Journal of Oral Science, Vol. 50, No. 2, 131-136, 2008

Original

Effects of ceramic thickness and curing unit on light transmission through leucite-reinforced material and polymerization of dual-cured luting agent

Milena C. Pazin, Rafael R. Moraes, Luciano S. Gonçalves, Gilberto A. Borges, Mário A. C. Sinhoreti and Lourenço Correr-Sobrinho

Department of Restorative Dentistry, Dental Materials Division, Piracicaba Dental School, State University of Campinas, Piracicaba, SP, Brazil

(Received 30 October 2007 and accepted 13 March 2008)

Abstract: This study investigated the influence of ceramic thickness and curing unit on light transmission through leucite-reinforced material and polymerization of a dual-cured resin luting agent. Discs of Empress Esthetic (Ivoclar Vivadent) of 0.7-, 1.4- and 2-mm thickness were prepared. Variolink II (Ivoclar Vivadent) was placed in a 1-mm-thick cylindrical mold, and light-activated through ceramic for 40 s, using QTH or LED units. The samples were divided into dual, light, and chemically-polymerized control groups. Knoop hardness indentations were made on the top and bottom surfaces. Data were subjected to split-plot design three-way ANOVA and Tukey's test ($P < 0.05$). The light spectrum transmitted through ceramic was obtained using a spectrometer. Samples activated through 1.4- and 2-mm-thick discs showed lower hardness than all others groups, except for the chemical control group. Dual and light-polymerized control samples showed similar hardness to those activated through the 0.7-mm ceramic, whereas chemically polymerized control samples showed similar hardness to those activated through 1.4- and 2-mm ceramics. No significant differences in hardness were detected between the curing units or between the top and bottom layers. No significant alteration in the light spectrum profile was observed for both units, irrespective of the ceramic thickness. (J. Oral Sci. 50, 131-136, 2008)

Keywords: ceramic; hardness; light spectrum; luting agent; polymerization.

Correspondence to Dr. Lourenço Correr-Sobrinho, Department of Restorative Dentistry, Dental Materials Division, Piracicaba Dental School, State University of Campinas, Av. Limeira, 901 – 13414-903 – Piracicaba, SP, Brazil
Tel: +55-19-2106-5348
Fax: +55-19-2106-5218
E-mail: sobrinho@fop.unicamp.br

Introduction

In the last decade, various technological advances have led to the development of new materials such as glass-infiltrated, heat-pressed and copy-milled ceramics, exhibiting high strength. Along with the strength of the material, the luting technique is also important for the clinical success of a restoration (1). Adhesive cements are used to improve the fracture resistance of ceramics, allowing more effective stress transfer from the restoration to the supporting tooth (2). Although adhesive luting does not improve the bonding to ceramics with high crystalline content, glass ceramics are acid-sensitive and undergo surface degradation by hydrofluoric acid, thus yielding to a topographic pattern that favors micromechanical bonding.

Resin-based agents are generally used for luting glass-ceramics. These materials are classified according to their activation mode, that is, chemical, photo or dual activation (3). In order to obtain high bond strengths after cementation, optimal luting agent polymerization is required (4). In addition, inadequate curing is associated with poor mechanical properties (5,6). Several investigators have reported on the light attenuation effect promoted by ceramics (4,7-12). The degree of this attenuation depends upon characteristics such as crystalline structure, thickness and shade of the restorative (10,11), which interfere with light transmittance and, as a result, may decrease the total

energy reaching the luting agent.

Furthermore, the light source might also affect the polymerization process. The primary characteristic of light, which interferes with its transmission, is the irradiance level, as ceramics might absorb up to 50% of the light energy (13). However, the light emission profile of different light-curing units (LCUs) may also interfere with the polymerization potential (11). During adhesive cementation procedures, different light sources or thicknesses of the intervening ceramic might affect the polymerization of resin luting agents, and thus interfere with the clinical performance of restorations. Nonetheless, little is known about the influence of different LCUs on the characteristics of the light transmitted through ceramic.

The purpose of this study was to investigate the influence of ceramic thickness and light source on the characteristics of the light being transmitted through a glass ceramic and polymerization of a dual-cured resin-luting agent. The tested hypothesis was that the luting material would present lower hardness values with increasing ceramic thickness, irrespective of the light source.

