Influence of Ceramic Shade and Thickness on the Polymerization Depth of **Different Resin Luting Cements**

Seramik Renk ve Kalınlığının Farklı Rezin Yapıstırma Simanlarının Polimerizasyon Derinliği Üzerine Etkisi

ABSTRACT Objective: Polymerization depth of resin cements is important to notify the clinician that the thickness and shade of all ceramic restorations can influence the polymerization of cements. The aim of this study was to determine the effects of ceramic shade, thickness and exposure time on polymerization of resin cements. Material and Methods: Disk-shaped porcelain specimens were used in six colour shades, two different thickness and test samples were prepared from light and dual-cured resin cements under these porcelain disks. The degree of resin polymerization was determined by Barcol hardness on resins were polymerized with light exposures of 30 and 60 seconds. Results: According to data using Dunnett T3 test with multiple comparisons the experimental samples showed significant differences from the control groups except for A1 and B1 shades (p < 0.001). Conclusion: In all the test conditions, dual-cured resin cements were observed to show higher surface hardness values. Increase in value of ceramic shade caused a decrease in surface hardness particularly for light-cured cement and augment in porcelain thickness resulted in a decrease in hardness values for both of the cements and for each shade group.

Key Words: Resin cement, hardness tests

ÖZET Amaç: Hekimlerin, tam seramik restorasyonların kalınlık ve renklerinin rezin simanların polimerizasyon derinliği üzerine etkisini bilmesi son derece önemlidir. Dolayısıyla bu çalışmanın amacı; seramiğin renk, kalınlık ve ışık ekspoz süresinin rezin simanların polimerizasyonu üzerine etkilerini belirlemektir. Gereç ve Yöntemler: Altı farklı renk ve iki farklı kalınlıkta porselen diskler hazırlandıktan sonra bunların altında ışıkla polimerize olan ve dual (hem ışıkla, hem de kimyasal olarak sertleşebilen) etkili rezin siman test örneklerin polimerizasyon işlemleri tamamlanmıştır. Işık cihazı ile 30 ve 60 saniyelik polimerizasyonun ardından rezinlerin polimerizasyon derinliği Barcol testi ile belirlenmiştir. Bulgular: Dunnett T3 çoklu karşılaştırma testine göre A1 ve B1 renkleri hariç diğer renk grupları ile kontrol grubu arasında istatistiksel olarak anlamlı farklılıklar tespit edilmiştir (p< 0.001). Sonuç: Bütün test şartlarında dual rezin simanlar daha fazla yüzey sertlik değerleri ortaya koymuştur. Seramik rengindeki koyulaşma artışı ışıkla polimerize olan siman grubunda yüzey sertliği için belirgin düşüşe neden olmuştur. Ayrıca porselen kalınlığındaki artış da her renk grubu ve her iki siman çeşiti için de sertlik derecesini azaltmıştır.

Anahtar Kelimeler: Rezin siman; sertlik testleri

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evelopment of the etched ceramic restorations has enabled dentists to provide an esthetic alternative to conventional metal restorations over the last few decades.^{1,2} The various luting materials such as zinc phosphate, glass-ionomer and self-activated resin cements have been previously used for ceramic restorations. However, laboratory and clinical studies have demonstrated that all ceramic restorations luted with these ce-

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ments suffer from poor marginal quality, fractures and loss of retention. Therefore, visible light-cured and dual-cured composite resins have been recommended for luting ceramic restorations. Currently these are the materials of choice for ceramic inlays and onlays, full ceramic crowns and porcelain laminate veneers.³⁻⁵

One concern regarding resin-based luting agents is the polymerization depth of the composites since the thickness and the shade of the porcelain restorations have been shown to have some effects on polymerization of cements.^{1,3,5,6} Inadequate polymerization and consequently decreased hardness of the resin cements may influence their physiomechanical properties and eventually lead to several clinical problems such as restoration failure.⁷⁻⁹

