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ORIGINAL ARTICLE

Effects of ceramic shade and thickness on the micro-mechanical properties of a light-cured resin cement in different shades

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Abstract

Objective. The aim of this study was to evaluate the micro-mechanical properties of a light-cured resin cement in four different shades when polymerized through a leucite-reinforced glass-ceramic in different shades and thicknesses. ***Materials and methods.*** A light-cured resin cement in four different shades (HV+1, HV+3, LV-1 and LV-3) was selected for this study. The specimens were cured by using a LED-unit (Bluephase®, IvoclarVivadent) for 20 s under a leucite-reinforced glass-ceramic (IPS Empress® CAD, IvoclarVivadent) in two different shades (A1 and A3) of different thicknesses (1 and 2 mm). Specimens cured directly, without an intermediate ceramic, served as control. The specimens were stored after curing for 24 h at 37°C by maintaining moisture conditions with distilled water. Micro-mechanical properties (indentation modulus, E; Hardness, HV; creep, Cr) of the resin cements were measured with an automatic microhardness indenter (Fisherscope H100C, Germany). Twenty groups were included (n = 3), while 10 measurements were performed on each specimen. Data were statistically analyzed by using one-way ANOVA and Tukey's post-hoc test, as well as a multivariate analysis to test the influence of the study parameters. ***Results.*** Significant differences were observed between the micromechanical properties of the tested resin cements ($p < 0.05$). The resin cement shade showed the highest effect on the micromechanical properties (Partial-eta squared (η_P^2)-E = 0.45, η_P^2-HV = 0.59, η_P^2-Cr = 0.29) of the resin cement, followed by ceramic thickness (η_P^2-E = 0.38, η_P^2-HV = 0.3, η_P^2-Cr = 0.04) and ceramic shade (η_P^2-E = 0.2, η_P^2-HV = 0.26). ***Conclusions.*** Resin cement shade is an important factor influencing the mechanical properties of the material. Light shades of a resin cement express higher E and HV as well as lower Cr values compared with the darker ones.

Key Words: *Light-cured resin cement, hardness, ceramic thickness, indentation modulus, micromechanical properties*

Introduction

The use of ceramic materials has increased due to their natural appearance, fluorescence, biocompatibility, durability, chemical stability, high compressive resistance as well as their thermal expansion being similar to tooth structure [1]. The success of a ceramic restoration is mainly based on a high bond strength on the adhesion complex formed between the ceramic, resin cement and dental hard tissues [2]. In order to obtain high bond strength, an optimal curing of the luting materials is required [3]. Besides, the degree of polymerization affects the mechanical properties of the adhesive resin cements and, thus, the survival of the restoration [4]. To maintain an adequate polymerization of the material, an adequate quantity of light is required [5]. Several *in vitro* studies quantified a considerable light attenuation promoted by the ceramics [6–8]. Furthermore, the crystalline structure, the thickness, the opacity and the shade of the ceramics may play a role on the light attenuation to the resin cements under a ceramic restoration [9].

For the longevity of ceramic restorations, a vital importance is attributed to the luting materials [10]. Generally, resin based adhesive composites are used for luting the all-ceramic restorations [11,12]. These materials are classified according to their activation modes, which are chemical, photo or dual activation [13]. Light-cured resin cements are used under the thin esthetic restorations, especially on the anterior region such as porcelain laminate veneers. However, regardless of the restoration, the shade and thickness of the ceramic may vary considerably. Even an esthetic anterior restoration may have areas with more

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ceramic thickness especially on the incisal edge or with a darker shade, especially on the cervical third compared to a posterior one [14].

For cementation of porcelain veneers a light-curing luting resin is preferred by dentists due to its color stability when compared with dual-cured luting resins [15]. Nevertheless, it is important that there is enough light transmittance throughout the porcelain veneer to polymerize the light-cured luting resin. Therefore, the aim of this study was to assess the performance of a light-cured veneer luting resin cement in different shades beneath the two different shades of a leucite-reinforced glass ceramic in different thicknesses, by measuring the micromechanical properties in terms of indentation modulus (E), Vickers hardness (HV) and creep (Cr).