Materials and Methods

With the aim of creating molds to obtain ceramic specimens with different thicknesses, three cylindrical patterns were made with organic wax (Thowax; Yeti Dentalprodukte, Engen, Germany), invested with phosphate-based material (Esthetic Speed; Ivoclar Vivandent, Schaan, Liechtenstein) and heated at 850°C for 1 h in a ceramic oven (Austromat M; Dekema Dental-Keramiköfen, Freilassing, Germany). The leucite-reinforced glass ceramic IPS Empress Esthetic (Ivoclar Vivadent), shade A3, was heat pressed into the molds, using the EP600 furnace (Ivoclar Vivadent), in accordance with the manufacturer's instructions.

After cooling to room temperature, the ceramic discs were divested with 100-µm glass beads at 2 bar pressure using a microetcher (Danville Materials, San Ramon, CA, USA), and ultrasonically cleaned in water for 10 min (MaxiClean 750; Unique, Indaiatuba, SP, Brazil). Thereafter, the specimens were wet-polished with 120-, 220-, 320-, 400-, 600- and 1200-grit SiC papers (Norton S.A., São Paulo, SP, Brazil) to thicknesses of 0.7, 1.4 and 2 mm.

Figure 1 shows the experimental set-up of the study. Variolink II resin luting agent (Ivoclar Vivadent), shade A3, was tested. The material was placed into a cylindrical elastomer mold (inner diameter 5 mm × thickness 1 mm), which was prepared using polyvinyl siloxane impression material (Express putty; 3M ESPE, St Paul, MN, USA). The dark orange color of the mold impeded light transmittance through it, allowing the luting agent to be exposed to the polymerization light solely from above. A transparent polyester strip was placed over the filled orifice and one of the three ceramic discs was placed on the strip. Before activation, a constant and uniform 250-g cementation load was applied for 2 min, using a custom-made device (4).

Light-activation was conducted through ceramic, for 40 s, using a quartz-tungsten-halogen (QTH) curing unit (XL2500; 3M ESPE) or a recently introduced blue light emitting diode (LED), which presents additional ultraviolet lamps (Ultralume 5; Ultradent Products, South Jordan, UT, USA). The units were connected to an electrical voltage stabilizer, and the light guide tip was placed directly onto the ceramic surface. Since the light guide tip of the LCUs and the ceramic discs did not match in diameter, black adhesive tape was used to define an area that corresponded to the output diameter of the QTH light guide (7 mm) for the ceramic discs and the LED guide.

The output power (mW) of each LCU was measured with a digital power meter (Ophir Optronics Inc., Danvers,

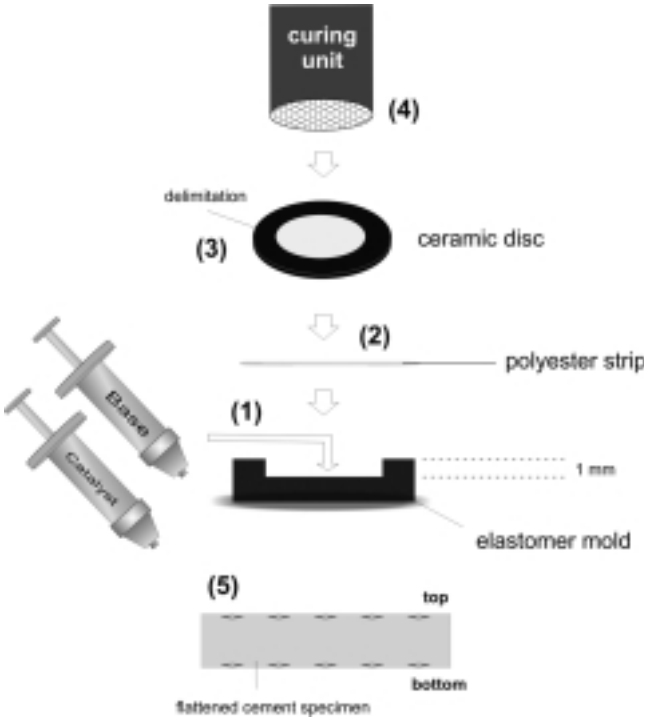

Fig. 1 Experimental set-up of the study. (1) base and catalyst pastes of the resin luting agent were mixed and inserted into the elastomer mold; (2) a polyester strip was placed over the filled mold; (3) a ceramic disc was positioned over the luting material; (4) a light-curing unit placed onto the ceramic surface for light-activation; (5) cross-sectional area of the flattened cement specimen for Knoop hardness readings at both top and bottom layers.

