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Effect of Optical Properties of Lithium Disilicate Glass Ceramics and Light-Curing Protocols on the Curing Performance of Resin Cement

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Abstract: This study aimed to investigate the effects of optical properties of lithium disilicate glass ceramics and the light-curing protocols (LCP) on the curing performance of light-cured resin cement. Lithium disilicate glass-ceramics with different optical properties were sectioned to produce ceramic specimens of 0.8 mm thickness. Irradiance through the ceramic specimens was measured by a radiometer. Light transmittance of ceramics was assessed using a UV/Vis spectrophotometer. The light-cured resin cement was injected into a Teflon mold and ceramics with different optical properties were placed on it, cured under different LCPs, and the degree of conversion (DC) and Vickers microhardness of the resin cement were separately measured by Micro-ATR/FTIR spectrometry and the microhardness tester. The shade ($p < 0.001$) and transparency ($p < 0.001$) of ceramics affect the irradiance of the light-curing unit. The transparency ($p < 0.001$) of the ceramic and light-curing protocols ($p < 0.001$) affect the DC and microhardness of resin cements. When the thickness of the ceramic is 0.8 mm, the light transmittance of the ceramic and the curing performance of the resin cement increase with the increase of the transparency of the ceramic. An appropriate increase in irradiance and exposure time can optimize the curing performance of resin cement. These factors should be taken into account by the clinician when designing the bonding solution for porcelain veneers.

Keywords: lithium disilicate; optical properties; light-curing protocols; irradiance; transmittance; degree of conversion; microhardness

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1. Introduction

With the development of dental treatment techniques, minimally invasive treatment is gradually recognized and widely applied clinically. Porcelain veneers are one of the key minimally invasive treatment techniques which are widely used by clinicians for their excellent aesthetic properties, minimally invasive dental preparation, good tissue adaptability et al. [1,2]. Studies have shown that the clinical success rate of porcelain veneers is 94.4% at 5 years, 93.5% at 10 years and 82.93% at 20 years [3]. However, a certain amount of failure still exists clinically. Common complications of porcelain veneers include debonding, fractured or chipped veneers, secondary caries, marginal discoloration and endodontic problems [4–7]. Among them, the problem of debonding is prominent.

Various aesthetic materials have emerged due to clinical aesthetic demands. The clinicians perform shade matching before ceramic materials selection to match the color of the ceramic with the natural teeth. Transparency and shade are two key properties in material selection. The interplay between shade, transparency and light can mimic natural teeth to a great extent.

Lithium disilicate glass ceramics have good biocompatibility, mechanical properties, and chemical stability and their excellent aesthetic properties can mimic natural teeth to great extent [8–10]. This kind of material is the most commonly used to produce porcelain veneers by either press techniques in the dental lab or chairside CAD/CAM [11].

Depending on the curing properties, resin cements can be divided into light-cured resin cements, chemical-cured resin cements and dual-cured resin cements [12]. Light-cured resin cements are commonly recommended for the bonding of porcelain veneers for better color stability and longer clinical working time [13].

The adhesive mechanism between the restoration and the tooth structure involves the bonding of the hard tooth tissue to the resin cement and the bonding of the resin cement to the restoration [14]. Incomplete curing of resin cement as a bonding medium between the restoration and the tooth may lead to unstable restoration color, reduced bond strength, microleakage, increased risk of caries, increased post-operative sensitivity and toxicity from residual monomers [15–18] which may finally lead to the failure of the treatment. The proper curing of the resin cement therefore plays an important role in the clinical success and long-term longevity of porcelain veneers [19].

Studies show that the material types, thickness, transparency and shade of the ceramic materials can have an effect on the curing light reaching the resin cement [20–22]. Higher light intensities or longer exposure times improve the curing performance of resin cements [23,24]. However, most of these experiments are studies on the degree of polymerization of resin cement on a single factor. When the clinician is bonding the porcelain veneer, the design of the curing plan needs to consider a variety of influencing factors. At present, there is a lack of comprehensive research on the degree of polymerization of resin cements by a variety of influencing factors. Therefore, this experiment is devoted to studying the influence of various influencing factors on the curing performance of resin cement, and provides certain reference suggestions for the bonding of porcelain veneers.

