Original Article

Influence of medium-translucency monolithic zirconia thicknesses and light-curing time on the polymerization of dual-cure resin cements

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Aim: To investigate and compare the effects of different thicknesses of medium-translucency monolithic zirconia and light curing times on the polymerization of two types of dual-cured resin cement. **Methods:** A total of 200 cement specimens were prepared from TheraCem and RelyX U200 cement. The specimens were divided into 5 groups: Group I, without interposing zirconia; Group II, 0.50 mm thickness; Group III, 1.00 mm; Group IV, 1.50 mm; and Group V, 2.00 mm thickness. Each group was subdivided into (1) RelyX U200 and (2) TheraCem. Each subgroup was subdivided according to the light-curing time into (a) 20 s and (b) 40 s (n =5). The polymerization was tested using Fourier-transform infrared (FTIR) spectroscopy and a Vickers microhardness tester. The data were statistically analyzed using ANOVA, an independent sample t-test, and Tukey's test at a significance level of 0.05. **Results:** The control group had the highest values of DC and VMH, followed by 0.50, 1.00, and 1.50 mm, respectively, while the 2.00 mm group showed the lowest values. The specimens irradiated for 40 s had greater DC and VMH than those irradiated for 20 s. RelyX U200 revealed higher values for both parameters compared to TheraCem cement. **Conclusion:** The polymerization of self-adhesive cement depends on the thickness of the monolithic zirconia, the light curing time, and the composition of the cement. The cement should be irradiated for a longer period than recommended to overcome the light attenuation of zirconia. TEGDMA-based self-adhesive cement showed a higher DC and VMH than BISGMA-based cement.

Keywords: Zirconium. Resin cements. Polymerization.

Introduction

All-ceramic restorations have been used for a long time because they meet the demands of both dentists and patients, including superior aesthetics and a natural appearance. Conventional zirconia (first generation) is a partially stabilized zirconia that was introduced over fifteen years ago. It was distinguished by its opaque appearance and poor aesthetics. As a result, it served as a framework for more aesthetic veneered materials[1]. Cracking or chipping of the veneer ceramic materials was the main reason for the clinical failure of veneered zirconia restorations[2]. To overcome the issue of ceramic veneer chipping, monolithic zirconia (second generation) was introduced as a full-contour zirconia restoration for single or multiple units. Several in vitro studies on the second generation have revealed increased strength in addition to higher translucency. Monolithic zirconia is an excellent choice for all-ceramic restorations with limited occlusal space and high occlusal loads owing to its good fracture resistance, mechanical properties, and biocompatibility[3-5].

The third generation of monolithic zirconia was introduced in 2015 as a fully stabilized zirconia. The translucency of these materials was compared to glass ceramics. Recent investigations have found that the monolithic zirconia of this generation can be effectively employed for restorations with less occlusal reduction and lower occlusal strength due to the lower fracture toughness of these materials[5,6].

The clinical performance of a zirconia fixed prosthesis is determined not only by the material's strength but also by the choice of an appropriate luting agent used to form a good bond between the dental structure and the restoration[7]. The success of the luting agent depends on polymerization's efficiency, which is determined by the degree of monomer conversion (DC) and the number of free radicals generated. Inadequate polymerization compromises the hardening of the resin cement, affecting its mechanical properties. Moreover, it leads to negative consequences, including increased water absorption, microleakage, secondary caries, and postoperative sensitivity[3,8,9].

Various methods have been used to evaluate the DC. The direct method (Fourier-transform infrared spectroscopy) is commonly used for DC analysis and provides reliable results[10,11] and the indirect method (Vickers microhardness test)[12]. Several factors may influence polymerization, including ceramic thickness, translucency, type of luting agent, and light-curing time[13-15].

This in-vitro study aimed to investigate and compare the effects of different thicknesses of medium-translucency monolithic zirconia and light-curing times on the polymerization efficiency of two newly generated dual-cure, self-adhesive resin cement.

The null hypothesis was that the different thicknesses of medium-translucency monolithic zirconia and the light-curing times had no effects on the VMH and DC of dual-cure resin cement (DCRC).

