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Research Article

Polymerization Efficiency of a Dual-Cured Resin Cement through Zirconia with Three Different Cusp Inclinations

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Assessing the polymerization efficiency of a dual-cured resin cement through zirconia with three different cusp inclinations is the aim of this study. Seventy-two resin cement (Multilink Speed) specimens were light-cured through the zirconia with three different cusp inclinations (30°, 20°, and 0°) for 20 s or 40 s ($n = 12$). For each group, the Vickers hardness (VH) measurement was performed on half of the specimens, whereas the degree of conversion (DC) was tested for the other half with a Fourier transform infrared spectrometer. The recorded data were analyzed with a two-way ANOVA. Varied VH (32.3 ± 3.2–40.3 ± 4.8) and DC ($50.5\% \pm 3.0\%$–$59.6\% \pm 2.4\%$) values were obtained from different groups. The cusp inclinations significantly affect the VH ($p = 0.001$) and DC ($p < 0.001$) of the resin cement, and the curing time exhibited a significant effect on the DC ($p = 0.014$) of resin cement but not on the VH ($p = 0.167$). In conclusion, the cusp inclination of zirconia significantly affects the polymerization efficiency of dual-cured resin cement.

1. Introduction

Because of low solubility, high flexural strength, excellent physics and chemical properties, easy handling and favorable aesthetics [1, 2], resin cement has become the most commonly used luting agent in dentistry [3]. According to its curing mode, resin cement can be classified into three types: light-cured, self-cured (chemical-cured), and dual-cured. Light-cured resin cement allows enough working time for clinicians before polymerizing it with light-curing unit, but it is difficult to cure the resin cement in the areas that are far from the light source. Self-cured (chemical-cured) resin cement can be cured without light source, but having limited working time and technique sensitivity. Dual-cured resin cement was developed as an attempt to combine the advantages of light-cured and self-cured resin cement [4]. Once the dual-cured resin cement being mixed, it can be polymerized with/without light exposure. The chemical-polymerizing mechanism feature of dual-cured resin cement ensures adequate polymerization of resin cement in the areas that are not easily accessible for light [4, 5]. However, by itself, the self-polymerizing mechanism of dual-cured resin cement is not only slower but also less effective than when using light activation as a supplement [3]. Lee et al. [6] evaluated the polymerization of six dual-cured resin cements and concluded that the curing speed using the light-cured was 15 to 322 times faster than that using the chemical-cured. Therefore, an adequate light-cured resin cement is critical for the dual-cured resin cement [7].

As a popular material in rehabilitation dentistry, zirconia is usually bonded with dual-cured resin cement [8, 9]. When the light penetrates through zirconia, light energy would be compromised [10–13], which affects the polymerization and