This study aims to compare the efficiency of different curing protocols in terms of the DC and Vickers microhardness of light-cured resin cement through lithium disilicate glass ceramics of different optical properties. The null hypotheses are: (i) the shade and transparency of lithium disilicate glass ceramics do not affect the irradiance of the light-curing unit, the transmittance of ceramic and the DC and microhardness of the resin cement; (ii) differences in light-curing protocols do not affect the DC and Vickers microhardness of the resin cement.

2. Materials and Methods

2.1. Specimens Preparation

2.1.1. Preparation of Glass Ceramic Specimens

Lithium disilicate glass ceramic blocks (IPS e.max CAD Ivoclar Vivadent, Schaan, Liechtenstein) were divided into four study groups according to different transparency and shade, namely HT A2; LT A2; HT BL2; LT BL2. Lithium disilicate specimens were fabricated by slicing CAD/CAM ceramic blocks by diamond discs in a slow-speed precision cutter (SMJ-200, Shenyang Kejing Automation Equipment Co., Ltd., Shenyang, China) into 14 mm × 12 mm slices of approximately 0.8 mm thickness. The ceramic slices were crystalized in a ceramic furnace (Programat P310, Ivoclar Vivadent, Schaan, Liechtenstein), in accordance with the manufacturer's instructions. The sintering procedure was as follows: the first stage of heating, after drying at 403 °C for 6 min, the temperature was increased to 820 °C at a rate of 90 °C/min and held for 10 s; in the second stage of heating, the temperature was increased to 840 °C at a rate of 30 °C/min and held for 7 min. Vacuuming in the first stage started at 550 °C and ended at 820 °C, and vacuuming in the second stage started at 820 and ended at 840 °C. Then, the sample was naturally cooled to room temperature at 700 °C for later use. After crystallization, each surface was ground with 400-, 600-, 800-, and 1200-grit silicon carbide sandpaper on a Precision Lapping/Polishing Machine (UNIPOL-802, Shenyang Kejing Automation Equipment Co., Ltd., Shenyang, China), polished by progressively finer diamond polishing paste down to 0.5 µm, and then ultrasonically cleaned in 95% ethanol for 10 min to eliminate any contamination, and dried. Specimens' thicknesses were assessed with a 0.001 mm accuracy digital caliper (Mitutoyo, Japan) in five different areas. Five percent hydrofluoric acid (Ivoclar Vivadent, Schaan, Liechtenstein) was coated on the surface of the ceramic specimen, acid-etched for 20 s, rinsed for 15 s, dried, and then coated with a silane coupling agent (Monobond N, Ivoclar Vivadent, Schaan, Liechtenstein), and the surface was washed and dried after standing for 60 s and set aside for use.

2.1.2. Preparation of Resin Cement Specimens

The resin cement specimens were prepared using a cylindrical Teflon mold with a diameter of 5 mm and a thickness of 500 μm. The glass slide was placed on a black table and a Teflon mold was placed on the top. Light-cured resin cement (Variolink N Transparent Base, Schaan, Liechtenstein) was injected into the Teflon mold. The Mylar strip was then placed on the mold to insulate it from oxygen. IPS e.max CAD ceramic slices of different optical properties were placed on a Mylar strip in the experimental groups. In the light-curing unit (Bluephase G2; Ivoclar Vivadent, Schaan, Liechtenstein), depending on the irradiance and exposure time, there were four light-curing protocols (LCP1: high irradiance mode for 40 s; LCP2: low irradiance mode for 40 s; LCP3: high irradiance mode for 20 s; LCP4: low irradiance mode for 20 s). The resin cement was cured separately according to the above protocols. The light-curing unit was held close to the resin cement specimen and illuminates perpendicular to the surface of the glass slide. These experiments were carried out in a dark room to avoid the influence of visible light.

