

POLYMERIZATION EFFICIENCY OF TWO DUAL-CURE CEMENTS THROUGH DENTAL CERAMICS

İki Dual-Cure Reçine Simanın Zirkonya Seramikleri Altında Polimerizasyon Etkinliğinin İncelenmesi

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ABSTRACT

Purpose: The aim of this study was to evaluate the effect of thickness of zirconia on curing efficiency of resin cements.

Materials and Methods: Four discs with 4.0 mm in diameter were prepared from non-HIP translucent zirconia blocks using a CAD/CAM system and feldspathic ceramic was layered onto discs. Thus, 4 ceramic disc samples were fabricated: (G) 0.5 mm zirconia- as a control group, (G1) 0.5 mm zirconia and 0.5 mm feldspathic, (G2) 1.0 mm zirconia and 0.5 mm feldspathic and (G3) 2.0 mm zirconia and 0.5 mm feldspathic ceramic layer. 2 different dual cure cements were polymerized using a LED curing unit. Degree of conversion was evaluated using Vickers Hardness Test and depths of cure of samples were measured. Data were analyzed statistically using One-way ANOVA and Tukey's HSD test ($p < 0.05$).

Results: Microhardness and depth of cure values were different under same thickness of ceramic discs for two resin cements. As the thickness of the zirconia discs increased, the microhardness values and depth of cure decreased.

Conclusion: Photocuring time cannot be the same for all clinical conditions, under thicker zirconia restorations (>2.0 mm), an extended period of light curing or a light unit with a high irradiance should be used.

Keywords: Ceramic thickness, polymerization, resin cement, zirconia

ÖZ

Amaç: Bu çalışmanın amacı zirkonya kalınlığının reçine simanın polimerizasyonu üzerine etkisinin değerlendirilmesidir.

Gereç ve Yöntem: 4.0 mm çapında 4 adet disk non-HIP zirkonya bloklardan CAD/CAM sistemi kullanılarak hazırlandı ve feldspatik seramik diskler üzerine tabakalama tekniği ile uygulandı. Bu şekilde 4 adet seramik disk deney örneği elde edildi: (G) 0.5 mm zirkonya kontrol grubu, (G1) 0,5 mm zirkonya ve 0,5 mm feldspatik, (G2) 1.0 mm zirkonya ve 0.5 mm feldspatik ve (G3) 2.0 mm zirkonya ve 0.5 mm feldspatik seramik katmanı. LED ışık cihazı kullanılarak 2 farklı dual cure reçine siman polimerize edildi. Dönüşüm derecesi Vickers sertlik değeri kullanılarak değerlendirildi ve örneklerin polimerizasyon derinliği ölçüldü. Verilerin istatistiksel değerlendirilmesi tek yönlü varyans analizi (ANOVA) ve Tukey's HSD testleri ile yapıldı ($p < 0.05$).

Bulgular: Mikrosertlik ve polimerizasyon derinliği, aynı kalınlıktaki seramik örnekler altında iki farklı reçine siman için anlamlı farklılık göstermiştir. Zirkonya kalınlığı arttıkça, mikrosertlik değerleri ve polimerizasyon derinliği azalmıştır.

Sonuç: Işıklı polimerizasyon süresi farklı klinik durumlar için aynı olamaz. Kalın zirkonya restorasyonlar altında (>2.0 mm) ışıkla polimerizasyonun süresini uzatmak veya yüksek enerjiye sahip bir ışık ünitesi kullanmak uygun olabilir.

Anahtar kelimeler: seramik kalınlığı, polimerizasyon, reçine siman, zirkonya

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Introduction

Translucency is defined as variable amount of light transmission or dispersed reflectance from a substrate surface (1). The translucency of dental ceramic systems depend on the thickness of the core material to be traversed by the light beam and chemical structure of its chemical nature, the amount of crystals, the size of particles and pores within the core matrix (1). This microstructure determines the amount of light that is absorbed, reflected and transmitted. All of these factors influence the translucency of core ceramics (2, 3). Recent studies have suggested that with the advance of computer-aided design/manufacturing (CAD/CAM) technologies, zirconia ceramics for dental restorations offer the best mechanical and esthetic properties for all-ceramic core materials (4-6). Zirconium-oxide ceramics based on partially stabilized with yttrium (Y-TZP) have become the most preferred, especially when a high mechanical resistance is required (7, 8). Crystalline content used to strengthen the ceramic reduces the translucency because of its non-uniform structure (9). Y-TZP consist of polycrystalline particles, therefore it has a different refractive index to the matrix, which could impact the quantity of light that passes through for activation of the resin-luting cement. The mechanical property of zirconia core has been studied but recent studies focused on its thickness. Ceramic translucency is also affected by the ceramic thickness (10).

