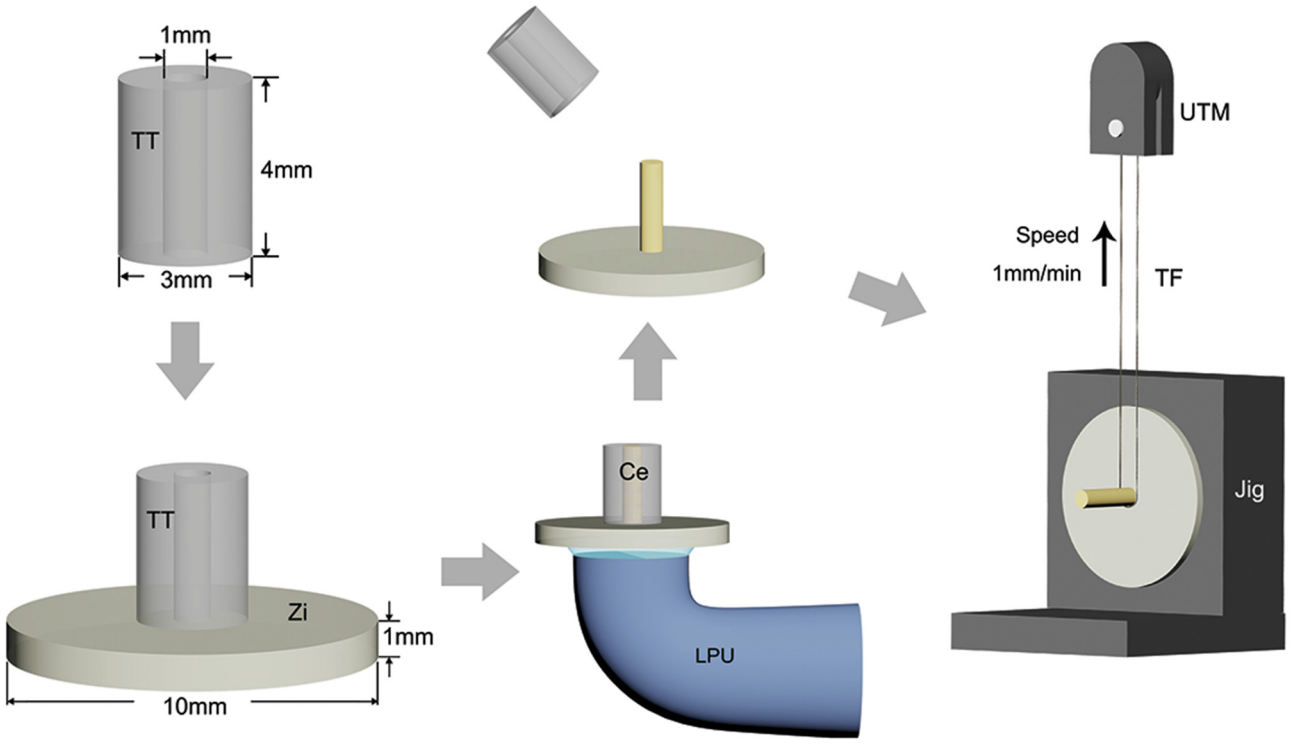

Figure 1. Schematic of μSBS specimen preparation and μSBS test. μSBS, microshear bond strength; Ce, cements; Jig, fixture to hold specimens; LPU, light-polymerization unit; TF, tungsten filament; TT, Teflon tubes; UTM, universal testing machine; Zi, zirconia.

Thermocycling was conducted by using a thermocycling machine (Automatic thermal cycling machine LXK-1; Xi'an Century Institute of Observation & Control Technologies) at a temperature between 5 °C and 55 °C and a dwell time of 60 seconds.