2.2. Testing Methods

2.2.1. Determination of the Irradiance

An LED light-curing unit was used in this study. In this experiment, two different irradiance modes of Bluephase G2 were selected: low irradiance mode and high irradiance mode. Use a radiometer (Bluephase Meter II, Ivoclar Vivadent, Schaan, Liechtenstein) to measure the actual irradiance, Mylar strips (control group) and ceramic slices were placed with different optical properties to measure the irradiance of each ceramic slice on the aperture of the irradiator. Each specimen was measured 5 times.

2.2.2. Measurement of Light Transmittance

Porcelain veneer samples with different optical properties were measured using a dual-beam UV/Vis spectrophotometer (UV-8000, Shanghai Metash Instruments Co., Ltd., Shanghai, China). The spectral range was set as 380–780 nm and the scanning interval was 1 nm; there were 4 groups in total, and each group was measured 3 times.

2.2.3. Degree of Conversion Measurement

Each group of cured specimens was examined using a Fourier Transform Infrared Spectrometer Attenuated Total Reflection Attachment (FTIR-850-ATR, Tianjin Gangdong Technology Co., Ltd., Tianjin, China), with the light wavelength recorded in horizontal coordinates and the absorbance in vertical coordinates. The absorbance spectra of uncured resin materials and cured resin specimens were acquired by 16 scans over a 4000–650 cm^{-1} range with a resolution of 4 cm^{-1}. DC was calculated by estimating the changes in peak height ratio of the absorbance intensities of aliphatic C=C peak at 1637 cm^{-1} and that of an internal standard peak of aromatic C=C at 1608 cm^{-1} during polymerization, in relation to the uncured material. DC% for each specimen was calculated using the following equation:

$$\mathrm{DC\%} = 1 - \left\{ \frac{\frac{(\mathrm{Abr1637\ cm^{-1}})}{(\mathrm{Abr1608\ cm^{-1}})}\mathrm{cured}}{\frac{(\mathrm{Abr1637\ cm^{-1}})}{(\mathrm{Abr1608\ cm^{-1}})}\mathrm{uncured}} \right\} \times 100$$

2.2.4. Vickers Microhardness Measurement

The resin cement specimens were positioned on the microhardness tester (HV-1000A, Taiyuan Laihua Testing Instrument Co., Ltd., Taiyuan, China) and 3 measurements were made in three different positions at the top surface of each 5 mm diameter resin cement sample by applying a load of 100 gf for 10 s, for a total of 3 Vickers indents per sample, 15 indents per group and total 240 indents (n = 16).

2.3. Statistical Analysis

All statistical analyses were done using SPSS statistical software (v22.0, IBM, Armonk, NY, USA). Statistical analysis was performed using three factors (ceramic shade, transparency and light-curing protocols). The normal distribution of the irradiance, transmittance, DC, and Vickers microhardness data were investigated using the Shapiro–Wilk test, which demonstrated that the data formed a normal distribution. One-way ANOVA was used to analyze the effect of ceramic transparency and shade on irradiance. Two-way ANOVA was used to analyze the transmittance of ceramics with different transparency and shade. The equality of the dispersion was evaluated using Levene's test, and the differences between groups were analyzed using three-way ANOVA, and Tukey's HSD test was used as a post hoc test ($\alpha = 0.05$ for all analyses).

3. Results

3.1. Irradiance of the Light-Curing Unit

The mean and standard deviation of the irradiance are presented in Table 1. One-way ANOVA showed that the transparency ($p < 0.001$) and shade ($p < 0.001$) of lithium disilicate glass ceramics significantly affect the irradiance. No matter in the low irradiance mode or the high irradiance mode, the irradiance of the curing light will be significantly attenuated after the ceramic specimens of different shades and transparency are blocked. The irradiance obtained under high transparency (HT) ceramic specimens is better than that obtained under low transparency (LT). The irradiance obtained under the BL2 ceramic specimens is better than that obtained under A2.

Table 1. Irradiance reaching the radiometer when ceramics with different opacity properties are interposed between light emitted by light-emitting diodes and the radiometer (mean ± SD).

