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Effect of lithium disilicate veneers of different thickness on the degree of conversion and micro-hardness of a light curing and a dual curing cement

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Abstract

Aim: Various materials and systems to bond lithium disilicate to the tooth substrate are available to clinicians, who can adapt the material to each clinical situation to maximize the performance of indirect aesthetic restorations. This study aimed to evaluate the degree of

conversion (DC) and the microhardness (MH) of a dual- and a light-curing cement under lithium disilicate discs of different thicknesses.

Methods: 48 Lithium Disilicate (E-Max CAD) samples were prepared and divided into three groups (n=16) according to the thickness: A) 0.6mm; B) 1mm; C) 1.5mm. Each group was, further, divided into two subgroups (n=8) according to the resin cement employed: 1) NX3 (Kerr); 2) Choice2 (Bisco). A standardized quantity of cement was placed on sample and DC was evaluated with ATR FT-IR spectrophotometer (Thermo Scientific Nicolet IS10). 24h after DC was established, a Vickers Test was performed on the cement with a micro-indentometer (Leica Microsystems S.p.A., Milano, Italy). Results were statistically analysed with ANOVA test and significance was set for $p < 0,05$.

Results: Statistical analysis showed a significant influence of the cement type ($p=0,005$) on DC. MH results are influenced by thickness only between 0,6 mm and 1,5 mm when light-cured cement was employed

Conclusions: both the light-curing and the dual-curing cement reached comparable DC between 0,6 and 1,5 mm. However, the light-curing resin showed a higher DC and MH.

1. Introduction

Ceramic system technology has advanced quickly in recent years, and has become a valid option in the restoration of anterior and posterior teeth that require indirect prosthetic rehabilitations. The clinical success of ceramics is mainly due to its reliable bonding to dental hard tissues provided by luting materials [1, 2]. Moreover, the greater attention given to preserving sound dental tissue has contributed to the success of adhesive ceramic

restorations, especially on anterior teeth [3].

Among ceramic systems, lithium disilicate has gained popularity for anterior and posterior fixed full and partial restorations because of its physical properties[4]. In fact, while high-strength nonsilica-based ceramic substructure materials, such as alumina or zirconia, have high opacity and require translucent veneering porcelain to achieve adequate shade matches, lithium disilicate is a silica-based adhesive material that guarantees not only superior aesthetics and translucency, but also strength, wear resistance, and chemical durability [5]

Various materials and systems to lute lithium disilicate to the tooth substrate are available to clinicians, who can adapt the material to each clinical situation to maximize the performance of indirect aesthetic restorations. An important requirement for an ideal luting agent is its ability to provide superior mechanical properties to resist functional forces over the lifetime of a restoration [6]. Adequate polymerization is crucial to obtain optimal physical properties and high clinical performance of resin materials. As a result of suboptimal polymerization, a low monomer-polymer conversion rate with a higher residual quantity of double bonds is obtained, causing inferior physical properties and increased water sorption and solubility [7]. Various factors may affect resin polymerization, and as a consequence, may also affect the choice of the cement, such as the optical properties, the resin cement activation mode, the light curing unit characteristics [8], and the thickness of the material employed [9]. Lithium disilicate veneer cementation may be performed using either light-curing or dual-curing activation. Light-curing cements have a polymerization mechanism that only allows material setting in the presence of a light source that activates photo-initiators and starts the polymerization reaction. A great advantage of these materials

is their ease of use due to their set-on-command and unlimited working time [10]. However, the absence or attenuation of light irradiance caused by the thickness, shade, and low translucency of the ceramic material could reduce the quality of the polymerization reaction [11]. Thus, dual-cure cements were introduced to attempt to overcome this problem. In fact, these materials combine a light-curing mechanism of polymerization with self-curing components that initiate the polymerization reaction in the absence of light [11, 12]. However, even if the polymerization process does not require a light source, allowing a uniform set of materials [10], dual-cure materials require a setting reaction slow enough to allow sufficient working time, but quick enough to permit finishing of the restoration [10], because the polymerization reaction is not controllable from the moment the base and the catalyst paste are mixed together and the polymerization starts. Moreover, the deficiency of chemical-cure components can result in a higher concentration of unreacted double bonds, lower hardness, and higher solubility of cements, which can influence chemical stability in the oral environment.[13]

The purpose of this *in vitro* study was to evaluate the degree of conversion (DC) and the micro-hardness (MH) of a dual-cure and light-cure cement under lithium disilicate discs of different thicknesses which intended to simulate anterior veneers. The null hypotheses tested were that ceramic thickness does not affect (1) the DC or (2) the MH of the tested cements regardless of the cement-curing mode (light-cure vs. dual-cure).