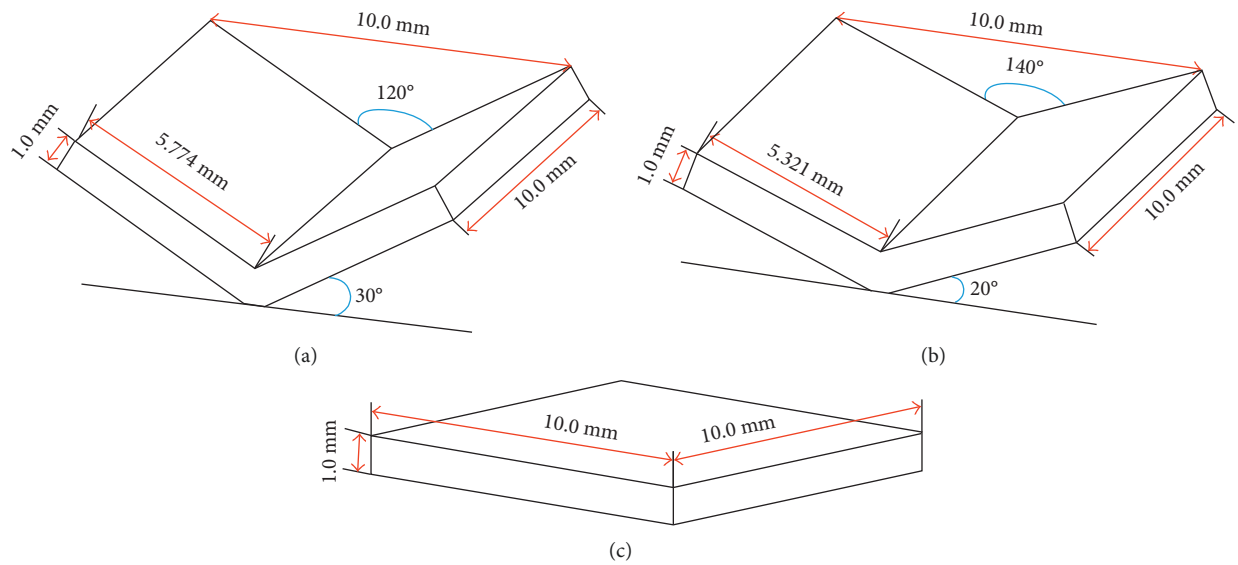


Figure 1: Schematic diagram of the monolithic zirconia specimen design. The length and width of monolithic zirconia were 10.0 mm, and the thickness was 1.0 mm. The monolithic zirconia specimen consisted of two planes: the angles between the plane and horizontal were (a) 30°, simulating 30° cusp inclination and (b) 20°, simulating 20° cusp inclination. (c) The monolithic zirconia specimen was a flat plane, simulating 0° cusp inclination.

clinical performance of resin cement [14]. However, most of the present studies investigating polymerization of resin cement through zirconia employed flat zirconia specimens [15, 16]. In reality, the surfaces of artificial zirconia-based restorations are not flat but with tooth cusps and other anatomical structures. The anatomical structures may affect the distance between the light source and the luting agent and affect the polymerization of light-cured resin cement eventually. Cusp inclinations are the most important structures on the surface of artificial restoration. Therefore, the purpose of this *in vitro* study was to evaluate the effect of cusp inclination of zirconia on the polymerization efficiency of a dual-cured resin cement. The null hypotheses were as follows: (1) the cusp inclination of zirconia would not affect the surface microhardness of the dual-cured resin cement and (2) the cusp inclination of zirconia would not affect the degree of conversion of the dual-cured resin cement.

2. Materials and Methods

2.1. Monolithic Zirconia Design. Three stereolithography files were drawn with a three-dimensional computer-aided design (CAD) software (Solidworks, Dassault System), simulating three different cusp inclinations of 30°, 20°, and 0°, as shown in Figure 1.

2.2. Monolithic Zirconia Preparation. The designed stereolithography files were imported into the ZENOTEC CAD software program (Wieland), and 72 monolithic zirconia (the tested groups are shown in Table 1, $n = 12$ for each group, 6 for VH test and 6 for DC test) were milled from zirconia blocks (Wieland) in Vita shade A2 color and sintered in accordance with the manufacturer's instructions.

Table 1: Tested groups of the study.

Group	Cusp inclination	Curing time
A1	30°	20 s
A2	30°	40 s
B1	20°	20 s
B2	20°	40 s
C1	0°	20 s
C2	0°	40 s

$n = 12$ for each group, 6 of the specimen for VH test and 6 for DC test.

Table 2: Characteristics of the monolithic zirconia tested.

Surface roughness	39.3 nm (sq)*
Flexural strength	≥900 MPa
Chemical composition	Zirconium oxide ($ZrO_2 + Y_2O_3 + HfO_2$) > 99% Yttrium oxide (Y_2O_3) : 4.5% ~ 6.0%; Hafnium oxide (HfO_2) : ≤ 5% Aluminum oxide + other oxides ≤ 1%

*Tested by authors and other information is provided by the manufacturer.

The characteristics of monolithic zirconia are shown in Table 2.

2.3. Specimen Preparation. A commercial dual-cured resin cement (Multilink Speed, Ivoclar Vivadent) was used for present study. The dual-cured resin cement was mixed following the manufacturer's instructions, placed into a customized rubber mold, and then covered with a Mylar strip (0.05 mm in thickness, Microdont). The monolithic zirconia was put above the Mylar strip. Finally, the dual-cured resin cement was light-cured for 20 s or 40 s by a light-curing unit (Elipar S10, 3M-ESPE) with an intensity of