The tested null hypothesis were that: (i) shade of resin cement,(ii) shade and (iii) thickness of ceramic would not influence the micro-mechanical properties of the luting cement.

Materials and methods

A leucite-reinforced glass-ceramic (IPS Empress® CAD; IvoclarVivadent, Schaan, Liechtenstein) in two different thicknesses was selected for this study. VITA shades A1 and A3 of low translucency were selected for the ceramic. The descriptions of the materials used in this study are summarized in Table I.

Ceramic slices of 1 mm and 2 mm in thicknesses were prepared from the IPS Empress® CAD ceramic blocs by cutting with a low speed saw (Isomet® Low Speed Saw, Buehler®, Lake Bluff, IL). They were grounded with silicon carbide abrasive papers of grit 400, 600 and 1200 (Leco® VP 100, LecoInstrumente GmbH, Moenchengladbach, Germany).

A light-cured veneer luting resin (Variolink Veneer, IvoclarVivadent, Schaan, Liechtenstein) in four different shades of high value (HV) +1, HV+3, low value (LV) -1, and LV -3 was selected for this study. The manufacturer suggests a curing time of 10–30s in each direction for the use of Variolink Veneer luting resin when luting the restorations to the teeth with anatomical contours. However, the measurements were made from the top plane surface of each specimen in this study. This means that curing took place only in one direction at an angle of 90° and, thus, perpendicular to the horizontal, plane surface of the specimens. Therefore, luting resin films were cured by use of a LED-unit (Bluephase®, IvoclarVivadent, Schaan, Liechtenstein, 1200 mW/cm^2) with a curing time of 20 s. The combination of all parameters gives a total of 20 groups, each containing three thin luting-resin films of ca. 170 µm in thickness. In order to avoid oxygen-inhibition during polymerization, Mylar strips (Frasaco, Tettnang, Germany) were positioned over the luting resins before the curing procedure. The curing unit was directly centered on specimens' surface to transmit the maximum energy of light onto the surface of measurement (Figure 1). After curing, the specimens were stored for 24 h at 37°C by maintaining moisture conditions with distilled water.

E, HV and Cr were evaluated by using an automatic microhardness indenter (Fischerscope H100C, Fischer, Sindelfingen, Germany) according to DIN 50359-1:1997-10 [16,17]. For each specimen, 10 indentation points were selected ($n = 3$). All the indentations were completely under the curing light contact area at the center of the resin cement. The distances between the indentation points were less than 1 mm [15,16].The test procedure was carried out in a force-controlled manner. The test load increased and decreased at a constant rate, varying between 0.4 mN and 30 mN. The load and the penetration depth of the indenter were continuously measured during the load–unload hysteresis. The Universal hardness is defined as the test force divided by the area of indentation under the applied load. From a multitude of measurements stored in a database supplied by the manufacturer, a conversion factor between Universal hardness and Vickers hardness was calculated and implemented into the software such that the measurement results were indicated in units of the more familiar HV. The indentation modulus was calculated from the slope of the tangent of the indentation depth curve at maximum force and is comparable with E of the material. By measuring the change in indentation depth for 5 s with a constant test force of 30 mN, a relative change

Table I. Materials used in this study.

Brand name	Manufacturer	Composition	Remarks	Shade	LOT Number
Variolink Veneer	IvoclarVivadent, Schaan, Liechtenstein	Dimethacrylates, inorganic fillers, ytterbium trifluoride, catalysts and stabilizers, pigments	Light-cured resin cement	HV+1	M13040
				HV+3	L33190
				LV -1	L42483
				LV -3	L37468
IPS Empress®CAD	IvoclarVivadent, Schaan, Liechtenstein	SiO_2, Al_2O_3, K_2O, Na_2O, other oxides, pigments	Leucite reinforced glass-ceramic Low translucency	A1	M02654
				A3	L22802

*According to manufacturers' information.