The influence of restorative composite shade on polymerization has been extensively investigated. However, little information is available concerning the correlation between polymerization depth and shade of ceramic restorations.^{10,11} It was shown that shade of porcelain may also affect the translucency and light scattering and consequently the polymerization depth of photo-activated resin cements under the restoration. In addition the thickness of ceramic material placed between the light source and the cement may affect the degree of polymerization.^{12,13}

In 1990, Blackman et al.³ reported that the thickness and type of ceramic used affected the polymerization of luting resins because of absorption of the visible light by the dental ceramic. Approximately half of the light energy emitted by the source reached the composite. Strang et al.¹⁴ found that the shade of ceramic did not markedly affect the amount of light absorbed by the ceramic or the setting times of composites. They showed that porcelain absorbs 40% to 50% of the light and that increased porcelain thickness required increased exposure times for resin polymerization. In their study, a ceramic thickness of 0.5 and 1.5 mm was used. This thickness is suitable for the laminate veneers but inlays and onlays require a minimum thickness of 1.5 to 2 mm.

The present study examined and compared the effects of light exposure time, ceramic shade and thickness on the surface hardness of underlying visible light-cured and dual-cured composite resin luting cements. Barcol hardness measurements were used to determine the effects of two different exposure time, ceramic shade and thickness on resin based luting cements.¹⁵

MATERIAL AND METHODS

Twelve porcelain disks were constructed using Colorlogic Veneer Porcelain (Ceramco Inc., Burlington, USA) according to manufacturer's instructions. These disk-shaped specimens, 10 mm in diameter, were made in two different thicknesses (1.5 and 2.5 \pm 0.05 mm) and six colour shades (A1, A3, B1, B3, C1 and C3). The shades of the majority of dental ceramics in current use are keyed to the Vita Lumin Shade guide (Vita Zahnfabrik H.Rauter GmbH, Säckingen, Germany), which is normally arranged in groups designated by the shade letters A, B, C and D.¹¹ Therefore in the present study this shade guide was selected. These ceramic specimens were glazed on one side of each specimen.

Two composite resin-based cements that visible light-cured (Tetric Flow, Ivoclar Vivadent AG, Schaan, Liechtenstein) and dual-cured (RelyX ARC, 3M Dental Products, St. Paul, USA) with same shade were selected for this study. Following manufacturers' instructions, the control groups for each of resin cements were prepared. Each resin sample were placed in a Plexiglass mould, which had a hole, 6 mm in diameter and approximately 1 mm deep, and covered with a celluloid strip. A glass microscopic slide (1 mm thicknesses) was placed over the strip, and the resin samples were polymerized with a visible-light source (QHL 75 Curing Light, Dentsply Caulk, Milford, USA). These samples constituted the control groups for each of resin cements. The experimental samples were made using the same technique described above except they were sandwiched between a porcelain disk and the brass plate instead of glass microscopic slide (Figure 1). The resins were then polymerized through the porcelain disks. Resin samples were light-cured with same visible-light source, at an ex-



FIGURE 1: Method used for polymerization of resin cements through porcelain disks or glass microscopic slide.

posure time 30 and 60 seconds, showing an average intensity of 500 mW/cm^2 .

In this study 54 different combinations including the control groups were existed. The samples were mounted on brass plate so that surfaces polymerized against glass and discs were exposed for surface hardness measurements. After polymerization of specimen, a measurement device (Barber Colman Impressor, Barber Colman Co, Illinois, USA) was used to determine Barcol hardness on the surface that had been directly exposed to the light. The degree of polymerization was determined by four Barcol hardness measurements that were made on each resin sample, one in each quadrant. Their average values were recorded as the final hardness values. Hence, 54 different combinations provided 270 resin samples and 1080 measurements.

Data analysis was performed using a standard statistical software package (SPSS 11.0 for Windows, SPSS Inc., Chicago, Illinois, USA). Average hardness data were analysed using three–way analysis of variance (ANOVA). Multiple comparisons were performed with Dunnett T3 post-hoc test because of unequal group variances. The level of statistical significance was set to p < 0.001 to determine the difference so similar clinical characteristics of light-cured and dual-cured composite resin cements.