Although luting zirconia based restorations can be performed with conventional cements, adhesive cementation has been recommended for improving the clinical retention (11). In recent years, improvements in all-ceramic systems and adhesive resin cements have contributed considerably to esthetic dentistry (12). Resin-based composite cements are the standard material preferred to be used in luting a ceramic prosthetic to tooth structures (13). Resin cements are preferred because of their high bond strength to ceramic, low solubility, good esthetics, easy handling and superior mechanical properties (14). Resin cements are desirable in many clinical situations such as short or tapered prepared tooth structure. Retention of a dental restoration to tooth structure and sealing of the marginal gap between the restoration and tooth are dependent on the luting agent's ability to bond to the surface of the ceramic (15). In addition, it is likely that strong chemical adhesion would lead to enhanced long-term fracture and fatigue resistance in the oral environment (16).

According to the polymerization methods, resin

cements have three types: self-cure, light-cure and dual-cure and their selection is based primarily on the intended use (17). Self cure resin cements have a shorter working time, but their use is not limited by porcelain thickness. These cements are uniformly set even at the bottom of deep cavities, where access for curing light is limited. Light-cure resin cements have ideal working time, but to prevent inadequate polymerization, they shouldn't be used if the restoration is thicker than 3 mm. Dual cure resin cements have in their composition both photo initiator (camphoroquinone) and the chemical activation components (peroxide/amine) to achieve the best polymerization and working results. For a dual polymerization cement, an adequate amount of light is required to initiate the polymerization process (18). The composition, thickness, opacity and shade of the ceramic may weaken the light from the curing unit used to polymerize the resin cement under the ceramic restoration (19). As crystalline ceramics are opaque, they would be expected to attenuate more light (20). Limited information is available on the effect of composition, opacity, and thickness of ceramic materials on light attenuation.

The mechanical property of zirconia core has been studied in many aspects, but few studies have focused on its translucency and effect of its thickness on light transmission. The aim of this study was to evaluate the effect of thickness of zirconia on curing efficiency of resin cements. The first hypothesis tested was that the curing efficiency of the dual-cure resin cement is not affected by the thickness of the zirconia restoration. The second hypothesis was that the different dual cure resin cements are not affected by the thickness of the restoration when used under zirconia structures.

Materials and Methods

Specimen preparation for Vickers hardness

In order to evaluate the efficiency of substructure thickness on curing efficiency of resin cements, 4 disc samples with 4.0 mm in diameter were prepared (Table 1). The zirconia substructures were prepared from non-hot isostatic pressing (HIP) blocks (Kavo Everest BIO ZS-blanks; KaVo Dental GmbH, Baden-Wurttemberg, Germany) using CAD/CAM system (Kavo Everest; KaVo Dental GmbH, Baden-Wurttemberg, Germany) and sintered according to manufacturer's instructions. The feldspathic layering (GC Initial Zr, GC Europe, Leuven, Belgium) was performed using a stainless steel mold in order to achieve a standard thickness.

Table 1. *Substructure and restoration thicknesses of the groups.*

Groups	Zirconia thickness (mm)	Feldspathic ceramic thickness (mm)	Restoration thickness (mm)
G	0.5	-	0.5
G1	0.5	0.5	1.0
G2	1.0	0.5	1.5
G3	1.5	0.5	2.0

To evaluate the degree of conversion, Vickers hardness numbers (VHN) of the resin specimens were evaluated according to ISO 4049 (21). In the present study to evaluate the ceramic thickness on curing efficiency of resin cements, two dual cure resin cements were used; Panavia F 2.0 TC (Kuraray Medical Inc, Japan) and Bisco Duo-Link (Bisco Inc., Itasca, IL, USA). Both resin cement specimens were mixed and applied according to the manufacturer's instructions. To obtain the specimens, resin cements were placed in teflon molds (4.0-mm diameter, 6.0-mm height). The mold was placed onto a strip of the transparent film on a glass microscope slide and filled with the test materials. A second strip of the transparent film was put on top, followed by the second microscope slide. The mold and strips of film were pressed between the glass slides to remove the excess material. The microscope slide covering the upper strip of film was removed and the relevant ceramic disc and the tip of the light source was gently placed against the strip of film. The LED curing unit (Elipar S10, 3M, ESPE, Saint Paul, MN, USA) was used for 20 s exposure time. The unit had a wavelength range of 430-480 nm and a power density of 1200 mW/cm². 4 groups (n=12) were prepared; using Panavia F 2.0 (Kuraray, Tokyo, Japan) and 4 groups (n=12) were prepared; using Bisco Duo-Link resin cement.