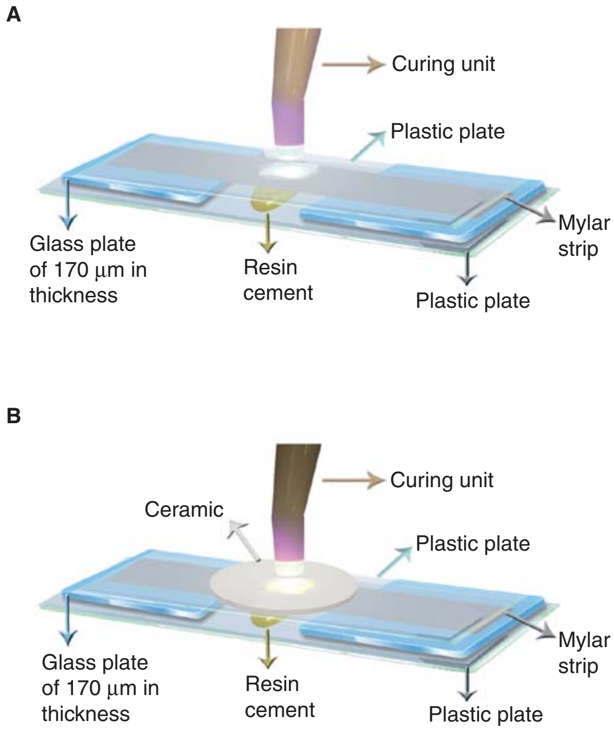

Figure 1. (A) Illustration for the application of curing unit onto the resin cement directly without any ceramic. (B) Illustration for the application of curing unit onto the resin cement under the ceramic. Light probe dimension: 10 mm in diameter; Testing area dimension: 10 mm in diameter on the center of the resin cement; Ceramic slice dimensions: L × W × H = 12 × 14 × 1 or 12 × 14 × 2 mm.

in the indentation depth was calculated. This was taken as value of Cr of the materials.

Results were compared using the one-way ANOVA and the Tukey's HSD post-hoc tests, as well as a multivariate analysis (general linear model) to test the influence of luting resin shade, ceramic shade and ceramic thickness ($\alpha = 0.05$) (SPSS 21.0).

Results

As a function of tested parameters, which are ceramic shade, ceramic thickness and resin cement shade, descriptive statistics of the micromechanical properties of the resin cements are shown in Table II. The statistical analysis revealed significant differences in the micromechanical properties of the tested resins (Table II) ($p < 0.05$).

In general, HV +1 resin cement exhibited higher E and HV, as well as lower Cr values than the other values of resin cement (Table II). HV +1 resin under 1 mm-A1 ceramic exhibited the highest E (3.6 ± 0.4), highest HV(17.3 ± 2.9) and lowest Cr (4.7 ± 0.2) values. There was no statistical difference between the E values of the HV +1 resin without ceramic (3.2 ± 1) and LV -3 resin without ceramic (3.5 ± 0.4) ($p > 0.05$). LV -1 resin under 2 mm-A3 ceramic showed the lowest E value (1.8 ± 0.1), which was not statistically different from LV -3 resin under 2 mm-A3 ceramic (1.9 ± 0.1) ($p > 0.05$). LV -3 (7.2 ± 0.7) and

Table II. Indentation modulus (E), Vickers hardness (HV) and creep (Cr) values of the resin cements as a function of the tested parameters.