Experimental Groups		**Low Irradiance Mode (mW/cm²)**		**High Irradiance Mode (mW/cm²)**	
Control	Control	700 ± 10.00^{Aa1}	Attenuation (%)	1258 ± 8.37^{Ba1}	Attenuation (%)
A2	HT	348 ± 8.37^{Ab2}	50.29	630 ± 7.07^{Bb2}	49.92
A2	LT	308 ± 8.37^{Ac2}	56.00	468 ± 8.37^{Bc2}	62.80
BL2	HT	408 ± 8.37^{Ab3}	41.71	726 ± 11.40^{Bb3}	42.29
BL2	LT	330 ± 7.07^{Ac3}	52.86	588 ± 8.37^{Bc3}	53.26

When different capital letters indicate that the transparency and shade of the ceramic specimens are the same, the difference between the groups with different irradiance patterns is statistically significant; when different small letters indicate that the shade and irradiance pattern of the ceramic specimens are the same, there are statistically significant differences between different ceramic specimen's transparency groups. Different numbers indicate that when the transparency and irradiance pattern of the ceramic specimens are the same, there are statistically significant differences between different ceramic specimen's shade groups; $p < 0.05$.

3.2. Light Transmittance

The transmittance of ceramics with different transparency and shade is shown in Figure 1. The results of two-way ANOVA showed that the transparency of ceramic specimens affected the transmittance of ceramics ($p < 0.05$). Ceramic transmittance increases with increasing transparency. Shade did not affect the transmittance of ceramic specimens ($p > 0.05$), and there was no interaction between the two factors.

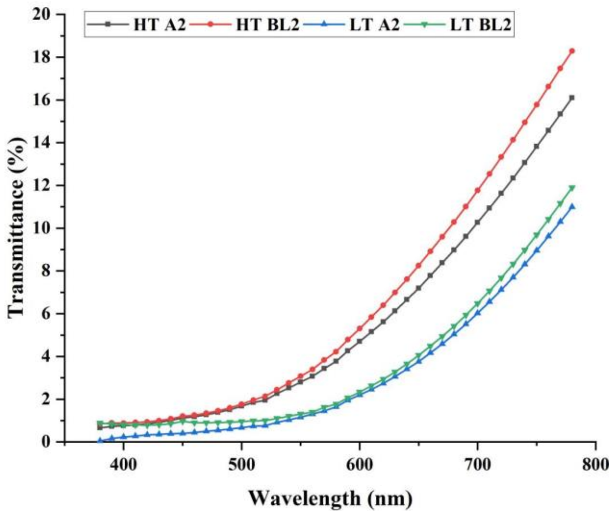

Figure 1. Transmittance of lithium disilicate glass-ceramics with different shades and transparency at 0.8 mm thickness.

3.3. Degree of Conversion

The mean and standard deviation of the DC for light-cured resin cement activated through different optical properties of lithium disilicate glass-ceramics and four light-curing protocols were reported in Figure 2. Three-way ANOVA showed that the transparency ($p < 0.001$) and light-curing protocols ($p < 0.001$) all had significant effects on the DC of the light-cured resin cement. The shade of ceramic did not affect the DC of the light-cured resin cement ($p > 0.05$). Furthermore, the results show that there was no interaction among the three factors of ceramic shade, transparency and light-curing protocols. The results of the Turkey HSD test are displayed in Figure 2.