The specimens were examined by using a stereomicroscope (Stemi SV 11; Zeiss) at ×50 magnification. The areas where residual resin cement is attached to the zirconia surface were measured with photographs from an imaging software program (Image J; National Institutes of Health). The failure mode was classified by the proportion of residual resin on the bonding area as follows: ≤1/3 was adhesive failure and >1/3 was mixed failure.[22,23] The specimens were further observed by using a scanning electron microscope (Quanta; FEI) at 15 kV to investigate the fracture surface topography.

The degree of conversion (DC) was measured by using the Fourier transform infrared spectrometer (NICOLET 5700; Thermo Fisher Scientific) with an attenuated total reflectance accessory. The spectra were recorded with 20 scans at a resolution of 4 cm^{-1} in the 4000-400 cm^{-1} band. For each group, 6 specimens were measured.

For RelyX U200, the aliphatic C=C absorbance peak at 1637.3 cm^{-1} and the aromatic C-C reference peak at 1608.4 cm^{-1} were measured. The DC was calculated from the changes in the ratios of the absorbance peak of C=C and C-C at different times. The following equation was used:

$$(C{=}C)\% = \frac{[\text{abs(aliphatic C=C)/abs(aromatic C–C)}]\text{polymerized}}{[\text{abs(aliphatic C=C)/abs(aromatic C–C)}]\text{unpolymerized}}$$

$$DC\% = 1-(C{=}C)\%$$

For SpeedCEM, the peak height ratios of C=C of the recorded Fourier transform infrared spectra before and after polymerization were assessed for the calculation of DC because of the absence of the typical aromatic C-C absorbance peak.[24] The DC was recorded both immediately and 24 hours after light activation.[13,18]

The μSBS and DC data were analyzed with a 2-way ANOVA, followed by post hoc comparisons with the Tukey honestly significant difference test. Failure modes were analyzed by using a chi-square test, and multiple

Table 3. Two-way ANOVA

Source of Variation	df	Sum of Squares	F	P
Group	3	1241.072	31.935	<.001
Time	1	594.281	45.875	<.001
Group×time	3	717.458	18.461	<.001
Error	88	1139.972		
Total	95	3692.783		

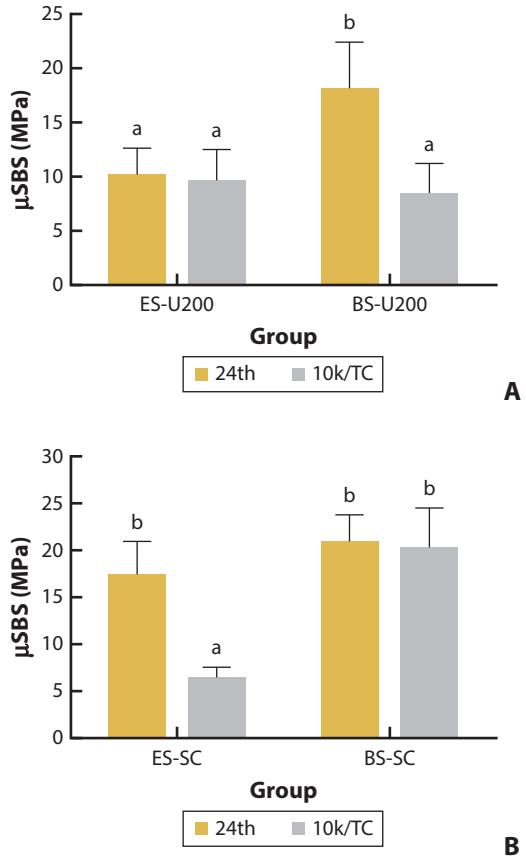

Figure 2. Microshear bond strength test result. A, Microshear bond strength test result of RelyX U200. B, Microshear bond strength test result of SpeedCEM. Same letters above bars indicate no statistically significant differences between groups (Tukey HSD test, $P>.05$). 24 h, 24 hours of storage in H_2O at 37 °C in complete darkness. 10k/TC, 24 hours of storage in H_2O at 37 °C in complete darkness, followed by 10 000 thermocycles. μSBS, microshear bond strength; BS, Bluephase Style; ES, Elipar S10; HSD, honestly significant difference; SC, SpeedCEM; U200, RelyX U200.

comparisons of the rate of adhesive failure were carried out by using the chi-square segmentation method.[25] Statistical analyses were performed by using a statistical software program (IBM SPSS Statistics, v21.0 for Mac; IBM Corp) (α=.05 for all tests).