Materials and Methods

Zirconia specimens' preparation

The materials and their composition tested in the current study are recorded in Table 1. The monolithic zirconia specimens were prepared from partially sintered yttrium

oxide−stabilized zirconium blocks (IPS e. Max ZirCAD MT blank disc, Ivoclar Vivadent Schaan, Liechtenstein). Cubic specimens (10×10 mm) of different thicknesses (0.50, 1.00, 1.50, and. 2.00 mm, n = 8 for each thickness) were fabricated using a CAD/CAM system (Hint-ELs, Griesheim, Germany). After that, the zirconia specimens were separated from the blank disc and carefully finished using a fine fissure diamond bur to remove any excess. The specimens were sintered using a special furnace, following the manufacturer's recommendations (Programat S1 1600, Ivoclar Vivadent, Schaan, Liechtenstein). Sintering was performed for 2.5 h at 1500 °C with heating and cooling rates of 10 °C /min, then Specimens were carefully polished with 600, 800, and 1200 grit silicon carbide polishing papers (Sailbrand, China) with a water-cooling system, cleaned for 15 min with an ultrasonic cleaner (Shenzhen Langee Ultrasonic Electric Co., China), and air dried for the 20s. A digital caliper (Bosch, Germany) was used to standardize the total thickness of the zirconia specimens (± 0.01 mm). Finally, the external specimen surfaces were glazed to remove any surface defects.

Table 1. The composition of the tested materials

Materials	Type	Manufacturer	Composition
IPS e.max ZirCAD (MT)	Monolithic zirconia	Ivoclar Vivadent Schaan, Liechtenstein	86.0 − 93.5% Zirconium oxide (ZrO2), > 6.5 % − ≤ 8.0 % Yttrium oxide (Y2O3), ≤ 5.0% Hafnium oxide (HfO2), ≤ 1.0% Aluminium oxide (Al2O3), ≤ 1.0% Other oxides.
RelyX U200	Dual-cured self-adhesive resin cement	3M ESPE; Seefeld, Germany	Base: Methacrylate monomers with the phosphoric acid group, Triethylene glycol dimethacrylate (TEGDMA), Silanated filler, Initiators, and stabilizers. Catalyst: Methacrylate monomers, Silanated fillers, Alkaline fillers, Initiators, Pigments. Filler content: 72 wt.%.
TheraCem	Dual-cured, self-adhesive resin cement	BISCO, Schaumburg, U.S.A.	Base: calcium base filler, glass filler, bisphenol glycidyl dimethacrylate (BISGMA), fluoride components, amorphous silica, and initiators Catalyst: Methacryloyloxydecyl Dihydrogen Phosphate (MDP), glass fillers. Filler content: 60-65 wt.%.

Cement specimens' preparation

Two hundred specimens were prepared from the two newly generated dual-cure self-adhesive resin cement: TheraCem (BISCO, Schaumburg, USA) and RelyX U200 (3M ESPE, Seefeld, Germany) to test DC and VMH (100 specimens for each test, n =5). Five experimental groups were formed:

Group I: Control group, cement specimens were irradiated without interposing monolithic zirconia specimens.

Group II: Cement specimens were irradiated through 0.50 mm-thick monolithic zirconia specimens.

Group III: Cement specimens were irradiated through 1.00 mm-thick monolithic zirconia specimens.

Group IV: Cement specimens were irradiated through 1.50 mm-thick monolithic zirconia specimens.

Group V: Cement specimens were irradiated through 2.00 mm-thick monolithic zirconia specimens.

Each main group was randomly subdivided according to the type of resin cement tested: (1) RelyX U200 cement and (2) TheraCem cement. Each subgroup was further divided according to the light-curing time as follows: (a) 20 s and (b) 40 s.