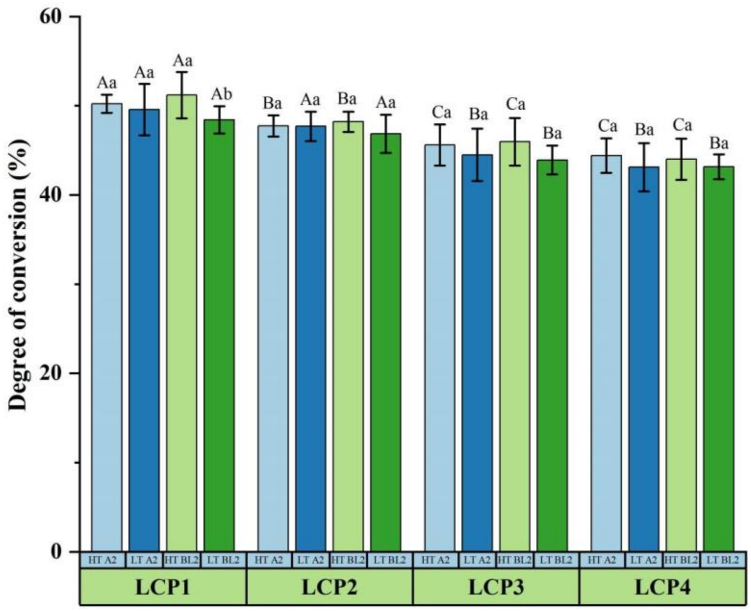

Figure 2. Degree of conversion observed for the different light-curing protocols and different ceramic optical properties. Different capital letters indicate statistically significant differences between groups for different light-curing protocols; different lowercase letters indicate statistically significant differences between different transparency groups of ceramic specimens; $p < 0.05$.

3.4. Vickers Microhardness

The mean and standard deviation of Vickers microhardness measured from the light-cured resin cement using four different protocols and four optical properties as shown in

Figure 3. Three-way ANOVA showed that the transparency ($p < 0.001$) and light-curing protocols ($p < 0.001$) all had significant effects on the DC of the light-cured resin cement. The shade of ceramic did not affect the microhardness of the light-cured resin cement ($p > 0.05$). Furthermore, the results show that there was no interaction among the three factors of ceramic shade, transparency and light-curing protocol. The results of the Turkey HSD test are displayed in Figure 3.

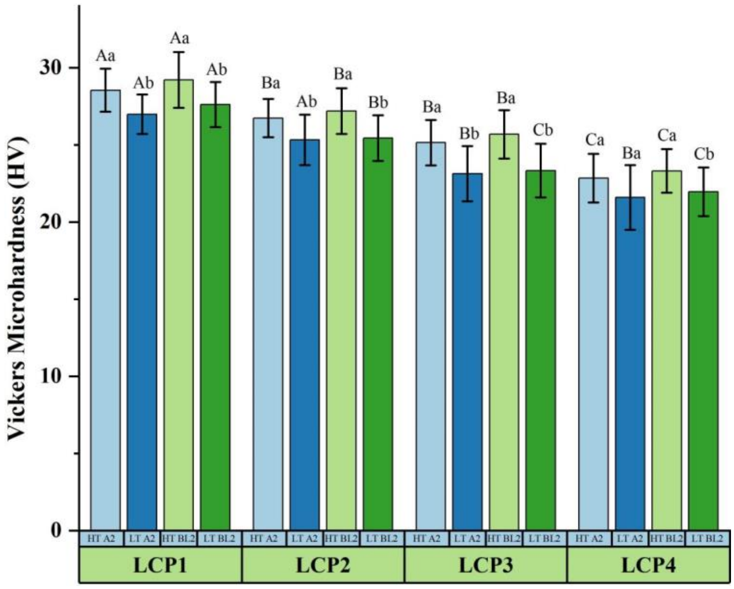

Figure 3. Vickers microhardness observed for the different light-curing protocols and different ceramic optical properties. Different capital letters indicate statistically significant differences between groups for different light-curing protocols; different lowercase letters indicate statistically significant differences between different transparency groups of ceramic specimens; $p < 0.05$.

4. Discussion

This study evaluated the effect of optical properties of lithium disilicate glass ceramics and light-curing protocols on the DC and Vickers microhardness of the light-cured resin cement. According to the results of this study, the first null hypothesis was partially rejected. The transparency of lithium disilicate glass ceramic affects the irradiance of the light-curing unit, the ceramic transmittance, the DC and the microhardness of the resin cement. The second null hypothesis was partly rejected. Different light-curing protocols affect the DC and microhardness of the resin cement.