RESULTS

The 2-way ANOVA results indicated that the factors "group" and "time" significantly influenced the μSBS values (P<.001). Likewise, the interactions between these 2 factors were significant (P<.001; Table 3). Significant differences were also observed at both the 24-hour time and the 10k/TC aging level (P<.001; Fig. 2). The bond strengths of ES-SC and BS-U200 groups significantly decreased after thermocycling (P<.001 for both groups), whereas no significant difference was found in the ES-U200 and BS-SC groups. At the 24-hour aging level, the μSBS values of the BS-U200 group were significantly higher than those of the ES-U200 group (P<.001). After aging, the μSBS values dropped in both the groups, but this change was more significant in the BS-U200 group (P<.001; Fig. 2A). For SpeedCEM, no significant difference in μSBS values at the 24-hour aging level was found in groups polymerized using the monowave (ES-SC) or polywave (BS-SC) LPUs. After thermocycling, the μSBS values differed significantly between the groups (P<.001; Fig. 2B).

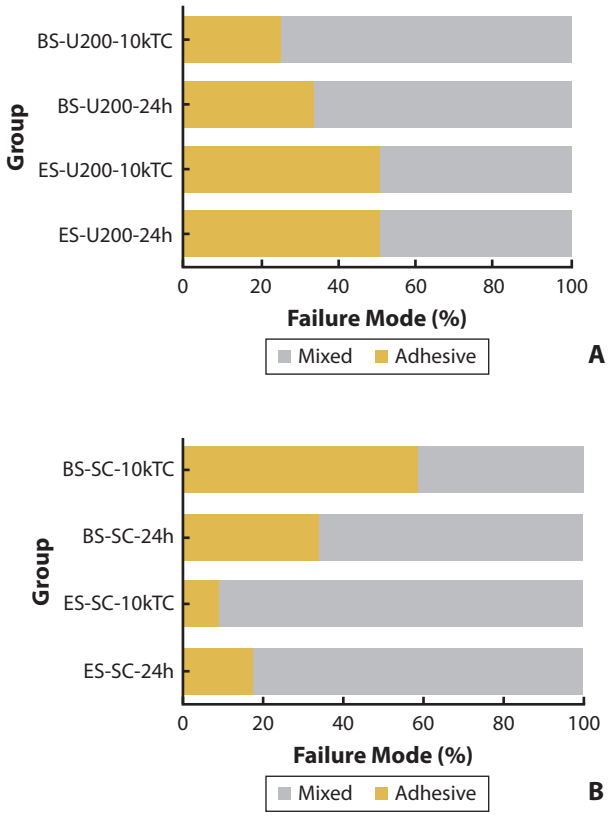

Figure 3. Failure mode analysis result. A, Failure mode analysis result of RelyX U200. B, Failure mode analysis result of SpeedCEM. Mixed, mixed failure. Adhesive, adhesive failure. 24 h, 24 hours of storage in H_2O at 37 °C in complete darkness. 10k/TC, 24 hours of storage in ddH_2O at 37 °C in complete darkness, followed by 10 000 thermocycles. BS, Bluephase Style; ES, Elipar S10; SC, SpeedCEM; U200, RelyX U200.