Depth of cure

The specimens were taken out of the mold, and the uncured material was removed using a plastic spatula according to ISO 4049 (21). To evaluate the depth of cure, the height of the cylinders of cured material was measured with a digital micrometer (Mitutoyo Corp., 150 mm series, Kanagawa, Japan) to an accuracy of 0.01 mm. Following the procedure, specimens were stored in dry, light-proof containers for 24 hours.

Universal hardness

To measure the universal hardness of the samples, the specimens were embedded in a programmable automatic mounting press (Metkon Ecopress 100, Metkon Ins., İstanbul, Turkey) by cold curing methyl-methacrylate using cylindrical molds and, to obtain a smooth surface for hardness testing, longitudinally wet-flattened with 240-, 320-, 400-, 600-, and 1200-grit SiC papers. For measuring the universal hardness, three measuring points were determined on each specimen. Vicker's hardness measurements were made using a micro hardness tester (Wolpert Wilson Instruments, 400 Series Vickers Hardness Tester, Esslingen, Germany) with a 50-g load applied for 15 seconds in the cross-sectional area at three depths that were determined as 100 µm- 300 µm and 500 µm deep from the top surface of each specimen (22).

Statistical analysis

Data were analyzed statistically using One-way ANOVA and Tukey HDS test (SPSS for Windows 15.0; SPSS, Chicago, IL, USA) with significance level at $p < 0.05$. Pair wise comparisons among groups were analyzed by Student's t test at the 0.05 level of significance.

Results

The depth of cure and the VHN values of Bisco and Panavia resin cements polymerized using a LED light source under zirconia discs of different thickness were compared using One-way ANOVA and Student's t tests. The results of statistical analysis are displayed in Table 2 and Table 3.

Table 2. Evaluation of depth of cure under different ceramic thicknesses with different resin cements (SD: standard deviation).

Thickness	Depth (mm)		p
	Panavia F	Duo-Link	
	Mean±SD	Mean±SD	
G	7.21±0.09	7.18±0.23	0.601
G1	6.76±0.33	6.66±0.37	0.488
G2	6.31±0.34	4.80±0.27	0.001**
G3	4.42±0.73	4.12±0.72	0.332

Student's t test * $p < 0.05$, ** $p < 0.01$

Table 3. Mean VHN values of groups under different ceramic thicknesses with different resin cements (SD: standard deviation).

Measurement Depth (µm)	Resin Cement	Groups				p
		G	G1	G2	G3	
		Mean±SD	Mean±SD	Mean±SD	Mean±SD	
50	Panavia	69.95±5.35	65.26±2.55	62.80±2.83	52.10±3.47	0.001**
	Duo-Link	66.05±3.49	58.43±4.96	55.18±3.89	47.78±1.93	0.001**
100	Panavia	62.67±4.70	58.00±4.14	54.15±2.73	49.33±4.82	0.001**
	Duo-Link	56.12±3.39	48.29±1.59	44.78±4.10	39.37±4.51	0.001**
150	Panavia	53.15±7.64	49.00±3.89	43.64±2.52	39.41±3.37	0.001**
	Duo-Link	48.15±3.35	43.47±1.77	39.23±5.99	29.68±3.82	0.001**

Oneway ANOVA test ** $p < 0.01$

For all the measurement depths (100 µm, 300 µm and 500µm), Panavia samples had significantly higher VHN values compared to those in Bisco samples under each zirconia subgroup (Table 3). In the G subgroup of the samples, the difference between Panavia and Bisco groups were less, and it was found to be statistically significant ($p < 0.05$). However, as the thickness of the zirconia disc increased as in G1, G2 and G3 subgroups, the difference between measurements of resin groups increased and it was found to be highly significant ($p < 0.001$).

As the VHN values in/of each resin cement group were evaluated according to the measurement depth, following evaluations were reached:

For the first and second measurement depths (100 µm and 300 µm) in the Panavia and Bisco groups, the difference between different zirconia subgroups were statistically significant ($p < 0.01$). The VHN values for G subgroup were significantly higher than the ones

in G1, G2 and G3 subgroups ($p < 0.05$; $p < 0.01$). G3 subgroup had the lowest VHN value in all subgroups and this difference was significantly lower than that in G1 and G2 subgroups ($p < 0.01$). There was no statistically significant difference between VHN values of G1 and G2 subgroups ($p > 0.05$).