In the process of making porcelain veneers, the thickness of general porcelain veneers is usually 0.8−1.0 mm. In order to imitate the thickness of ordinary porcelain veneers in clinical practice, the thickness of the ceramic specimens in this experiment was 0.8 mm. In order to mimic the optical properties of natural teeth, clinicians usually choose different transparency and shade ceramic materials according to the patient's abutment. Furthermore, HT A2, LT A2, HT BL2, and LT BL2, as representatives of the popular conventional shade and beautiful white shade in the production of clinical porcelain veneers, were also included in the experimental design.

The irradiance varied greatly under the different curing modes for the Bluephase G2 light-curing unit. Regardless of the variation in the optical properties of lithium disilicate glass ceramics, all specimens showed statistically significantly lower light transmission compared to the direct light activation group (control group); the highest irradiance was measured beneath HT BL2 ceramic specimens, and the lowest irradiance was measured beneath LT A2 ceramic specimens. The ceramic becomes an obstacle to the light reaching the resin cement, and glass ceramic interposition leads to an attenuation of the irradiance of the curing light, the irradiance attenuation through all ceramic specimens was around 40−65%, a result that is consistent with previous studies [25,26]. Researchers generally consider irradiance above 400 mW/cm^2 to be sufficient. In this experiment, in the low irradiance mode, the irradiance of ceramic specimens with a thickness of 0.8 mm and optical properties of HT A2, HT BL2, and LT A2 are all lower than 400 mW/cm^2. This result reminds us that we should pay attention to the influence of ceramic optical properties on irradiance. For the bonding of ceramic veneers, a light-curing unit with slightly higher irradiance should be appropriately selected.

Direct light transmittance is one of the methods to evaluate the translucency and opacity of aesthetic all-ceramic materials [27]. In this study, we found that the shade did not affect the light transmittance. This may be related to the fact that the difference between the selected shades is not significant. When a light beam passes through a material, it gradually loses intensity, a process often referred to as attenuation. Attenuation is caused by light interacting with matter in two fundamental ways: scattering and absorption [28]. Due to the insignificant difference between the selected shades, the scattering and absorption of light passing through the ceramic specimen are not sufficient to change the light transmittance.

The DC is usually used to directly evaluate the degree of polymerization of resin cement which is significantly related to the mechanical properties [29], volume shrinkage [30] and other material characteristics [31]. The degree of polymerization of a resin has a significant linear relationship with its hardness [32]. Therefore, the degree of polymerization of the resin is often indirectly evaluated by measuring the microhardness of the resin after polymerization.

Regarding the effect of lithium disilicate glass ceramic optical properties on the DC and Vickers microhardness of the resin cement, the ceramic optical properties had a significant effect on the DC of the resin cement. At present, most of the studies on the DC and microhardness of the resin cement on the optical properties of ceramics are only researched on the single factor of transparency or shade, and few studies are on the optical properties (transparency and shade) as a single factor as a whole. Previous studies have shown that transparency [33] or shade [15,21] can affect the DC and microhardness of the resin cement. Furthermore, DC or microhardness increases as the transparency of the ceramic decreases [32] or the shade saturation decreases [21,34]. In this experiment, the shade did not affect the DC and microhardness of resin cement, which may be attributed to the following reasons. According to Strang et al. [35], ceramic shade does not significantly affect the amount of absorbed light for ceramic samples with thicknesses less than 1.5 mm. In this experiment, the thickness of lithium disilicate glass ceramics is 0.8 mm, less than 1.5 mm. Therefore, it can be explained why the ceramic shade does not affect the polymerization performance of the resin cement. On the other hand, the microstructure of ceramic crystals, including crystal volume, refractive index, particle size, filler and matrix composition and the relative amount, nature, shape and particle size distribution of the crystalline phases affect the optical properties of the ceramic [36,37]. Studies have shown that lithium disilicate glass ceramics have a higher crystal content compared to leucite-based ceramics and feldspathic ceramics. The crystal content of leucite ceramics is approximately 35%, the crystal content of feldspar ceramics is approximately 30%, and the crystal content of lithium disilicate glass ceramics is approximately 65% to 70% [37]. The higher the crystalline content, the more the crystals will block the light and the more serious the irradiance attenuation will be [38]. Therefore, it is presumed that the shade difference is not significant and the high crystal content of lithium disilicate glass ceramics is less likely to have an effect on the polymerization properties of the resin cement through the ceramic specimens. In addition, the shade did not affect the light transmittance and it can also be understood that the polymerization performance of the resin cement was not affected by the shade of ceramic.