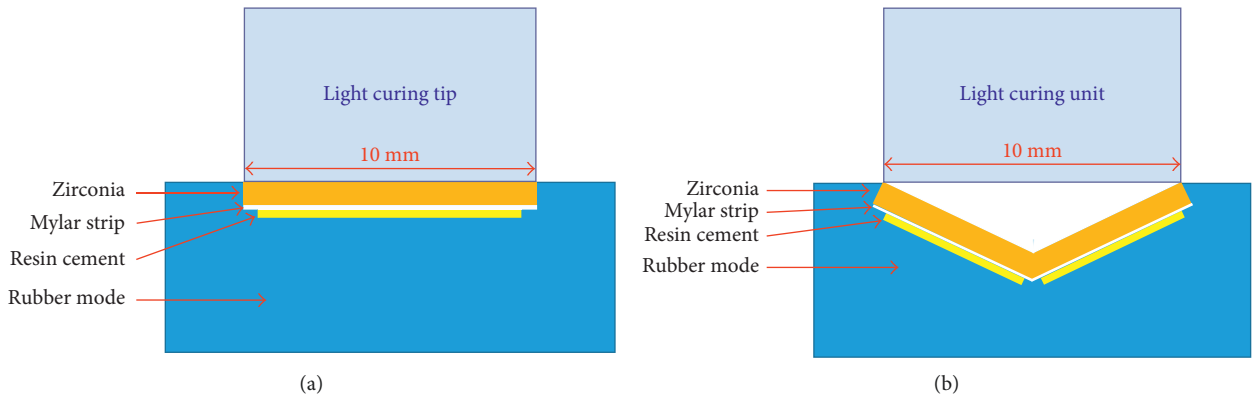


Figure 2: The light polymerization procedure of the resin cement under zirconia of (a) 0° cusp inclination and (b) 20° or 30° cusp inclination.

1200 mW/cm^2 from the zirconia upper surface. During the polymerization procedure, the light-curing unit tip was kept in close contact with the zirconia border (Figure 2). The intensity of the light-curing unit was verified with a radiometer (Bluephase, Ivoclar Vivadent) after five resin cement specimens were light-cured. After polymerization, the dimensions of the resin cement specimens were identified with a digital caliper (Peakmeter, Shenzhen Xin Huayi Instrument Co., Ltd.). The length of the resin cement specimen was 8.0 mm, and the width of resin cement specimen was 4.7 mm (30° cusp inclination groups), 4.3 mm (20° cusp inclination groups), or 8.0 mm (0° cusp inclination groups). The thickness of the resin cement specimens was 0.5 mm. Then, the resin cement specimens were stored at room temperature in a light-proof box for 24 h to avoid further exposure to light [17].

2.4. Surface Microhardness Measurement. The microhardness of the resin cement specimens' surfaces that are close to the monolithic zirconia were tested with a digital Vickers hardness tester (HXD-1000TM/LCD) with 250-gram force for 15 s [18]. Every resin cement specimen was tested at three randomized locations, and relevant values were averaged as a single Vickers hardness (VH) for each resin cement specimen [19].

2.5. Degree of Conversion (DC) Measurement. Sixty milligrams of bromatum kalium powders was pressed by a tablet machine (Specac 2T) for 15 s, and the tablet was tested by a Fourier transform infrared (FTIR) spectrometer (Thermo Scientific Nicolet iS50 FTIR) as the test background. Then, a small amount of unpolymerized resin cement was mixed with bromatum kalium powders at a ratio of 1 : 150, and sixty mg of the mixed powder was pressed by a tablet machine and tested by FTIR. The resin cement specimens were then smashed into powder and mixed with bromatum kalium powders at a ratio of 1 : 150. Sixty mg of the mixed powder was pressed into a tablet and tested by FTIR. The settings of the FTIR were as follows: 4000–650 cm^{-1} range, 4 cm^{-1} resolution, and 32 scans per spectrum. The spectra were recorded.

The absorption peaks of the aromatic double bonds were recorded at 1590 cm^{-1} (Abs 1590), and the peaks of the aliphatic double bonds were recorded at 1638 cm^{-1} (Abs 1638) [20, 21].

The DC was calculated by the following equation [22–24]:

$$\mathrm{DC} = \left(1 - \frac{\text{Absa } 1638 \times \text{Absb } 1590}{\text{Absb } 1638 \times \text{Absa } 1590}\right) \times 100\%, \qquad (1)$$

where Absa 1638 and Absa 1590 are the areas of the absorption peaks of the polymerized dual-cured resin cement and Absb 1638 and Absb 1590 are the areas of the absorption peaks of the unpolymerized dual-cured resin cement.

Table 3: The mean and standard deviation of VH and DC values for the tested groups.