MA, USA). Irradiance (mW/cm^2) was computed as the ratio of the output power by the defined area of the light guide. The characteristics of each LCU are shown in Table 1. In addition to specimens activated through different ceramic thicknesses, dual-cured control specimens were obtained by photo-activation without using ceramic, while photo-cured control specimens were obtained using only the base paste (which contains the photo-initiator) of the luting agent, also irradiated without the intervening ceramic. Chemically-cured control samples were obtained by shielding the material from exposure to both ambient and polymerizing lights.

After preparing the specimens, they were placed in light-proof containers and stored at 37°C, for 24 h. In order to obtain a smooth, planar surface for hardness testing without polishing the surfaces, the specimens were embedded in epoxy resin (Buhler, Lake Bluff, IL, USA) and transversally flattened with 320-, 400-, 600- and 1200-grit SiC papers (Norton S.A.) in a water-cooled automatic polisher (APL-4; Arotec, Cotia, SP, Brazil). Knoop hardness measurements were performed with an indenter (HMV-2; Shimadzu, Tokyo, Japan) under a load of 50 g for 15 s. Five indentations were made on both top and bottom surfaces of the luting agent, as shown in Fig. 1. The average of the five readings was recorded as the Knoop hardness number (KHN) for each layer. In total, 55 samples were tested; that is, five specimens for each curing mode-light source combination.

After performing both normality and equal variance tests, data were analyzed by three-way ANOVA (curing unit × ceramic thickness × luting agent layer), with a split-plot design for comparisons within the same specimen (top × bottom), followed by Tukey's test ($P < 0.05$). Additionally, the spectral distribution of the light transmitted through each ceramic specimen was obtained, for both curing units, using a computer-controlled spectrometer (USB2000; Ocean Optics, Dunedin, FL, USA), and compared with the control (without the intervening material).

Results

Hardness results are shown in Table 2. The statistical analysis showed that the only factor that was significant

Table 1 Characteristics of the light-curing units

Unit	Manufacturer	Irradiance*	Peak of emission**
XL2500 QTH	3M ESPE	905 mW/cm^2	484 nm
UltraLume 5 LED	Ultradent	1585 mW/cm^2	454 nm

*Values confirmed with a digital power meter.
**Data obtained with a computer-controlled spectrometer.

Table 2 Knoop hardness means (standard deviations)

Group	Layer	Light-curing unit*	
		QTH	LED
Dual-cured control	top	45.2 (4.9) ^{A,a}	42.8 (2.6) ^{A,a}
	bottom	45.0 (2.9) ^{A,a}	42.4 (1.9) ^{A,a}
Photo-cured control	top	41.1 (2.3) ^{A,a}	39.8 (2.5) ^{A,a}
	bottom	41.5 (2.6) ^{A,a}	40.1 (1.8) ^{A,a}
0.7 mm ceramic	top	38.8(1.4) ^{A,a}	41.7 (1.0) ^{A,a}
	bottom	38.5 (2.4) ^{A,a}	41.4 (1.7) ^{A,a}
1.4 mm ceramic	top	37.6 (1.6) ^{A,b}	37.2 (3.2) ^{A,b}
	bottom	37.4 (1.2) ^{A,b}	35.7 (3.1) ^{A,b}
2 mm ceramic	top	35.2 (1.8) ^{A,b}	36.1(1.2) ^{A,b}
	bottom	34.8 (1.3) ^{A,b}	34.4 (0.8) ^{A,b}
Chemically-cured control		31.3 (3.4) ^{b}	

Means followed by different capital letters in the same line, and small letters in the same column, were statistically different at $P < 0.05$.
*Five specimens were tested for each curing mode-light source combination.

was 'ceramic thickness' ($P < 0.001$). Samples activated through 1.4- and 2-mm-thick discs showed lower hardness than all others groups ($P < 0.05$), except for the chemically-cured control group. Dual- and photo-cured control samples showed similar hardness to samples activated through the 0.7-mm ceramic, whereas chemically-cured control specimens showed similar hardness to those activated through 1.4- and 2-mm discs. On the other hand, no significant differences were detected between the LED and QTH curing units ($P = 0.609$), and no significant differences were detected between the top and bottom layers of the luting agent ($P = 0.777$), irrespective of the ceramic thickness.