Degree of Conversion (DC)

One hundred specimens were made from two types of dual-cured resin cement using a Teflon mold with standard dimensions (2 mm inner diameter and 1 mm thickness, n = 5) to test the degree of conversion[8]. The molds were positioned on glass slides which were covered with transparent strips to prevent material bonding. Each cement was mixed following the manufacturer's recommendations and packed into molds. One more transparent strip and a glass slide were placed at the top and pressed lightly to remove excess material.

After removing the glass slide, one of each thickness of the zirconia specimens was positioned on top, and then the tip of the curing device was carefully placed on the upper surface of the zirconia specimens to permit light to pass through the zirconia toward the cement (Fig.1A). The light-curing device (LED, Guilin woodpecker, Medical instrument Co., Ltd., Germany) with a light intensity output of 1600 Mw/cm^2 was
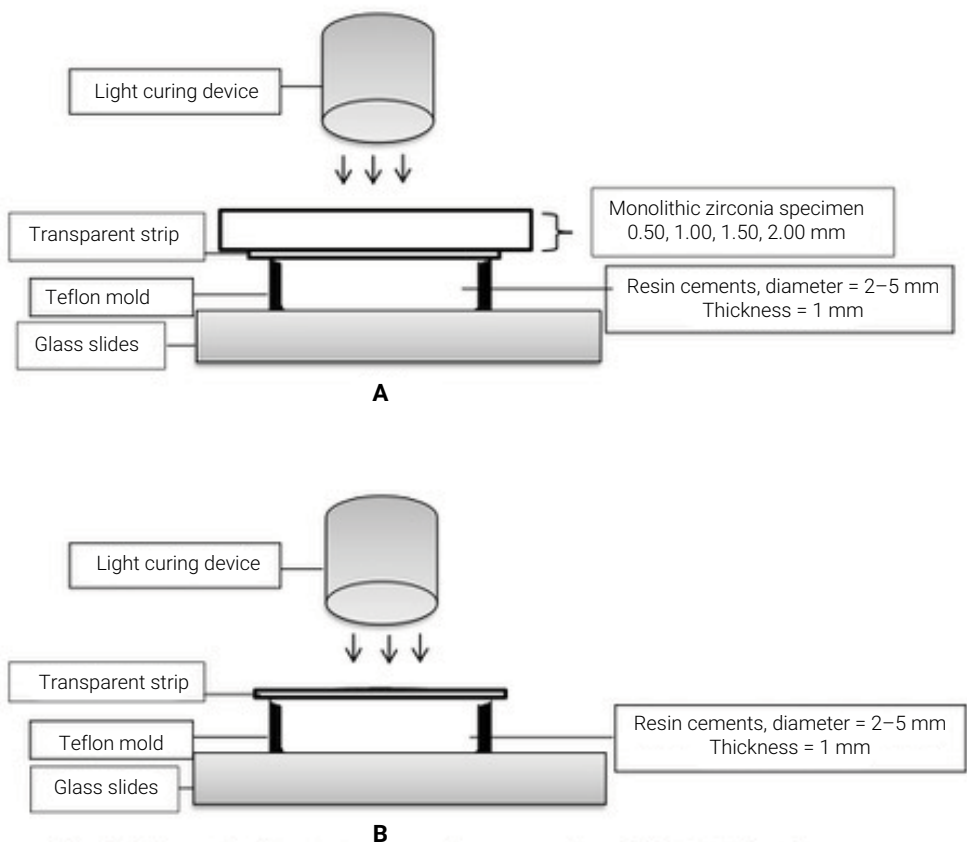

Figure 1. Schematic diagram showing the preparation of the tested resin cement specimens (A, with interposing of different thicknesses of monolithic zirconia. B, direct activation without interposing of monolithic zirconia).

used to irradiate cement specimens with an exposure time of the 20s or 40s. Light intensity was checked using a radiometer (Bluephase Meter II, Ivoclar Vivadent). The control group was prepared by direct activation without interposing zirconia specimens (Fig.1B). The specimens were left for 5 min to self-cure and carefully polished with 600 and 800-grit silicon carbide polishing paper. Finally, the specimens were kept at 37°C for 24 h in completely dark containers to prevent further exposure to light before testing.