Porcelain Shade	Porcelain Thickness (mm)	Exposure Time (second)	Surface Hardness ± SD
Glass		30	54.00 ± 0.82
		60	60.50 ± 0.58
A1	1.5	30	41.50 ± 0.58
		60	53.00 ± 0.82
	2.5	30	31.50 ± 1.00
		60	51.75 ± 0.50
A3	1.5	30	17.75 ± 0.50
		60	43.75 ± 0.50
	2.5	30	10.50 ± 0.58
		60	19.50 ± 0.58
B1	1.5	30	29.25 ± 0.96
		60	44.00 ± 0.00
	2.5	30	20.00 ± 0.82
		60	39.00 ± 0.82
B3	1.5	30	17.75 ± 0.50
		60	42.50 ± 0.58
	2.5	30	10.00 ± 0.00
		60	19.25 ± 0.96
C1	1.5	30	29.00 ± 0.82
		60	43.75 ± 0.50
	2.5	30	15.25 ± 0.50
		60	37.00 ± 0.00
3	1.5	30	12.25 ± 0.50
		60	28.50 ± 1.29
	2.5	30	10.25 ± 0.50
		60	10.25 ± 0.50

Porcelain Shade	Porcelain Thickness (mm)	Exposure Time (second)	Surface Hardness ± SD
Glass		30	69.75 ± 0.96
		60	69.75 ± 0.96
A1	1.5	30	65.75 ± 0.50
		60	67.00 ± 0.82
	2.5	30	61.00 ± 0.82
		60	64.75 ± 0.50
A3	1.5	30	53.50 ± 0.58
		60	54.00 ± 0.82
	2.5	30	48.25 ± 0.50
		60	52.00 ± 0.82
B1	1.5	30	64.50 ± 0.58
		60	66.75 ± 1.70
	2.5	30	61.25 ± 0.50
		60	63.75 ± 0.50
B3	1.5	30	50.50 ± 0.58
		60	51.75 ± 0.50
	2.5	30	47.50 ± 0.58
		60	49.00 ± 0.00
C1	1.5	30	50.75 ± 0.50
		60	52.25 ± 0.50
	2.5	30	18.00 ± 0.00
		60	38.00 ± 0.00
C3	1.5	30	14.25 ± 0.50
		60	28.75 ± 1.70
	2.5	30	10.50 ± 0.58
		60	11.00 ± 0.81

TABLE 2: Mean surface hardness values for dual-activated resin cement for different porcelain shade and thickness at dif-

RESULTS

ANOVA results of Barcol hardness data of visible light-cured and dual-cured composite resin luting cement samples are shown in Table 1 and Table 2. According to the Dunnett T3 multiple comparisons test, the experimental samples showed significant differences from the control groups except for A1 and B1 shades. Within the experimental groups, A1 demonstrated significant differences from B3, C1 and C3, while A3, B1, B3 and C1 showed significant differences from C3 (Table 3). For the light-cured composite resin cement the thickness of the porcelain was found to have an effect on the surface hardness for control and between experimental samples. For this cement A3, B3 and C3 exhibited differences from the control groups. Furthermore the porcelain thickness was statistically significant in C1during 30 second curing period and 60 second for A3, B3 and C3. Among the all the samples tested resin cements polymerized with shade C3 porcelain groups were found the softest (p < 0.001) (Figure 2).

The ceramic thickness was found to cause statistically significant differences between the experimental and control samples for dual-cured resin cements. However no differences were observed amongst the experimental specimens in the same cement. These findings were different from those for light-cured resin samples. When the effects of shades on the surface hardness of the dual-cured resin cement were investigated no significant differences were found between A1 and B1also between A3, B3 and C1. The C3 group demonstrated significant differences from the all dual-cured resin samples (p < 0.001) (Figure 3).