An emission spectrum concentrated in the 420 to 500 nm range, with a peak of emission at 484 nm, was observed for the QTH device (Fig. 2). In contrast, for the LED unit, a narrower spectrum of wavelengths, concentrated in the 440 to 480 nm range, and with a peak of emission at 454 nm, was observed. Additionally, for the LED unit, another curve, concentrated in the 390 to 410 nm range (ultraviolet), was detected. Figure 3 illustrates the light spectrum profiles for the different curing units emitted through the different ceramic thicknesses. When compared with the control profile for each light source, no significant alteration for any thickness was observed; the wavelength peak position on the curve was approximately the same.

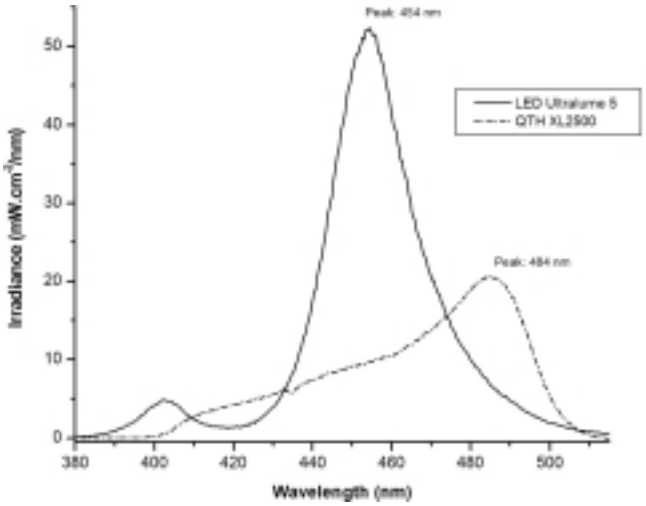

Fig. 2 Light spectrum profiles. For the QTH unit, an emission curve concentrated in the 420 to 500-nm range, with a peak of emission at 484 nm, was observed. For the LED unit, a narrower curve concentrated in the 440 to 480-nm range, with a peak of emission at 454 nm, and another curve in the ultraviolet range were detected.

Discussion

The present results showed no significant differences in the polymerization potential of both LED and QTH curing units, despite the different radiant exposures of the units (40 and 28 J/cm^2, respectively). Ozturk et al. (14) described similar outcomes for degree of conversion and surface hardness when assessing QTH and LED lights with different output irradiances. This result indicates that the light energy differences for the photo-activation simulations tested here were not great enough to yield significant differences in hardness. In addition, this suggests that the development of hardness in dual-cured luting agents is not dependent on the light source, as long as the irradiance level for the wavelength region effective to activate the photo-initiator is similar.

However, the tested hypothesis was only partially accepted, as the groups light-activated through 1.4- and 2-mm-thick ceramics showed similar hardness values between themselves but significantly lower values than all