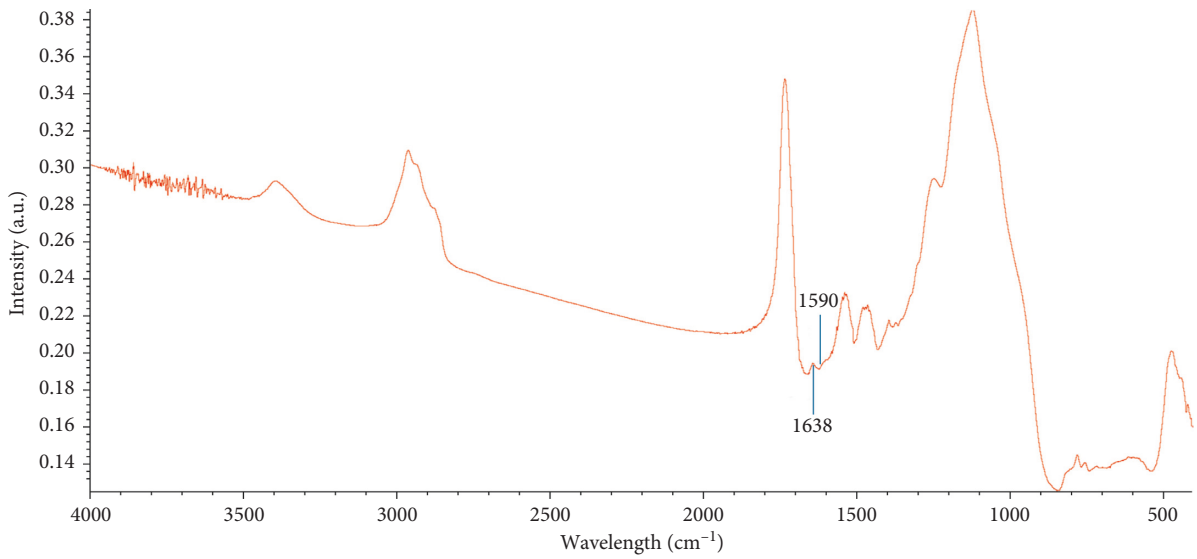
Group	Cusp inclination	Curing time	VH	DC (%)
A1	30°	20 s	32.3 ± 3.2^c	50.5 ± 3.0^b
A2	30°	40 s	33.3 ± 3.5^c	53.0 ± 3.2^b
B1	20°	20 s	33.6 ± 3.7^c	52.2 ± 3.3^b
B2	20°	40 s	36.0 ± 4.4^b	55.4 ± 2.2^a
C1	0°	20 s	40.3 ± 4.8^a	59.6 ± 2.4^c
C2	0°	40 s	33.6 ± 3.7^c	52.2 ± 3.3^b

Different superscript letters in columns indicate the statistical difference ($p < 0.05$).

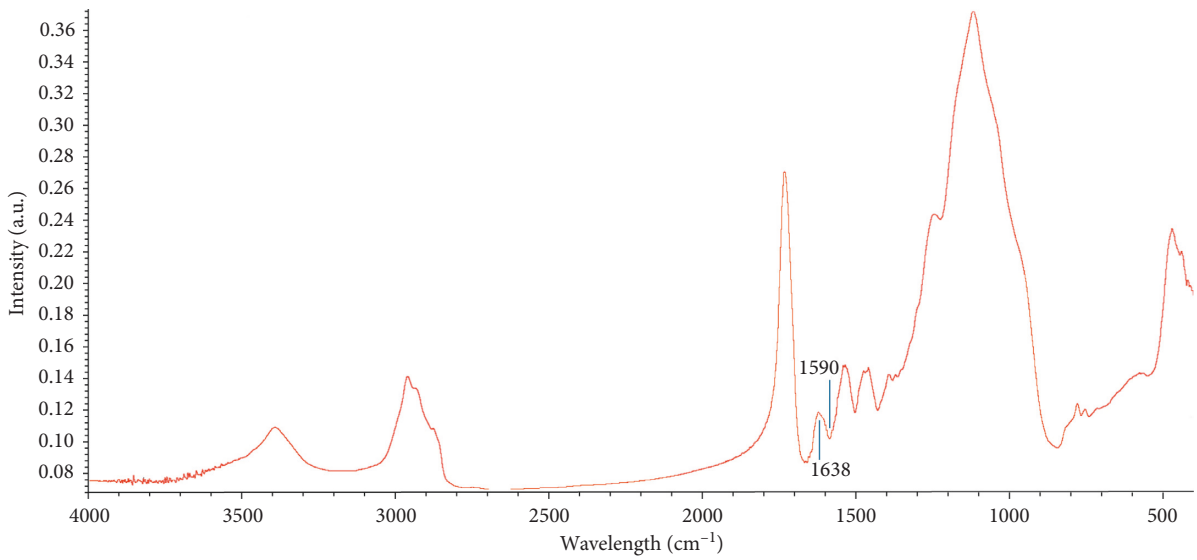
2.6. Statistical Analysis. VH and DC values were statistically analyzed using a software package (SPSS 20.0, IBM) at a significance level of 0.05. The Shapiro–Wilk test was applied to confirm the normal distribution of the data before the ANOVA. Two-way ANOVA was employed to determine the factors which affect the VH and DC.