DISCUSSION

The hardness of a material is generally related to several of the mechanical properties. In terms of resin-based luting cements one of the factors affecting micro-hardness of the material is their polymerization type. Polymerization depth of visible light or dual-cured cements may vary according to a number of factors, including the chemical com-

Shade (I)	Shade (II)	Mean Difference (I – II)	Significance
Glass -	A1	8.97 ± 0.22	0.043
	A3	26.09 ± 0.22	0.000
	B1	14.94 ± 0.22	0.002
	B3	27.47 ± 0.22	0.000
	C1	28.00 ± 0.22	0.000
	C3	47.81 ± 0.22	0.000
A1 -	A3	17.13 ± 0.18	0.001
	B1	5.97 ± 0.18	0.897
	B3	18.50 ± 0.18	0.000
	C1	19.03 ± 0.18	0.000
	C3	38.84 ± 0.18	0.000
A3 -	B1	-11.16 ± 0.18	0.216
	B3	1.38 ± 0.18	1.000
	C1	1.91 ± 0.18	1.000
	C3	21.72 ± 0.18	0.000
B1 -	B3	12.53 ± 0.18	0.080
	C1	13.06 ± 0.18	0.024
	C3	32.88 ± 0.18	0.000
B3 -	C1	0.53 ± 0.18	1.000
	C3	20.34 ± 0.18	0.000
C1 -	C3	19.81 ± 0.18	0.000



FIGURE 2: Surface hardness values for light-activated composite resin cement for different test conditions.

position of the material, optical properties, intensity of the light source, type of photoactivation method and exposure time.¹⁶ Moreover, polymerization depth of composite resin cements may be a concern with porcelain thickness and shade.^{10,13} Adequate polymerization of the cements is important to ensure optimum mechanical properties. Incomplete polymerization of resins and decreased hardness may also contribute to early restoration failure. The efficiency of polymerization or poly-



FIGURE 3: Surface hardness values for dual-activated composite resin cement for different test conditions.

merization depth is vital for light-cured composites, not only to ensure optimum physiomechanical properties, but also to ensure that clinical problems do not arise because of cytotoxicity of inadequately polymerization of material.^{7,8}

Especially the choice of resin luting cement is of paramount importance to be able to obtain a successful luting for all ceramic restorations. Therefore the chemical and physical properties of the cement should initially be evaluated. In addition the polymerization behaviour of the cements may exhibit some differences with the porcelain they are used with. Particularly for the light-cured composite resin luting cements, the porcelain shade and the thickness of the restoration may negatively alter their polymerization properties of the cement. This may cause inadequate polymerization depth. Therefore the adequacy of light-cured resin polymerization is solely dependent on the intensity of the light reaching the resin. Dual-cured resin, although primarily activated by visible light, has the facility to self-cured should the light not fully polymerize the resin. The results of this study revealed that three factors influenced the polymerization depth of the light and dual-cured composite resin luting cements: exposure time of cements, shade and thickness of porcelain structure.

In recent years, LED systems which produces a specific wavelength for the polymerization of camphorquinone, are produced as an alternative for the halogen systems.^{17,18} It is claimed that, there is a correlation between the surface micro hardness of composite resin and its exposure protocols. However current researches disclosed the fact that, both systems show similar polymerization properties.^{19,20} Therefore in this research to obtain a standard polymerization protocol, halogen light source which has large wavelength intervals was used. By using this method we intend to compare the surface properties of different composites which are subjected to the same polymerization process, as bringing the head of the polymerization instrument in contact with the porcelain disk specimen's surface.

Depth of polymerization may be appraised directly and indirectly. Direct methods, which actually evaluate the degree of conversion, such as infra-red spectroscopy, laser Raman spectroscopy and nuclear magnetic resonance micro-imaging have not been accepted for routine use because of their time consuming, complex and costly in nature.^{19,21,22} Indirect methods include scraping, visual examination, dye uptake and surface hardness tests.²³ The scrape test has been accepted for use by standard organisations even though it gives no indication of quality of polymerization. Incremental surface hardness measurements have been utilized in many studies because it has been shown to be an indicator of degree of polymerization.^{4,7,24} Furthermore surface hardness could be measured from various sites of specimens. This technique was selected because it was more suitable for the purpose of this study.