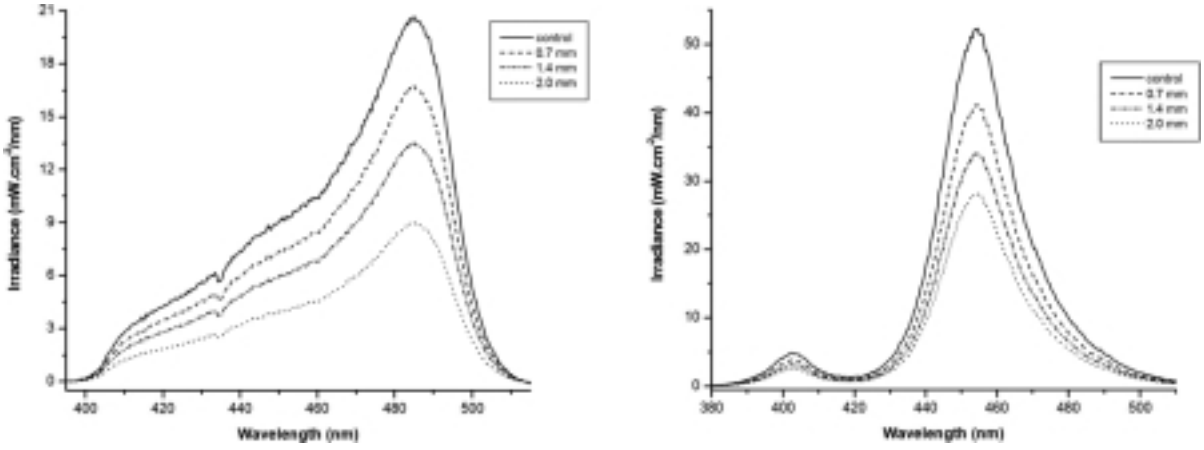

Fig. 3 Light spectrum profiles for indirect exposure. No significant alteration was observed in the emission profile for activation through ceramic when compared with the control profile, regardless of the unit or the ceramic thickness.

other groups, except the chemically-cured group. This finding is probably related to the light attenuation promoted by the intervening material. The lower light energy might affect the polymer development primarily by decreasing the double bond conversion, since the polymerization process is dependent on the radiant exposure delivered to samples (6). The lower the dose of energy reaching the luting material, the lower the degree of conversion and hardness.

Moreover, as indirect activation reduces the level of irradiance reaching the luting material, the polymer network development might be affected not only by decreasing the monomer conversion, but also by interfering with the type and degree of cross-linking (15). High-intensity lights may favor the formation of more densely cross-linked networks (15) by generating a multitude of polymer growth centers. Therefore, more densely cross-linked polymers would disclose higher hardness outcomes. Nonetheless, the group in which the luting agent was cured through a 0.7-mm-thick ceramic showed similar findings in both photo and dual-cured control groups. This is probably related to an insignificant light attenuation effect promoted by the thin intervening material, leading to similar conversion of double bonds and cross-linking.

The top and bottom layers of the luting agent showed similar hardness, and this is probably related to the light exposure procedures. During activation through the 0.7-mm ceramic disc, or without using ceramic, sufficient light energy was probably delivered to both surfaces of the specimens and, consequently, no significant influence in hardness occurred; another potential explanation is related to an additional chemical curing effect. In addition, no significant differences were detected between the top and bottom layers for the photo-cured control group. However, for the groups light-activated beneath thicker ceramic specimens, no significant differences between the top and bottom layers were observed either. This might also be the result of the additional chemical curing effect that compensates for the lower light energy reaching the bottom layer. Jung et al. (8), using the same luting material tested here, reported that the use of self-curing catalyst always increased the hardness values, irrespective of the light source or the ceramic thickness.

Similar hardness outcomes were observed for the photo- and dual-cured control groups, and both were significantly harder than the chemically-cured group. This outcome suggests that dual agents depend on light exposure to achieve enhanced properties. Indeed, it has been reported that the self-curing component itself is not enough to ensure high hardness (7). However, it should be considered that, for self-cured resins, the time needed for the chemical reaction to take place might be up to 10 min. Therefore, it could be speculated that the immediate exposure to light and formation of cross-linked polymer chains might have interfered with the chemical curing, because a large number of self-polymerization promoters would be entrapped in the polymeric network, thus being unable to respond to the chemical curing. However, this effect is not yet recognized in literature and needs further investigation.

With regard to the spectral emission of the LCUs, no significant influence of the ceramic material was detected, irrespective of its thickness. However, when comparing the spectral profile between the light sources, the LED unit showed a narrower spectrum of wavelengths and a different peak of emission, while also presenting another curve of emission concentrated in the ultraviolet range of the electromagnetic spectrum. In spite of this, in the present study, no significant differences between the light sources were detected. The irradiance level for the wavelength region effective to activate camphorquinone (around 468 nm) was similar for the two LCUs, which may have resulted in the similar hardness values.

Empress Esthetic used in this study, is a recently introduced, leucite-reinforced glass ceramic with increased translucency, smaller grain size, and leucite crystals distributed in a more homogeneous mode than its predecessor, Empress (16). Although little is know about the transmittance characteristics of both materials, the outcome of the present study indicates that Empress Esthetic allowed irradiance transmission without interfering with the wave properties of the light. However, this result should be restricted to this ceramic; different outcomes might be observed for less translucent materials.