Ceramic shade	Ceramic thickness (mm)	Resin cement shade	E	HV	Cr
Reference without ceramic	0	HV+1	$3.2 \pm 1^{h,i}$	$16 \pm 3.5^{i,k}$	$4.9 \pm 0.5^{b,c,d}$
		HV+3	$2.3 \pm 0.2^{c,d}$	$9 \pm 1.4^{b,c,d}$	$5 \pm 0.3^{b,c,d}$
		LV -1	$3 \pm 0.5^{g,h}$	$13.5 \pm 2.4^{g,h}$	$5 \pm 0.2^{b,c,d,e}$
		LV -3	$3.5 \pm 0.4^{i,k}$	$13.1 \pm 1.9^{f,g,h}$	5.4 ± 0.3^{h}
A1	1	HV+1	3.6 ± 0.4^{k}	17.3 ± 2.9^{k}	$4.7 \pm 0.2^{a,b}$
		HV+3	$2.6 \pm 0.1^{e,f}$	10 ± 0.8^{d}	$5.1 \pm 0.3^{d,e,f,g}$
		LV -1	$2.8 \pm 0.3^{f,g}$	$12.3 \pm 1.7^{f,g}$	$5 \pm 0.3^{b,c,d,e}$
		LV -3	$3.3 \pm 0.2^{h,i,k}$	$13.1 \pm 2.2^{f,g}$	$5.3 \pm 0.3^{g,h}$
	2	HV+1	$3 \pm 0.2^{g,h}$	$13.3 \pm 1.6^{g,h}$	$4.8 \pm 0.3^{a,b}$
		HV+3	$2.5 \pm 0.1^{d,e,f}$	$10.2 \pm 0.8^{d,e}$	$4.9 \pm 0.2^{b,c,d,e}$
		LV -1	$2.4 \pm 0.1^{c,d,e}$	$10.3 \pm 1^{d,e}$	$4.9 \pm 0.2^{b,c}$
		LV -3	$2.5 \pm 0.2^{d,e,f}$	$9.5 \pm 1.2^{c,d}$	$5.1 \pm 0.3^{c,d,e,f,g}$
A3	1	HV+1	$3.4 \pm 0.3^{i,k}$	$14.6 \pm 1.4^{h,i}$	4.8 ± 0.2^{b}
		HV+3	$2.6 \pm 0.1^{d,e,f}$	9.6 ± 0.6^{d}	$5.2 \pm 0.2^{e,f,g,h}$
		LV -1	$2.5 \pm 0.1^{d,e,f}$	10 ± 0.7^{d}	$4.9 \pm 0.3^{b,c,d}$
		LV -3	2.7 ± 0.3^{f}	9.7 ± 1.2^{d}	$5.2 \pm 0.2^{f,g,h}$
	2	HV+1	$2.7 \pm 0.2^{f,g}$	$11.7 \pm 1.1^{e,f}$	$4.8 \pm 0.3^{a,b}$
		HV+3	$2 \pm 0.1^{b,c}$	$8 \pm 0.7^{a,b,c}$	$5 \pm 0.4^{b,c,d,e}$
		LV -1	1.8 ± 0.1^{a}	$7.8 \pm 0.5^{a,b}$	4.6 ± 0.3^{a}
		LV -3	$1.9 \pm 0.1^{a,b}$	7.2 ± 0.7^{a}	$5.2 \pm 0.3^{f,g,h}$

Superscript letters show statistically homogeneous sub-groups ($p > 0.05$).

Table III. Effect of study parameters on the micromechanical properties of resin cements.

Parameters	Partial Eta-squared values		
	E	HV	Cr
Resin shade	0.45	0.59	0.29
Ceramic shade	0.20	0.26	—*
Ceramic thickness	0.38	0.3	0.04
Resin & ceramic shade	0.05	0.03	0.02
Resin shade & thickness	0.05	0.08	0.02
Ceramic shade & thickness	0.02	0.02	—*
Resin shade & ceramic shade & thickness	—*	0.03	0.02

*Statistically no significant effect ($p > 0.05$).
The higher the partial eta-squared value, the stronger the effect of study parameters on the measured micromechanical properties.

LV -1 (7.8 ± 0.5) resin cements under 2 mm-A3 ceramic showed the lowest HV values among all groups. LV -3 resin cement showed the highest Cr values for all of its sub-groups (Table II).

Table III presents the level of the effect of different ceramic shades, ceramic thicknesses and resin cement shades on the micromechanical properties of the resin cements by showing the eta-squared values derived from ANOVA analysis. The effect of resin shade on the E and HV values of the resins was the highest, followed by ceramic thickness and ceramic shade, respectively. As for the creep, the highest effect on this property was exerted by the resin shade, whereas the ceramic thickness exhibited a very low effect and the ceramic shade showed no impact (Table III). Ceramic shade (p = 0.463) and ceramic shade together with the ceramic thickness (p = 0.52) did not affect the Cr values of the resins. The combined impact of these three parameters on the E values was not statistically significant (p = 0.09).