Much research has been done on the polymerization depth of composite resin cements, and these studies reported adverse effects of increasing ceramic thickness on hardening of light-cured and dual-cured resin cements.^{3,5,25-28} Differing from the present study, the previous investigations did not correlate the parameters of exposure time, shade and thickness of porcelain each other in one study. Blackman et al.³ while were investigating the effect of porcelain thickness in light-cured resin cements, found that different porcelain types have different effects in terms of thickness; and specimens with 3-4 mm porcelain thickness generally caused inadequate polymerization. They revealed that this less cure was not compensated even with an increase in light exposure time. In 1999 El-Mowafy et al.⁵ tested polymerization characteristics of self-activated and dual-activated resin cements underneath ceramic inlay restorations. Their findings showed that the surface hardness of the specimens reduced significantly if ceramic thickness is over 2-3 mm's.

In order to merge the advantages of chemically and photo-activated materials dual-activated composite resin luting cements have been developed. The chemical activation component is expected to ensure complete polymerization at the bottom of deep cavities, whereas photo-activation allows immediate finishing after exposure to the light. Many studies have revealed that the chemical activation mechanism alone is less effective than dual-activation, and may be almost ineffective for some materials. ^{35,27} In 2001, Hofmann et al.² evaluated the comparison of photoactivation versus chemical or dual-activated of resin cements in one study. Their some results showed that dual-activation produced better mechanical properties than photo-activation alone.

Ideally, dual-cured resin cements should be capable of achieving a degree of hardening through

self-cured similar to that achieved through dualcured. This is to ensure adequate polymerization of the cement in those areas underneath restorations that are inaccessible to the visible light.^{5,12,28} When visible light-cured resin cements are used for ceramic restorations, there appear to be distinct differences in what can be expected from different resin-ceramic combinations. The ceramic material under which a resin cement is cured seems to exert a considerable influence on the degree of resin polymerization achievable, much the same as thickness does with direct composite resin restorations cured by visible light.^{3,6,29}

Cardash et al.¹ examined the effects of porcelain shades on resin luting cements. The shade factor was found to be effective on visible light-cured resins for the specimens with 2 mm porcelain thickness. In the same study, dual-cured resin cements demonstrated higher surface hardness values with same experimental conditions. The authors also showed that increasing the exposure time for the visible-light cured cements lead to a surface hardness that is similar to dual-cured resins. The influence of prolonged exposure time was more effective on visible light-cured than the dual-cured resin cements. Moreover Abate et al.¹⁵ showed that exposure time had no effect on the hardness values of composite restorative resins when there is no ceramic layer on the resin specimens. These results were in agreements with our finding for the control group samples which were directly exposed with visible-light.

Exposure time, shade and thickness of restoration were the important three factors that would interact with each other for the cementation of an all-ceramic restoration. Thus investigation of these three parameters in one study was assumed to be more meaningful by the authors of this study. In the light of this finding, polymerization degree of the cements appear to be directly related to the porcelain shade. An increase in value of shade might diminish the polymerization. The factors related to this phenomenon could be that the materials in order to give ceramic shade may hinder the transmission of the visible light. Furthermore an increase in porcelain thickness might aggravate this hindrance especially in the darker porcelain shades.

The results suggest that the shades A1 and B1 are possibly the most suitable for ceramic restorations in terms of their effects on resin polymerization in this study. The shade C3 appears not to provide an adequate polymerization. However, if there is a certain indication for this shade, it is recommended that dual-cured composite resin cements should be preferred for luting.

In general, extending the exposure time has a positive effect on polymerization; C3 group samples still showed certain failures particularly if the porcelain thickness is increased. Extending the exposure time was apparently more important for the materials require direct visible light exposure for all polymerization.

CONCLUSIONS

1. In all the test conditions, dual-cured composite resin luting cements were observed to show higher surface hardness values than those of light cure.

2. Increase in value of ceramic shade caused a decrease in surface hardness particularly for light-cured composite resin cement.

3. Augment in porcelain thickness resulted in a decrease in hardness values for both of the cements and for each shade group.

4. Inadequate polymerization that occurred due to increase in porcelain thickness was compensated by extending exposure times except for the cases of C3 in dual-cured and A3, B3, C3 in lightcured composite resin luting cement.

5. From the standpoint of polymerization degree, dual-cured resin cements is preferred for ceramic restorations of 2.5 mm thickness or more.

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