Fourier-Transform infrared (FTIR) spectroscopy (BRUKER ATR-DIAMOND FT-IR, Germany) was used to evaluate the DC. The spectrophotometer system operated in the range of 500–3500 cm^{-1} with a resolution of 4 cm^{-1}. A small amount of uncured resin cement was evaluated using a spectrophotometer, and the resulting spectrum was considered an unpolymerized reference. DC was calculated using a method that evaluated the changes in the ratio of aliphatic C=C (1638 cm^{-1}) to the aromatic C-C bond (1608 cm^{-1}) for the uncured and cured conditions based on the following equation[8].

$$DC\% = 1 - \left\{ \frac{1638 \text{ cm}^{-1} / 1608 \text{cm}^{-1} \text{ cured}}{1638 \text{ cm}^{-1} / 1608 \text{cm}^{-1} \text{ uncured}} \right\} \times 100$$

Vickers microhardness test (VMH)

One hundred specimens were made from two types of cement using a mold with standard dimensions (5 mm diameter and 1 mm thickness, n =5) to test the VMH[9]. The specimens were prepared using the same procedure mentioned above. The polymerized specimens were evaluated using a Vickers microhardness tester (OTTO WOLPER-WERKE, Germany). A load of 0.5 kg was applied with a dwell time of 30 s. Four indentations were made on the surface of the specimen. The four values were averaged to obtain the Vickers hardness number (VHN) of each specimen. The VHN was calculated in kg/mm^2 using this equation:

$$VHN = 1.8544 * L / D^2$$

Where L = applied load recorded in (Kg), D = mean diagonal length recorded in (mm).

Statistical Analysis

SPSS (version 25, IBM Corp., Armonk, USA) was used to analyze the data statistically. An ANOVA was applied to determine the significant differences between all different thicknesses of zirconia. Tukey's post hoc test was used to compare the significant groups. To investigate the differences between the two tested groups of resin cement and light-curing times, an independent samples t-test was used. $P \leq 0.05$ was considered statistically significant.

Results

The mean values and standard deviation (SD) of the DC and VMH tests for RelyX U200 and TheraCem resin cement for different monolithic zirconia thicknesses in the 20s and 40s are recorded in Tables 2 and 3, respectively. A one-way ANOVA for both resin

cement revealed significant changes in the mean values of the DC and VMH tests ($P \leq 0.05$) between different monolithic zirconia thicknesses at both light-curing times.

Table 2. Mean, standard deviation (SD) of the degree of conversion for different thicknesses of monolithic zirconia and light curing time for both resin cements.

Resin Cement	Zirconia Thickness (mm)	Time	
		20 s Mean ± SD	40 s Mean ± SD
RelyX U200	0.00 mm	68.8 a ± 0.45	73.4 A ± 0.44
	0.50 mm	65.5 b ± 0.85	70.6 B ± 1.24
	1.00 mm	62.4 c ± 1.41	67.9 C ± 2.18
	1.50 mm	58.6 d ± 0.50	63.1 D ± 1.59
	2.00 mm	54.0 e ± 2.27	58.6 E ± 0.82
TheraCem	0.00 mm	65.0 a ± 1.98	68.7 A ± 0.70
	0.50 mm	62.1 b ± 0.97	66.3 B ± 1.61
	1.00 mm	59.1 c ± 1.57	62.7 C ± 2.37
	1.50 mm	53.6 d ± 0.80	58.2 D ± 0.97
	2.00 mm	50.1 e ± 2.41	55.7 E ± 1.06

Different letters are significantly different depending on Tukey's test. Number of specimens = 5

Table 3. Mean, standard deviation (SD) of the Vickers microhardness test (kg/mm^2) for different thicknesses of monolithic zirconia and light curing-time for both resin cements.