Adequate irradiance or total energy ensures adequate conversion of the resin monomer, which is a prerequisite for the desired polymerization properties of the resin. In this study, the light-curing protocol significantly influenced the DC and microhardness of the resin cement (Figures 2 and 3). For this experiment, we designed four light-curing protocols, ranked according to the total energy (total energy = irradiance × exposure time):

LCP1 > LCP2 > LCP3 > LCP4. The DC and microhardness of resin cement under these four light-curing protocols also conformed to this law; the curing performance was positively correlated with irradiance and exposure time, which is similar to previous research [24,39]. Although the curing performance generally showed this pattern, in some groups, the comparison between groups showed that the difference was not significant. This may be due to the fact that Variolink N transparent base is a light-cured resin cement, which contains dimethacrylate. In dimethacrylate-based resins, the trapping of free radicals is related, their polymerization forms a highly crosslinked network, and free radical capture becomes the main method of termination [40,41]. The irradiance after penetrating the ceramic specimen is decreased, so that the number of captured free radicals is reduced, leading to a delay in diffusion limitation to higher conversion and prolonging the life of free radical conversion, further leading to a compromise in the degree of polymerization [26].

For the clinical success of light-cured resin cements, adequate polymerization is always necessary. The higher DC and microhardness for resin material provide increased mechanical properties and bond strength. The current research on irradiance or total energy on the curing performance of resin cements has shown that higher irradiance or total energy results in higher DC and microhardness [42], but too high irradiance or too high total energy may not always lead to higher polymerization performance of resin cements [26,43]. High irradiance can compensate for the attenuation of light as it passes through the restoration, but too high a level of irradiance can lead to a decrease in the hardness of the resin and promote polymerization shrinkage so that microleakage occurs at the interface between the nest hole and the resin, which cannot be closely fitted [44]. Due to the rapid formation of a highly cross-linked polymeric mesh on the resin surface at too high an irradiance, the area of light passing through is reduced, resulting in a low frequency of cross-linking of the polymeric chains, which leads to a reduction in hardness [45]. However, if the irradiance level of the light-curing unit is too low, the use of extended exposure times alone will not ensure that the composite resin can be fully cured [46]. Therefore, either too high or too low an irradiance is detrimental to the polymerization reaction. This reminds clinicians to fully consider the above factors, choose a curing light with appropriate irradiance and design an appropriate lighting protocol for the bonding of porcelain materials.

The limitation of this study is that the type and thickness of ceramics may affect the optical properties of ceramics. This research only experimented on ceramics with different optical properties under one type of ceramics. For other types of ceramics, further research is needed. The polymerization of a light-cured luting agent is a very complicated process. In addition to the light-curing protocols, intrinsic factors such as the filler content, monomer composition, viscosity and photo-initiator systems of the light-cured luting agent and extrinsic factors such as light guide tip positioning, irradiation mode, light-curing units and emission spectrum will affect its polymerization efficiency. Different types or brands of light-cured luting agents shall be also investigated in future research.

5. Conclusions

Within the limitations of this in vitro study, we conclude that:

(1) When the thickness of lithium disilicate glass ceramics is 0.8 mm, the transparency has an influence on the irradiance, ceramic transmittance and the polymerization performance of the light-cured resin cement, and increases with the transparency of lithium disilicate glass-ceramics. Ceramics with high transparency have higher transmittance, less attenuation of irradiance and easier to obtain excellent polymerization properties. The shade of ceramic did not affect the ceramic transmittance and the curing performance of resin cement.

(2) Light-curing protocols affect the polymerization performance of the resin cement. An appropriate increase in irradiance and exposure time can improve curing performance.

When clinicians design curing protocols during porcelain veneer treatment, all these factors should be considered.

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