Discussion

The present study analyzed the micromechanical properties of a light curing composite resin cement in four different shades. The resin cements polymerized under different thicknesses and shades of a ceramic and the effect of resin cement shade, ceramic shade as well as ceramic thickness on the micromechanical properties was evaluated. A favorable luting material was reported to have high values of HV and E along with small values of Cr [15]. Therefore, all these effects were expressed in terms of E, HV and Cr measured on the thin resin cements.

Clinically, full ceramic restorations are cured finally when the restoration is completely positioned after the application of both the resin cement and the adhesive bonding agent to the adhesion surfaces [18,19]. As the ceramic absorbs a certain percentage of the emitted light, an adequate curing of the adhesive resin cement under the ceramic is essential for the success of a restoration [2,19]. Therefore, the micromechanical properties of a material can be a factor of influence when the material is in clinical service [20].

Differences in color between different shades of the resin cements were noticed and can be understood as different values of the color coordinates a* and b*. Different shades of luting materials may give different colors to the final restoration based on the fact that resin cements contain some opaque ingredients in different amounts [21]. The selected resin cement (Variolink Veneer) for this study is available in seven shades or degrees of translucency, ranging from the High Value +3 (HV+3), corresponding to an opaque white bleach shade, via the highly translucent Medium Value 0 (MV0), to the Low Value -3 (LV -3) shade showing a yellow-reddish tinge [17]. Whereas HV+3 and HV+1 contain more opaque and bleach content than low values, LV -3 and LV -1 include more yellow colorant than the bleach shades. Therefore, these four different shades of the light-cured resin cement were selected for this study.

Surface hardness is a parameter frequently used to evaluate material surface resistance to plastic deformation by penetration [22]. HV+1 specimens exhibited higher E and HV values as well as lower Cr values among all ceramic shades and thicknesses. Furthermore, resin cement shade was the most effective parameter on the micromechanical properties of the resin cements in this study. Therefore, transparent resin cements may absorb more light and the depth of polymerization of these resins can be much more than that of the opaque ones.

The partial eta-squared is defined as the proportion of total variation of the dependent variable attributable to a factor, excluding other factors from the total non- error variation. It represents an index of strength of association between an experimental factor and the dependent variable and ranges normally between 0–1 [23]. In the present study, the ternary product of all parameters showed no effect on E values of the resins, whereas it affected the HV and Cr values slightly.

Since this study is mainly focused on the effect of both resin and ceramic shade, polymerization time was kept constant. When examined individually, the color of the resin as well as the color of the ceramic has influenced the micromechanical properties of the resins. This may be explained by lighter materials transmitting more light than darker ones.

The effect of ceramics on the mechanical properties of resin cements has been investigated in many *in vitro* studies [14–16]. However, limited information exists about the micromechanical properties of resin cements in different shades when cured, especially under the dental ceramics of different thicknesses and different shades. Kilinc et al. [14] evaluated the Vickers microhardness of resin cements in corresponding shades when cured under the ceramics of different shades and thicknesses. They reported that ceramic thickness has an intense effect on the resin microhardness and light transmission. Furthermore, only one resin was significantly influenced by the shade of the ceramic in their study. In agreement with their study, we found that ceramic thickness was more effective on the micromechanical properties of the resin cements than ceramic shade.

The present study demonstrates that resin cement shade, ceramic shade and thickness influence the micromechanical properties of the selected resins. Therefore, these factors can play a role in the clinical success of the ceramic restorations. However, the present study is limited by including only one kind of ceramic and resin cement as well as a constant exposure time. Furthermore, no long-term measurement was performed by assessing the behaviour of the tested materials after aging. Therefore, further research is needed to investigate other possible factors affecting the mechanical behavior of resin cements.

Conclusion

Within the limitations of this study, the following conclusions may be drawn:

(1) The resin cement shade has a significant effect on the micromechanical properties of the resin cement.
(2) Resin cements in light colors express better performance compared to the dark-colored resin cements.
(3) Ceramic thickness and shade affect the micromechanical properties of the resin cements.
(4) Light color ceramics of thin thickness allow more light transmission.

Declaration of interest: The authors report no conflicts of interest. The authors alone are responsible for the content and writing of the paper.

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