3. Results and Discussion

The mean and standard deviation VH and DC values of all tested groups are shown in Table 3. The representative FTIR spectra are shown in Figure 3. Two-way ANOVA reveals that cusp inclination significantly affected the VH ($p = 0.001$) and DC ($p < 0.001$) of the resin cement (Tables 4 and 5). The curing time did not significantly affected the VH ($p = 0.167$) but significantly affected the DC ($p = 0.014$) of the resin cement. No significant interaction was found between cusp



(a)



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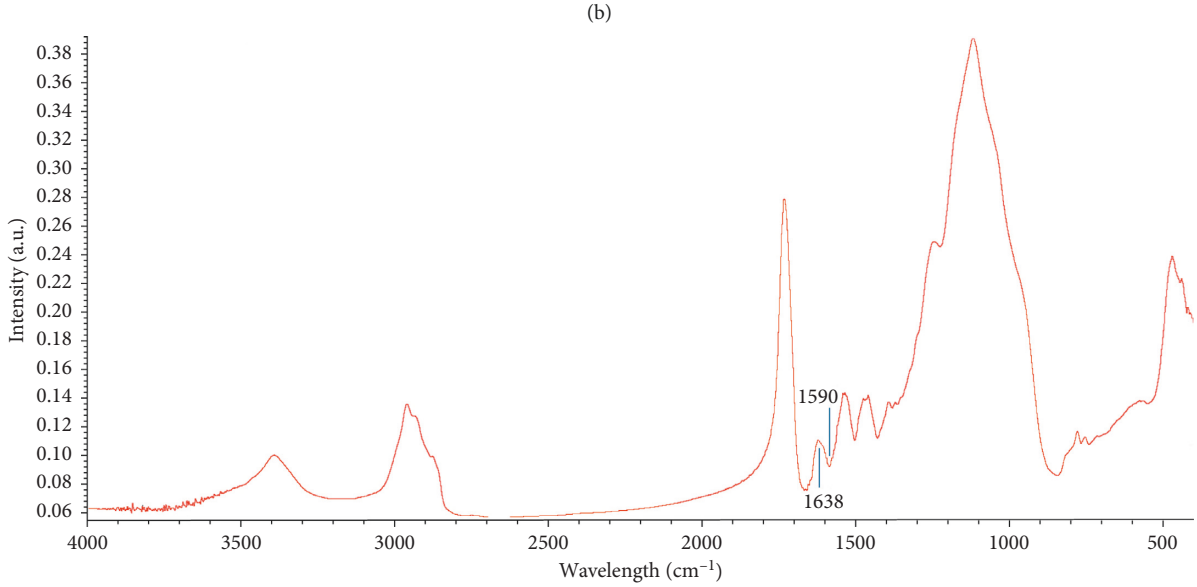
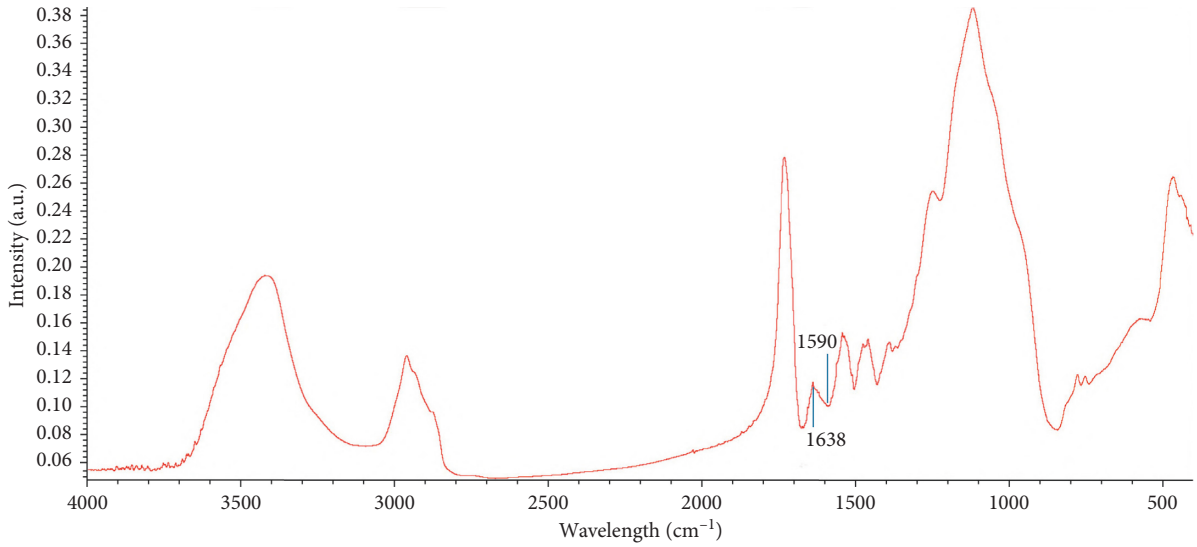