The Pearson chi-square test found significant differences in the rate of adhesive failure among all

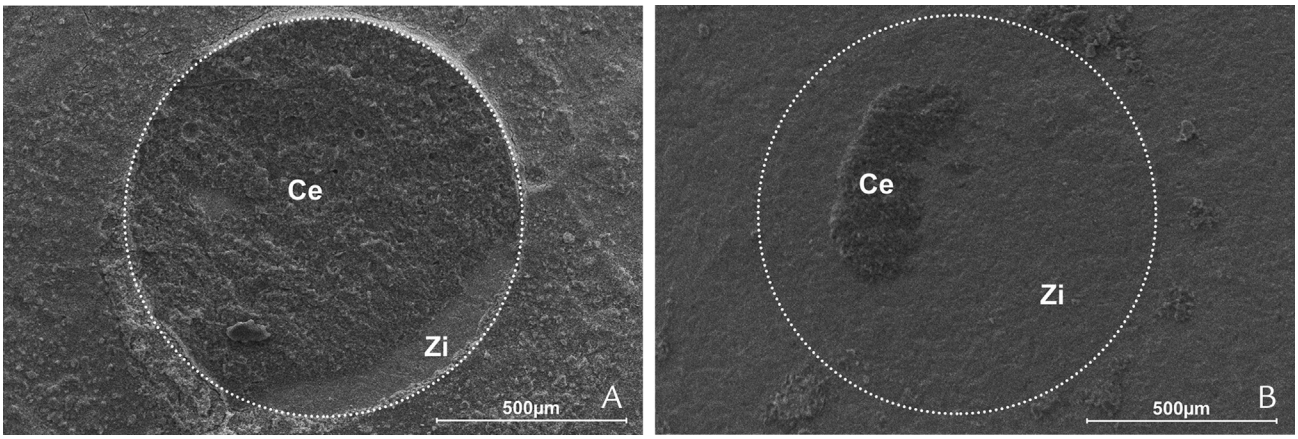

Figure 4. Representative scanning electron microscope images of specimens (original magnification ×80). A, Typical mixed failure. B, Typical adhesive failure. Ce, cements; Zi, zirconia.

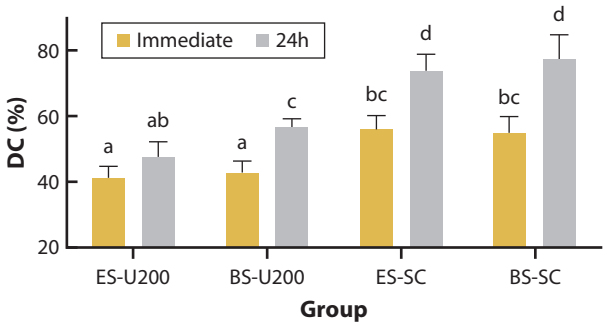

Figure 5. Degree of conversion. Same letters above bars indicate no statistically significant differences among groups (Tukey HSD test, $P>.05$). Immediate, immediate test after light polymerization. 24 h, 24 hours storage in ddH_2O at 37 °C in complete darkness. BS, Bluephase Style; DC, degree of conversion; ES, Elipar S10; HSD, honestly significant difference; SC, SpeedCEM; U200, RelyX U200.

groups (χ^2=480.000, $P<.001$; Fig. 3). Multiple comparisons showed that the differences among the rates of adhesive failure of the ES-U200, BS-U200, and BS-SC groups were nonsignificant (χ^2=0.260, $P=.612$) and that their rates of adhesive failure were significantly higher than those of the ES-SC group (χ^2=6.790, $P=.009$). No significant difference was observed between the rates of adhesive failure of the 2 subgroups (24 hours and 10k/TC) in each group. The typical surface morphology of adhesion areas under scanning electron microscope observation is shown in Figure 4.

Significant differences were observed between immediate and 24-hour DC in all groups but ES-U200 (Fig. 5). No significant difference was found in the immediate DC of the resin cement when polymerized using different LPUs ($P=.405$ for U200 and $P=.708$ for SC). The DC of the SpeedCEM was significantly higher than that of the RelyX U200 at both test times, regardless of the LPU used.