2. Materials and Methods

2.1 Specimen preparation

Forty-eight Lithium Disilicate core drilled (IPS e.max CAD for CEREC and inLab LOT. R37085, Ivoclar) discs (1 cm in diameter, A2 shade LT) were equally divided into

three groups (n=16) according to the thickness of the material: Group A: 0.6 mm; Group B: 1 mm; Group C: 1.5 mm. Precision of discs thickness was checked with a digital caliper and discs with a discrepancy of more than 0,1 mm were excluded. A further group without ceramic, group D, was also considered as a control group. Each group was then randomly divided into two subgroups (n=8) according to the luting cement employed. Samples of subgroup 1 were prepared with NX3 dual-curing cement (Kerr Co, USA); samples of subgroup 2 were prepared with Choice2 light-curing cement (Bisco Inc., Shaumburg, IL, USA) (Table 1).

	Composition
NX3 (Kerr Co, USA)	<ul style="list-style-type: none"> • BisGMA, UDMA, EBPADMA, and TEGDMA • Proprietary monomers (GPDM) • Proprietary redox initiator • Camphorquinone (CQ)-based photo-initiator • Stabilizers including UV stabilizer • Bariumaluminosilicate glass filler • Nano-sized ytterbium fluoride filler • Colloidal silica <p>Filler by weight 67.5 % (Dual Cure version)</p> <p>Filler by volume 43.3 % (Dual Cure version)</p>
Choice2 (Bisco Inc., Shaumburg, IL, USA)	<ul style="list-style-type: none"> • Strontium Glass – concentration range <75% • Amorphous Silica – concentration range <25% • BisGMA– concentration range <10%

Table 1: Cement Composition

One side of each ceramic disc was etched with 5% hydrofluoric acid (IPS Ceramic Etching gel, Ivoclar Vivadent) for 20 s, then rinsed with tap water and immersed in alcohol in an ultrasonic bath for 5 min. Silane (Silane Primer, Kerr Co, USA) was applied to the etched surface, air-dried for 15 sec, and covered with a coat of bonding resin after 30 s (Optibond FL adhesive system, Kerr Co, USA) using a microbrush and thinned with air.

As regard group D, no adhesive procedures were performed, and cement was considered without ceramic apposition.

2.2 Degree of conversion measurement.

A ca.170 μm [14] thick plastic guide with a center hole 1 mm in diameter was placed on the diamond support of an ATR FT-IR (Attenuated Total Reflectance Fourier Transformed Infra Red) spectrophotometer (Thermo Scientific Nicolet IS10) to standardize a layer of luting cement between the sample surface and the FT-IR light beam. The luting cement was applied on the bonded surface of each specimen placed on the FT-IR light beam. The excess cement was eliminated, thereby creating a pressure that simulated the clinical cementation of indirect veneers until the disc contacted the plastic guide. For group D, cement was placed on the FT-IR diamond and thickened with a transparent Mylar strip. Polymerization of the cement was performed using a high power poly wave LED lamp (Valo-Ultradent South Jordan UT USA) for 60 s at 1400 mW/cm^2 , with the curing tip contacting the center of the discs and the light beam opposite to the cement layer.

The surface analysis was performed in ATR mode, in which the IR beam penetrated 1 μm into the material. The FT-IR spectra of the curing process were recorded every 2 s

$$\left. \begin{array}{c} \text{(C=C aliphatic/C=C aromatic) Polymer} \\ \text{---} \\ \text{(C=C aliphatic/C=C aromatic) Monomer} \end{array} \right\} *100$$

with a range between 4000-525 cm^{-1} and a resolution of 6 cm^{-1} . The spectra recorded immediately before activation of the poly wave LED lamp and 10 min after light exposure were fitted and used to evaluate the degree of conversion (DC) of the two tested materials. To determine the percentage of the remaining unreacted double bonds, the DC was assessed as the variation of the absorbance intensities peak height ratio of the methacrylate carbon double bond (peak 1634 cm^{-1}) related to an internal standard of aromatic carbon-carbon double bonds (peak 1608 cm^{-1}) before and after curing of the specimen, according to the following equation [15]:

$$\text{DC\%} =$$

2.3 Microhardness measurement

Twenty-four hours after DC evaluation, microhardness (MH) was measured using a Leica VMHT microhardness tester (Leica Microsystems S.P.A., Milano, Italy) equipped with a Vickers indenter, at exactly the same location at which DC was analyzed by the FT-

IR light beam. A pyramidal diamond indentation was obtained with a load of 100 g for 15 s. Three indentations were obtained for each specimen, and the mean value was considered for the statistical analyses. No indentations were for group D, without ceramic apposition.