(c)

Figure 3: Continued.



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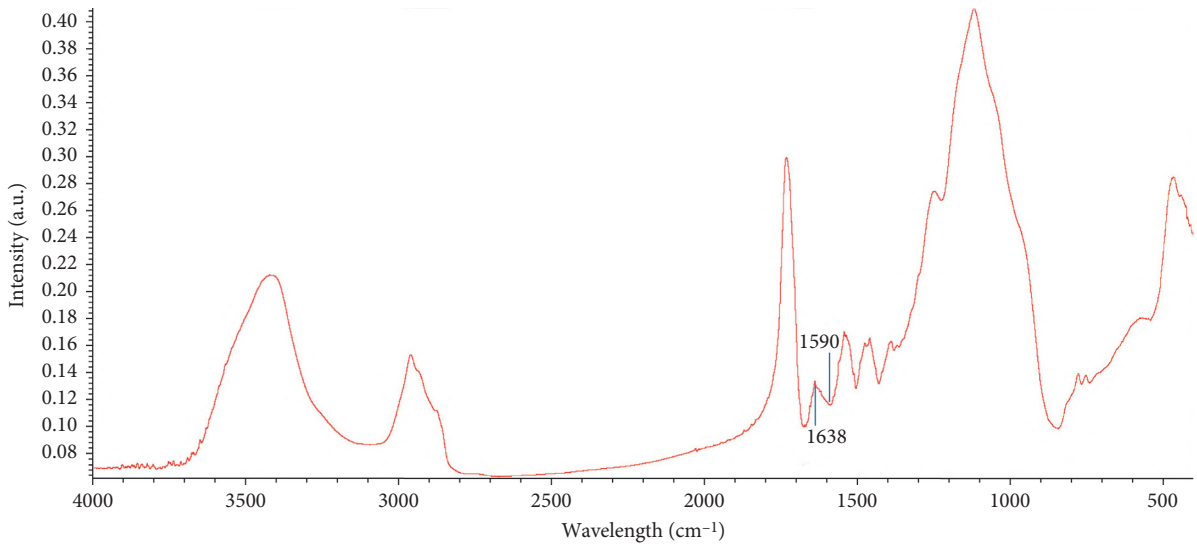