DISCUSSION

This study investigated the effect of monowave and polywave LPUs on the adhesion of resin cements to zirconia. The results of the μSBS test demonstrated that the different emitting spectra of LPUs significantly influenced the bond strength and aging resistance of self-adhesive resin cements bonded to monolithic zirconia. Significant differences were also observed in immediate and 24-hour DC results, conforming to the trend of the μSBS results. Thus, all null hypotheses were rejected.

The ES-SC and BS-U200 groups showed significant differences between 24-hour and 10k/TC bond strengths. This result can be attributed to the incompatibility between the emission spectra of the LPUs and the absorption spectra of the photoinitiators in the resin cements. The ES-U200 and BS-SC groups showed no significant difference before and after thermocycling. This result indicated that the compatibility between the emission spectra of the LPUs and the absorption spectra of the resin cements enhanced resistance against aging. The broader emitting spectrum of the polywave LPU provides additional radiant energy. Furthermore, the BS-SC group possessed relatively high bond strengths at both 24-hour and 10k/TC aging levels, indicating that the available wavelength range of the LPU matched that of the cement. Alqahtani et al[17] reported no significant difference in polymerization when the resin cement was polymerized by monowave and polywave LPUs through Empress esthetic ceramic.

The present results showed that the emission spectra of LPUs may influence the adhesion of resin cements to zirconia, an effect that may be related to the properties of zirconia materials, particularly translucency and thickness.[13,14]

The comparison of 24-hour DC values may effectively represent the effect of LPUs on polymers.[18,26,27] The light of short wavelengths emitted by the LPUs may be more efficient in activating photoinitiators.[17] The polywave LPUs emitted a broad spectrum with a shorter wavelength scale (lower than 400 nm^{-1}), which might explain the discrepancy in the μSBS values and DC results of the resin cements. SpeedCEM had a higher degree of conversion than RelyX U200 because of the coinitiators contained in SpeedCEM and thus showed more efficient conversion. In the ES-SC and BS-U200 groups, even when the chemical reaction made up for the acceptable 24-hour DC and μSBS values, the incompatibility between the available wavelengths of resin cements and LPUs accounted for the noticeable decrease of 10k/TC μSBS. The present results are consistent with those of previous studies and indicate that light activation is essential for conversion of resin materials.[6,13,26]

Light polymerization facilitates the rapid and thorough polymerization of resin cement, and its effect cannot be entirely replaced by chemical polymerization.[7,26] However, dental practitioners might overlook the importance of light polymerization while using dual-polymerizing resin cements. Checking the manufacturer information on composite resins before application can be helpful[13,18]; however, manufacturers should provide more detailed information on the recommended absorbance wavelength or type of LPUs.

The failure mode or debond pathway produced is relevant to the different failure mechanisms of resin cements or the way of load application.[22,23,28] The amount of mixed or cohesive failure in the shear bond strength test might be greater than that in the tensile bond strength test because of the relatively large bonding area and high nonuniform stress distribution at the largest load application.[23] Compared with μTBS test method, the μSBS specimens were prestressed only by mold removal before testing, an approach that prevented other potential interferences.[5,21]

In the future, the effect of LPUs of diverse emitting spectra or modes and zirconia in different degrees of translucency on bonding performance should be evaluated.

CONCLUSIONS

Within the limitations of this in vitro study, the following conclusions were drawn:

1. The emission spectra of LED units can significantly influence the bond strength, degree of conversion, and failure mode of dual-polymerizing self-adhesive resin cements bonded to zirconia at immediate and artificial aging levels.
2. The LPUs should provide light energy to match the absorption wavelengths of photoinitiators in resin cements.
3. Polywave LPUs are recommended because their emission spectra encompass the absorption wavelengths of CQ and newly developed photoinitiators, thereby matching most commercially available resin cements.

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Acknowledgments
The authors thank Dr Shikai Zhao for his assistance in preparing the manuscript, Associate Professor Hao Yu for his support in study design, and Professor Zhengzhi Wang (University of Wuhan University) for technical support with microshear bond strength test.

https://doi.org/10.1016/j.prosdent.2018.12.010