Resin Cement	Zirconia Thickness (mm)	Time	
		20 s Mean ± SD	40 s Mean ± SD
RelyX U200	0.00 mm	70.7 a ± 1.05	75.0 A ± 1.34
	0.50 mm	65.2 b ± 1.08	69.7 B ± 0.90
	1.00 mm	62.1 c ± 1.31	65.5 C ± 1.32
	1.50 mm	59.1 d ± 1.10	64.8 D ± 1.08
	2.00 mm	53.5 e ± 1.34	60.7 E ± 2.87
TheraCem	0.00 mm	52.1 a ± 1.22	57.3 A ± 0.83
	0.50 mm	48.6 b ± 1.55	54.3 B ±1.21
	1.00 mm	44.7 c ±1.14	51.2 C ±1.06
	1.50 mm	41.7 d ± 1.13	46.0 D ± 0.99
	2.00 mm	38.4 e ± 0.99	42.3 E ± 1.12

Different letters are significantly different depending on Tukey's test. Number of specimens = 5

Tukey's post hoc test exhibited that the specimens irradiated without zirconia interpose (control group) had the highest mean values of DC and VMH, followed by the specimens irradiated with 0.50, 1.00, and 1.50-mm thick zirconia, respec-

tively while specimens interposing with 2.00 mm had the lowest values compared to all groups, as shown in Tables 2 and 3. Based on these results, as the thickness of monolithic zirconia restoration increased, the VMH and DC values of resin cement decreased.

An independent *t*-test exhibited that there were significant changes between the two light-curing times for all groups. The specimens irradiated for the 40s had greater DC and VMH mean values than those irradiated for the 20s as shown in Tables 4 and 5. In addition, there were significant changes between RelyX U200 and TheraCem resin cement, as shown in Tables 6 and 7. RelyX U200 revealed significantly higher mean values for both parameters compared to TheraCem resin cement in all groups.

Table 4. Independent Samples *t*-test of the degree of conversion for the different thicknesses of monolithic zirconia between two light-curing times.

Resin Cement	Zirconia Thickness (mm)	t-test	df	SE	P-value
RelyX U200	0.00 mm	16.016	8	0.28	0.000**
	0.50 mm	7.575	8	0.67	0.000**
	1.00 mm	4.721	8	1.16	0.002**
	1.50 mm	5.897	8	0.74	0.002**
	2.00 mm	4.285	8	1.08	0.008**
TheraCem	0.00 mm	4.025	8	0.94	0.010**
	0.50 mm	4.996	8	0.84	0.002**
	1.00 mm	2.834	8	1.27	0.025*
	1.50 mm	8.193	8	0.56	0.000**
	2.00 mm	4.766	8	1.17	0.004**

SE = Standard Error, df = degree of freedom, * Normal Significant at $P \leq 0.05$, ** Highly Significant at $P \leq 0.01$

Table 5. Independent Samples *t*-test of the Vickers microhardness test for the different thicknesses of monolithic zirconia between two light-curing times.

Resin Cement	Zirconia Thickness (mm)	t-test	Df	SE	P-value
RelyX U200	0.00 mm	5.684	8	0.76	0.001**
	0.50 mm	7.188	8	0.63	0.000**
	1.00 mm	4.097	8	0.83	0.003**
	1.50 mm	8.217	8	0.69	0.000**
	2.00 mm	5.054	8	1.42	0.003**
TheraCem	0.00 mm	7.919	8	0.66	0.000**
	0.50 mm	6.425	8	0.88	0.000**
	1.00 mm	9.315	8	0.69	0.000**
	1.50 mm	6.279	8	0.67	0000**
	2.00 mm	5.859	8	0.67	0.001**

SE = Standard Error, df = degree of freedom, ** Highly Significant at $P \leq 0.01$

Table 6. Independent Samples *t*-test of degree of conversion for different thicknesses of monolithic zirconia between two resin cements.