In summary, the present results showed that ceramic thickness is a critical factor for the development of hardness in indirectly activated dual-cured resin luting agents. Although it is difficult to predict whether different clinical performances are likely to occur for restorations luted under similar conditions to those tested here, the use of high-intensity light sources or increasing the light exposure time is advisable when cementing thick ceramic restorations. However, the results of the present study did not take into account the effects that thermal changes and mechanical fatigue might present in the long-term performance of restorations. In addition, different irradiance levels of the curing devices, as well as different shades and opacities of the intervening restorative, might interfere with the hardness development of resin luting materials. Furthermore, it should be considered that the clinical success of luting agents depends not only on their mechanical properties, but also on their handling properties, bond strength to the tooth and restoration, film thickness,

and color stability.

In conclusion, polymerization of the dual-cured luting agent was dependent on the ceramic thickness, while the light source showed no significant effect. The indirect activation presented no significant effect on the characteristics of the light being transmitted through ceramic.

Acknowledgments

This study was supported in part by Fundação de Amparo à Pesquisa do Estado de São Paulo – FAPESP (Grant 06/53566-8) and Conselho Nacional de Desenvolvimento Científico e Tecnológico – CNPq (Grant 308128/2006-0), Brazil.

References

1. Malament KA, Socransky SS (2001) Survival of Dicor glass-ceramic dental restorations over 16 years. Part III: effect of luting agent and tooth or tooth-substitute core structure. J Prosthet Dent 86, 511-519
2. Groten M, Probster L (1997) The influence of different cementation modes on the fracture resistance of feldspathic ceramic crowns. Int J Prosthodont 10, 169-177
3. Rosenstiel SF, Land MF, Crispin BJ (1998) Dental luting agents: a review of the current literature. J Prosthet Dent 80, 280-301
4. Moraes RR, Correr-Sobrinho L, Sinhoreti MA, Puppin-Rontani RM, Ogliari FA, Piva E (2008) Light-activation of resin cement through ceramic: relationship between irradiance intensity and bond strength to dentin. J Biomed Mater Res B Appl Biomater 85B, 160-165
5. Danesh G, Davids H, Reinhardt KJ, Ott K, Schäfer E (2004) Polymerisation characteristics of resin composites polymerised with different curing units. J Dent 32, 479-488
6. Halvorson RH, Erickson RL, Davidson CL (2002) Energy dependent polymerization of resin-based composite. Dent Mater 18, 463-469
7. el-Mowafy OM, Rubo MH, el-Badrawy WA (1999) Hardening of new resin cements cured through a ceramic inlay. Oper Dent 24, 38-44
8. Jung H, Friedl KH, Hiller KA, Furch H, Bernhart S, Schmalz G (2006) Polymerization efficiency of different photocuring units through ceramic discs. Oper Dent 31, 68-77
9. Jung H, Friedl KH, Hiller KA, Haller A, Schmalz G (2001) Curing efficiency of different polymerization methods through ceramic restorations. Clin Oral Investig 5, 156-161
10. Soares CJ, da Silva NR, Fonseca RB (2006) Influence of the feldspathic ceramic thickness and shade on the microhardness of dual resin cement. Oper Dent 31, 384-389
11. Rasetto FH, Driscoll CF, Prestipino V, Masri R, von Fraunhofer JA (2004) Light transmission through all-ceramic dental materials: a pilot study. J Prosthet Dent 91, 441-446
12. Tango RN, Sinhoreti MAC, Correr AB, Schneider LFJ, Kimpara ET, Correr-Sobrinho L (2007) Knoop hardness of dental resin cements: effect of veneering material and light curing methods. Polymer Testing 26, 268-273
13. Brodbelt RH, O'Brien WJ, Fan PL (1980) Translucency of dental porcelains. J Dent Res 59, 70-75
14. Ozturk N, Usumez A, Usumez S, Ozturk B (2005) Degree of conversion and surface hardness of resin cement cured with different curing units. Quintessence Int 36, 771-777
15. Schneider LF, Moraes RR, Cavalcante LM, Sinhoreti MA, Correr-Sobrinho L, Consani S (2008) Cross-link density evaluation through softening tests: effect of ethanol concentration. Dent Mater 24, 199-203
16. Bühler-Zemp P (2004) IPS Empress Esthetic scientific documentation. Ivoclar Vivadent AG, Schaan, 3-5