For the third measurement depth (300µm) in the Panavia group, the difference between different zirconia subgroups was statistically significant ($p < 0.01$). The VHN values for G subgroup were significantly higher than those in G2 and G3 subgroups ($p < 0.05$; $p < 0.01$). There was no statistically significant difference between VHN values of G and G1 subgroups ($p > 0.05$). Also, there was no statistically significant difference between VHN values of G1 and G2 subgroups ($p > 0.05$).

- For the third measurement depth (300µm) in the Bisco group, the difference between different zirconia subgroups was statistically significant ($p < 0.01$).

The VHN values for G subgroup were significantly higher than those in G1, G2 and G3 subgroups ($p < 0.05$; $p < 0.01$). G3 subgroup had the lowest VHN value of all subgroups and this difference was significantly lower than that in G1 and G2 subgroups ($p < 0.01$). There was no statistically significant difference between VHN values of G1 and G2 subgroups ($p > 0.05$).

As the depths of cure of subgroups were compared, following results were found:

- There was no statistical significance between G Panavia and G Bisco subgroups ($p > 0.05$).

- There was no statistical significance between G1 Panavia and G1 Bisco subgroups ($p > 0.05$).

- The difference between G2 Panavia and G2 Bisco subgroups was statistically significant. ($p < 0.01$).

- There was no statistical significance between G3 Panavia and G3 Bisco subgroups ($p > 0.05$).

In the Panavia group, there is a statistically significant difference between depth of cure of sub-groups ($p < 0.01$) (Table 3). Depth of cure of G subgroup is highly significant compared to that of G2 and G3 subgroups ($p < 0.01$). Depth of cure of G3 subgroup is highly low compared to that of G1 and G2 subgroups ($p < 0.01$). There is no statistically significant difference between G and G1 subgroups ($p > 0.05$).

In the Bisco group, there is a statistically significant difference in the depth of cure of sub-groups ($p < 0.01$) (Table 3). Depth of cure of G subgroup is highly significant compared to that of G1, G2 and G3 subgroups ($p < 0.05$, $p < 0.01$). Depth of cure of G1 subgroup is highly significant compared to that of G2 and G3 subgroups ($p < 0.01$). Depth of cure of G2 subgroup is highly significant compared to that of G3 subgroup ($p < 0.05$, $p < 0.01$).

Discussion

The first hypothesis tested was partially rejected as the thickness of the zirconia discs increased, the micro hardness values and depth of cure decreased. The second hypothesis was also partially rejected since the micro hardness values and depth of cure values were different under the same thickness of zirconia discs for 2 resin cements tested.

Ideally, the dental restorative resin would have all of its monomer converted to polymer during the polymerization reaction. However, monomers display considerable residual unsaturation in the final product, and the degree of conversion (DC) ranges from 55% to 75% under conventional irradiation (23, 24).

As the light curing is applied in the polymerization of composite, DC depends on the energy received. The intensity of light and exposure time affects DC (24, 25, 26).

There is a relation between micro hardness of the resin cement and DC of the material. In several studies, changes in the resin cement shade and ceramic thickness resulted in significant differences in micro hardness of the materials and final polymerization (1, 27, 28) Micro hardness tests are accepted to be a reliable indirect test method for DC(29-33). Lower micro hardness values can be interpreted as a sign of incomplete polymerization and low DC.

The dual-cure resin cements were developed to ensure the polymerization which curing light can't reach. According to el-Mowafy *et al.* (34) in the ideal dual-curing situation the cement should have similar hardening through self-curing compared to dual-curing. However, it is reported by several authors that self-curing component is not enough by itself to achieve required hardness (35, 36).

Inadequate polymerization of the material results in the decrease of the mechanical properties increasing water sorption (37) and microleakage (38). Also, non-polymerized monomers can be debonded from the material and result in tissue inflammation. Therefore, it is of utmost importance to optimize the polymerization of the resin cement in order to obtain best clinical performance and healthy outcome for the patient. It has been well documented that the curing of dual-cure resin cement is affected negatively when used under ceramic restorations (30). However, there are very few studies investigating the curing efficiency of the dual-cure resin cement used under restorations with zirconia substructures. Adhesive cementation is preferred by clinicians because of certain advantages such as better resistance to micro leakage, ability to mask the original tooth color and better retention (39) but it is not documented as well as lithium-disilicate or similar all-ceramic systems in the literature.