Time	Zirconia Thickness (mm)	t-test	Df	SE	P-value
	0.00 mm	4.19	8	0.91	0.011**
	0.50 mm	5.83	8	0.57	0.000**
20 s	1.00 mm	3.47	8	0.95	0.008**
	1.50 mm	11.87	8	0.42	0.000**
	2.00 mm	2.64	8	1.48	0.029*
	0.00 mm	12.39	8	0.37	0.000**
	0.50 mm	4.71	8	0.91	0.002**
40 s	1.00 mm	3.59	8	1.44	0.007**
	1.50 mm	5.73	8	0.83	0.000**
	2.00 mm	4.85	8	0.60	0.002**

SE = Standard Error, df = degree of freedom, * Normal Significant at $P \leq 0.05$, ** Highly Significant at $P \leq 0.01$

Table 7. Independent Samples *t*-test of Vickers microhardness test for different thicknesses of monolithic zirconia between two resin cement.

Time	Zirconia Thickness (mm)	t-test	Df	SE	P-value
	0.00 mm	25.76	8	0.72	0.000**
	0.50 mm	19.62	8	0.84	0.000**
20 s	1.00 mm	22.24	8	0.78	0.000**
	1.50 mm	24.48	8	0.71	0.000**
	2.00 mm	20.31	8	0.74	0.000**
	0.00 mm	25.03	8	0.70	0.000**
	0.50 mm	22.75	8	0.67	0.000**
40 s	1.00 mm	18.80	8	0.75	0.000**
	1.50 mm	28.61	8	0.65	0.000**
	2.00 mm	13.34	8	1.37	0.000**

SE = Standard Error, df = degree of freedom, ** Highly Significant at $P \leq 0.01$

Discussion

The basic factors for the success of indirect ceramic restorations are the stable and strong bond between the luting agent and ceramic restoration and between the luting agent and dental tissues. The bond strength of resin-based luting agents is determined by satisfactory polymerization of the resin matrix[16]. Optimal polymerization of the cement is important for obtaining appropriate mechanical and physical properties and is considered a fundamental target for the success of ceramic-based restorations[13,17]. Clinically, the minimum thickness for monolithic zirconia restorations is 0.50 mm. However, to fabricate monolithic zirconia anatomical posterior restorations, the thickness can reach 2.00 mm, which may be critical to the polymerization

of the cement[18]. To simulate a clinical condition of monolithic zirconia cementation, the DCRC was irradiated from the top of the zirconia specimens with different thicknesses (0.50-2.00 mm).

The statistical analysis revealed that the mean DC and VMH values decreased significantly with increasing the thickness of the monolithic zirconia compared to the control group that was directly exposed to light (Tables 2 and 3). Based on this result, the first null hypothesis was rejected. The current study agrees with many previous studies[19-21]. A possible explanation for this result is that increasing the thickness of zirconia causes the light to be attenuated by the zirconia material. The combination of absorption, scattering, and reflection explains the attenuation of the light passing through the restoration, which negatively affects the polymerization reaction of the cement[10,15,22-25].

It has been reported that approximately 25% of the light energy reaching the cement is available at 1 mm thickness, and the polymerization efficiency of the dual-polymerized resin cement decreases by approximately 70% with the increasing thickness of indirect restoration[26]. Bragança et al.[27] 2020 showed that a 0.50 mm thickness of ceramic reduced the light reaching the resin cement by about 50%. On the other hand, some studies concluded that the DC of the resin cement irradiated under 0.50 and 1.00 mm thick ceramic specimens was similar to that of the control groups, but there was a decrease in the DC at thicknesses of 1.50 mm and above[28,29]. This disagreement may be due to differences in the methodology and materials used.

Regarding the light curing time, the specimens irradiated for 40 s showed higher values for both parameters than those irradiated for 20 s. Hence, the second null hypothesis was rejected. It appears that extending the exposure time is necessary to enhance the polymerization efficiency of the resin-based cement. These results agree with several authors who explained that the curing time used by dental clinicians is too short and may not be sufficient to achieve optimum polymerization. They suggested extending the curing time further than recommended by the manufacturer to compensate for the deficiency of light intensity and achieve proper chemical polymerization[9,22,27-31].