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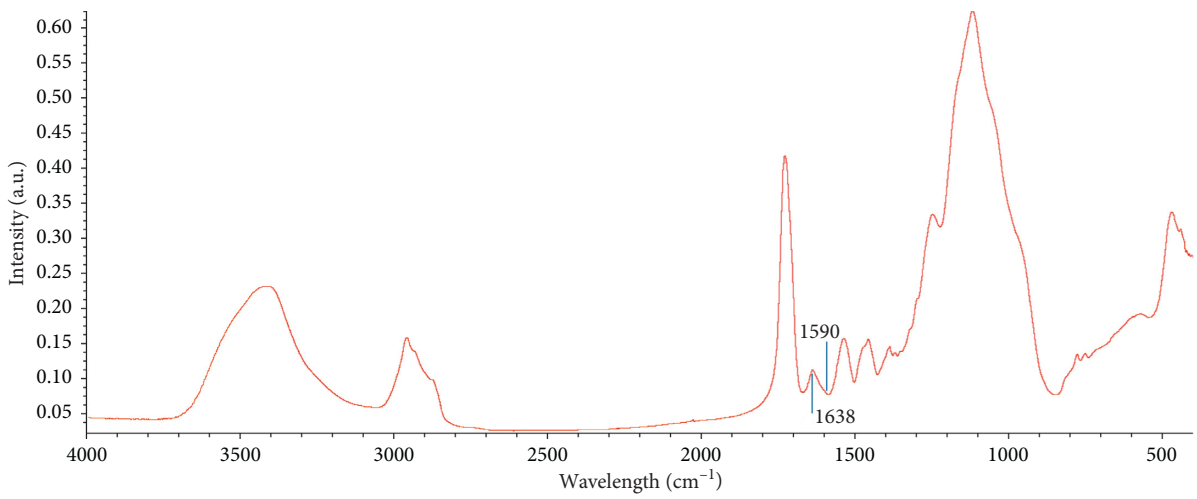
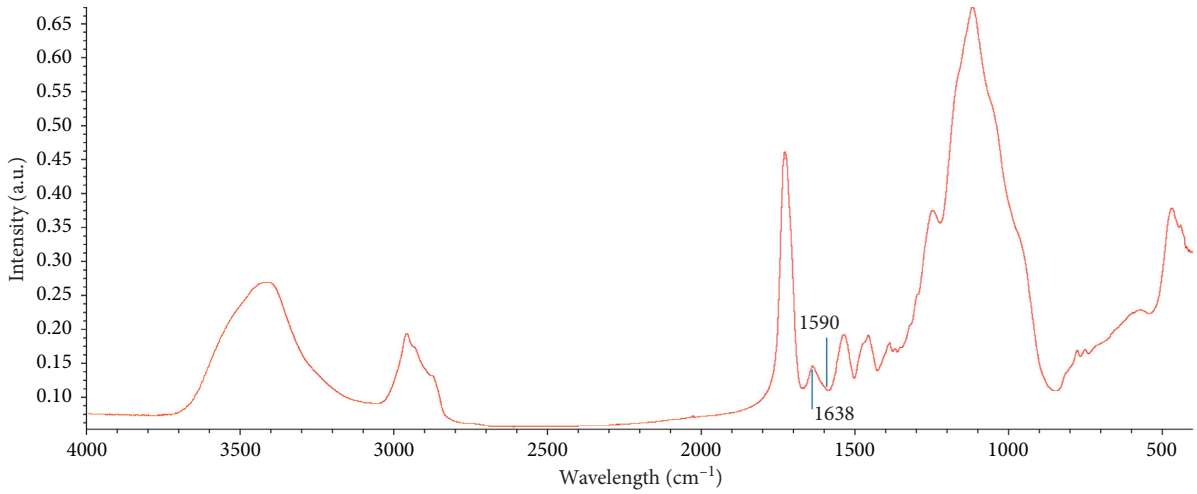


(f)

Figure 3: Continued.



(g)

Figure 3: Representative FTIR spectra of all tested groups. (a) Unpolymerized resin cement; (b) A1 tested group; (c) A2 tested group; (d) B1 tested group; (e) B2 tested group; (f) C1 tested group; (g) C2 tested group.

inclination and curing time ($p = 0.903$ for VH and $p = 0.941$ for DC).

Based on the results, all the null hypotheses were rejected. The extent of polymerization affects the mechanical properties of resin cement [25], and the mechanical properties of resin cement determine the long-term success of restoration [3]. It has been revealed that the light-curing mode [26], light intensity [27], shade [28] and thickness [29] of ceramics, curing protocol [30], and other factors may influence the polymerization efficiency of resin cement. The light can be absorbed and scattered by the structures of restoration [31], but limited information in the literature about the influence of anatomical structures of artificial restoration on the polymerization of resin cement is present. The ordinary cusp inclinations of posterior teeth are 30° and 20°, and 0° refers to the labial surface of the anterior teeth. Therefore, three characteristic zirconia specimens were designed to simulate the ordinary cusp inclinations. Moreover, the length and width of the zirconia were set in accordance with the diameter of light-curing unit tip (10 mm).

There are several methods used to test the polymerization efficiency of resin cement, and the microhardness and DC are the most commonly used indications in the literature [20, 32, 33]. Surface microhardness is an important indication reflecting the polymerization efficiency of resin cement directly [34, 35]. Hardness is an intrinsic property of materials independent of the hardness test methods employed [36]. The VH test was chosen for the present study which is analogous to the previous studies [37–39]. Differences in hardness between the top and bottom surfaces of the specimen were frequently used to detect the polymerization, but the present study employed a different specimen design (much thinner than those in the published studies). After curing, the bottom surface of the resin cement specimen was not good enough for the hardness test. Considering the aim of the present study was to evaluate the effect of cusp inclination of zirconia on polymerization of resin cement, testing of the upper surface of resin cement can also indirectly reveal the polymerization efficiency.

Table 4: Two-way ANOVA for the VH values.