Results of the study displayed that the increase in thickness of the translucent zirconia decreased the micro hardness and depth of cure of the dual-cure resin cement. This can be recognized as a decrease in the polymerization efficiency of the material. It can be speculated that the zirconia causes absorption and diffusion of light, therefore decreasing curing efficiency. It should also be noted that thickness of the material may not be the only parameter for the attenuation of light curing. Various causes for the light

diffusion within polycrystalline ceramic materials are reported (2, 39). Scattering of light may be a result of irregularities in the distribution of the phases, defects and voids at grain boundaries, optical anisotropy of the grains, grain size larger than the light wavelength, and different refractive indexes among the particles, and their chemical nature (40). Factors other than grain size may affect the behavior of light through the material. Minor dimensional, structural, and chemical differences in the grains and grain boundaries may result in an increase the opacity.

Strang *et al.* (42) evaluated the light-curing of resin cements under porcelain veneers and reported that, curing light is absorbed by the ceramic from 40% to 50%. It was also reported in the same study that shade of the ceramic does not significantly affect the light-curing efficiency for samples with thickness less than 1.5 mm. In our study, there was statistically significant difference in samples less than 1,5 mm. This contradiction with the previous similar study may be attributed to the zirconia substructure, it can be theorized that the light absorption percentages reported by Strang *et al.* (42) will increase when zirconia is used.

Therefore, it should be taken into consideration by the clinician that light-curing and by that account dual curing (43) under zirconia restorations may not be as efficient as the other, more translucent all-ceramic systems.

It can be speculated that zirconia translucency may be improved in the future. It has been reported that traditional ceramic materials display interesting optical properties when the grain size of sintered ceramics are reduced to nano dimensions (43). One example is alumina, which is known to become transparent as the grain size gets smaller (43). Zirconia grain size could be modified by the manufacturer using enhancers and by varying sintering conditions such as sintering time, pressure and temperature (7).

However, the usage of smaller grains seems to be limited by the impossibility of the phase transition when the grain size is less than approximately 0.2 μm . (44) Casolco *et al.* (45) described a technique to obtain translucent zirconia ceramics using both partially ($\text{ZrO}_2 - 3 \text{ mol}\% \text{ Y}_2\text{O}_3$) and fully stabilized ($\text{ZrO}_2 - 8 \text{ mol}\% \text{ Y}_2\text{O}_3$) nanostructured powders.

The resulting materials have a grain size of 55 nm, and the fracture toughness of $8.1 \text{ MPa}\cdot\text{m}^{1/2}$ suggests that these materials could be further developed for dental applications. Increasing translucency of the material may improve the curing efficiency even

though a thick zirconia framework is used. In the present study, two different resin cements were evaluated. Both materials are present as having translucent properties. However, the micro hardness and depth of cure values were significantly different for both materials when polymerized under same ceramic samples.

Different colors of the luting composite may significantly affect the appearance of the final restoration. Many different resin cement shades are available in the market. Former studies reported darker shades of composites are not cured to the same depth as lighter shades (46-48). Passos *et al.* (49) reported that no effect on %DC were observed when different resin cements were used in 100 μm thickness. However, they also reported that under restorations with darker feldspathic ceramic restorations, darker resin cements' curing efficiency is significantly decreased. Findings of the current study may add to this conclusion by speculating that different brands of translucent resin cements present in the market may not be cured in equivalent effectiveness.

An optimum internal gap size has been reported to be 50-100 μm for long-term performance of resin cements (50). However, Kohorst *et al.* (51) have reported that although the mean internal gap measurements ranged from 71.1 to 115.1 μm , the maximum gap size value reached at 183.6 μm for zirconia based fixed partial dentures. Also, increased film thickness of the resin cement may affect the fitting passivity of the restoration which may lead greater cement thickness far from ideal conditions. For all these reasons in this study, the measurement depths for cement hardness evaluation of the samples were chosen as 100, 300, and 500 μm in order to simulate clinical situation.

It can, therefore, be concluded that the photo curing time cannot be the same for all clinical conditions; nevertheless, the manufacturers recommend single set of curing parameters for all situations. Clinicians should be aware that, especially for dual-cure resin cement under thicker zirconia restorations (2.0 mm or more), an extended period of light curing or a light unit with a high irradiance should be used.

Conclusion

The curing efficiency of dual-cure resin cements under translucent zirconia restorations are affected negatively as the thickness of the restoration increases.

An extended period of light curing, if possible

with high irradiance light units, should be applied for the dual-cure adhesive cementation of thick zirconia restorations.

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None declared

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