Other factors may influence the polymerization of DCRC, including the type of luting agent, light irradiance, and post-curing times[13,14,22]. According to Sulaiman et al.[22] 2015, the amount of light-curing irradiance decreased as the thickness of the zirconia increased. Therefore, a high light irradiance device is preferred to increase the quantity of light reaching the cement, particularly for those under thick monolithic zirconia restorations. Moreover, light irradiance decreases with aging. Thus, clinicians should frequently check the conditions of light-curing devices to avoid inadequate polymerization[9].

Several authors confirmed that DC and VHN values significantly improved within the first 24 hours. This is clarified by the presence of unpolymerized monomers with the potential mobility to permit low-rate reactions. The "dark polymerization" of unpolymerized monomers may continue after the cessation of photoactivation[32]. "Dark polymerization" can adequately compensate for the deficiency in initial polymerization and reach the ultimate polymerization to obtain the best clinical performance[15].

RelyX U200 and TheraCem resin cement exhibited different behaviors when irradiated under the same conditions. This may be related to the variances in the cement compositions, including the amount and type of monomer structure, inorganic filler content, concentration, and quality of chemical and photoinitiators[33-35]. The results obtained from (FTIR) spectroscopy showed that the DC of TheraCem and RelyX U200 ranged from 50% to 74% as shown in Table 2. It has been proved that resins have a degree of monomer conversion (50-75%), which is clinically acceptable[36,37].

In all groups, RelyX U200 had significantly higher mean DC values than TheraCem resin cement; this could be attributed to the fact that RelyX U200 is a triethylene glycol dimethacrylate (TEGDMA)-based resin cement[38]. TEGDMA is a low-molecular-weight and high-mobility monomer that allows the formation of more cross-linking between polymeric chains, resulting in a high degree of polymerization[37,39]. Long-chain TEGDMA molecules are believed to improve the polymerization reaction, which continues for 24h after light activation. The polymerization efficacy increases with an increase in TEGDMA percentage owing to its reactivity and greater mobility[40], while TheraCem is Bisphenol glycidyl dimethacrylate (Bis-GMA)-based resin cement[41] which is a high-molecular-weight monomer that enhances the mechanical properties of the resins but decreases the DC[8,42]. Because of the higher degree of polymerization compared to Bis-GMA-based cement, it is recommended to use a self-adhesive resin cement with a high TEGDMA content[43]. Furthermore, Thera-Cem contains a methacryloyloxydecyl dihydrogen phosphate (MDP) functional group, which has negative effects on the polymerization reaction because it chemically interacts with the tertiary amine (co-initiator) and camphorquinone[44]. The high percentage of inorganic fillers incorporated into RelyX U200 may increase the degree of monomer conversion compared to TheraCem due to the lower amount of residual monomers.

RelyX U200 had higher mean VHN values than TheraCem in all groups. This result could be clarified by the fact that the RelyX U200 contains more inorganic filler (72 by weight) than TheraCem (60-65 by weight)[41]. The results of the current study are in agreement with other authors who stated that the surface hardness of resin cement depends on the proportion, type, and distribution of the inorganic filler content and that increasing the proportion of inorganic fillers enhances the surface hardness of the cement[14,15].

The limitation of this in vitro study was that the test conditions did not fully mimic the oral environment. Additional evaluations will be required to test the polymerization efficiency of DCRC under monolithic zirconia in vivo. Further research will also be needed to evaluate the impact of zirconia thickness on cement's optical characteristics.

Within the limits of the current study, it can be concluded that the polymerization efficiency of dual-cured self-adhesive cement depends on the thickness of the medium-transparency monolithic zirconia, light curing time, the monomer type, and the concentration of filler contents. Increasing the thickness of monolithic zirconia from 0.50 to 2.00 mm exhibited a negative effect on the polymerization efficiency of the underlying cement. The dual-cured resin cement should be clinically irradiated for a longer period than recommended by the manufacturer to overcome the light atten-

uation of zirconia material. TEGDMA-based self-adhesive cement showed statistically higher DC and VMH values than BISGMA-based cement. The DC values for both types of cement ranged within the clinically accepted limit of 50–75%. Thus, both RelyX U200 and TheraCem can be proposed as luting agents for monolithic zirconia restorations.

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Conflict of interest

None to declare.

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