Factor	Type III SS	*df*	MS	*F*	*p*
Cusp inclination	260.475	2	130.238	8.342	0.001
Curing time	31.341	1	31.341	2.008	0.167
Cusp inclination × curing time	3.192	2	1.596	0.102	0.903
Error	468.341	30	15.611	—	—
Total	46354.852	36	—	—	—

Table 5: Two-way ANOVA for the DC values.

Factor	Type III SS	*df*	MS	*F*	*p*
Cusp inclination	279.393	2	139.697	14.577	<0.001
Curing time	65.772	1	65.772	6.863	0.014
Cusp inclination × curing time	1.163	2	0.582	0.061	0.941
Error	287.493	30	9.583	—	—
Total	108136.930	36	—	—	—

DC is a key factor which plays a decisive role in the properties of resin cement and the prognosis of restoration [40]. In the literature, DC of resin cement is determined by Fourier transform infrared (FTIR) spectroscopy and Raman spectroscopy [41]. FTIR spectroscopy offers a direct approach to evaluate the depth of polymerization and is considered to be the best method [42, 43]. DC has been determined using the areas' ratio of peaks corresponding to aliphatic (1638 cm^{-1}) and aromatic (1608 cm^{-1}) of the FTIR spectra. However, FTIR spectra of methacrylate-based dental materials also present other peaks related to carbon bond stretching, at 1715 cm^{-1} and 1580 cm^{-1}. Additionally, 1608 cm^{-1} is a representative FTIR spectrum of Bis-GMA, which cannot be found in TEGDMA [44]. Since the tested

resin cement (Multilink Speed) in the present study is mainly based on TEGDMA and UDMA, 1590 cm^{-1} was used in the analysis of FTIR spectra.

The values of VH and DC in 0° cusp inclination (flat) zirconia were in agreement with the previous studies [19, 21, 45, 46]. The highest values of VH and DC were observed in C2 (0° cusp inclination) group, and the lowest values were found in A1 (30° cusp inclination) group. This phenomenon may be explained as follows: Firstly, the distance between light source center and the resin cement was different for each group. For groups C1 and C2 (0° cusp inclination), the distance was 1.00 mm (just the thickness of zirconia). As to groups B1 and B2 (20° cusp inclination), the distance was 2.70 mm (includes the thickness of zirconia). For groups A1 and A2 (30° cusp inclination), the distance was 3.50 mm (includes the thickness of zirconia). It has been reported that when the distance between the light source and the resin cement was greater than 2 mm, the resin cement became difficult to polymerize [47]. Secondly, because the light beam was not perpendicular to the zirconia surface for the A and B (30° and 20° cusp inclination) groups, the actual distance of the light transmitted from the zirconia surface to the resin cement was more than 1.0 mm. However, the thickness of the light transmitted from the zirconia surface to the resin cement was 1.0 mm for C (0° cusp inclination) groups. As the thickness of restoration increases, the polymerization efficiency of resin cement decreases [48]. Thirdly, the direction of light affects the energy transmission. In order to improve the polymerization efficiency, the light beam should be kept perpendicular to the restoration [49]. However, the light beam was angled to the inclined surfaces and resin cement because of obstruction of the tooth cusp in the A and B (20° and 30° cusp inclination) groups, while the light beam was perpendicular to the restoration surface and the resin cement in the C (0° cusp inclination) groups.

Lower VH and DC values are synonymous of incomplete polymerization of resin cement, which resulted in increasing water absorption [50] and microleakage [51]. Based on the present findings, the clinicians should be advised to select a light-curing unit tip with relatively small diameter and make the light-curing unit tip positioned perpendicular to the tooth-inclined surfaces when light cure the resin cement through zirconia restorations with a high cusp inclination. Moreover, since curing time significantly ($p = 0.014$) affects the DC of the resin cement in this study and the VH of specimens cured 40 s was higher, it seems that prolonging curing time is necessary and useful to improve the polymerization efficiency of resin cement [52].

Undoubtedly, the structures of restoration are much more complicated than the present study's setting. Further *in vitro* and *in vivo* studies are needed to confirm the present findings.

4. Conclusions

Within the limitations of the present study, the following conclusions may be drawn: the cusp inclination of zirconia significantly affected the polymerization efficiency of dual-cured resin cement. The polymerization efficiency of dual-cured resin cement decreased as the cusp inclination increased.

Conflicts of Interest

The authors declare that there are no conflicts of interest regarding the publication of this paper.

Authors' Contributions

Chang-Yuan Zhang and Hao Yu contributed equally to this work.

Acknowledgments

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