2.4 Statistical Analysis

To evaluate the effect of lithium disilicate thickness (0.6–1.0 mm and 1.5 mm), luting materials, and their effects on DC a two-way ANOVA was performed. To consider the effect of thickness on Vickers MH one-way ANOVA test and Bonferroni *post-hoc* were performed. The significance level was set at 95% ($p < 0.05$). All statistical analyses were performed using the Stata software package (StataCorp, 4905 Lakeway Drive, College Station, Texas 77845 USA).

3. Results

The mean and standard deviation values for DC obtained from the different subgroups are expressed in Table 2, while MH mean values and standard deviations are shown in Table 3.

Considering DC%, two-way ANOVA showed that only the cement factor significantly influenced the results ($p < 0,05$), whereas the thickness of the ceramic specimens and the interaction between the two factors had no significant effect. Light-curing cement performed significantly better than dual cement ($p < 0,05$). Moreover, the presence of a lithium disilicate disc with a thickness between 0,6 and 1,5 mm did not reduce DC% both for the light-curing and the dual-curing cement.

For Vickers MH ANOVA showed that within the same cement thickness influenced

hardness values only between 0,6 mm and 1,5 mm of the light-cured cement.

DC%	Dual Cement	Light-Curing Cement
0.6 mm	54.6 ±2.1 ^{aa}	60.9 ±5.3 ^{aa}
1.0 mm	42.7 ±12.5 ^{aa}	58.4 ±4.4 ^{aa}
1.5 mm	47.4 ±16.2 ^{aa}	53.4 ±7.2 ^{aa}
Control group	53.9 ±10.3 ^{aa}	56,26 ±2.31 ^{aa}

Table 2: Degree of Conversion and standard deviation of light curing and dual curing cements. Different superscript lower-case letters (in rows) indicate statistical differences between cements ($p < 0.05$). Different superscript upper-case letters (in columns) indicate statistical differences between different thicknesses within each material ($p < 0.05$).

MH	Dual Cement	Light-Curing Cement
0.6 mm	35.8 ±4.6 ^a	61.1 ±14.3 ^a
1.0 mm	31.3 ±2.8 ^a	57.7 ±4.9 ^{ab}
1.5 mm	36.3 ±10.5 ^a	52.2 ±6.6 ^b

Table 3: Microhardness of and standard deviation light curing and dual curing cements. Different superscript letters indicate statistical differences between different thicknesses within each material (in columns) ($p < 0.05$).

4. Discussion

The longevity of indirect adhesive restorations depends mainly on the quality of the dental-cement restoration interface [11,16]. To reach optimal physical and mechanical properties of composite resin cements under ceramic restorations, the conversion rate should be as high as possible [6, 17]. The method used in this study to assess the DC was

the Fourier Transformed Infrared Spectroscopy (FTIR), a well-established technique that allows direct quantification of unreacted C=C in a resin matrix [6, 18-20].

The present investigation evaluated the effect of lithium disilicate thickness on the degree of conversion of light-cure and dual-cure cement. Several authors have affirmed that the thickness and shade of the restorative material above the cement may affect light transmission and consequently, the DC [21]. To conduct this study, shade and translucency of specimens were standardized, and the curing process was performed using a continuous light application with a poly wave LED lamp during the entire irradiation time at an intensity of 1400 mW/cm.²

The results obtained in this *in vitro* study support the first null hypothesis, because ceramic thickness did not affect DC values within the same group. The only factor that influenced the quality of polymerization was the material used with the light-cure cement, which yielded a significantly higher DC than the dual-cure cement.

Thickness of the lithium disilicate of 0.6 mm and 1.5 mm had no effect on the DC% of the two cements tested. Previous studies reported controversial findings on this topic. Zhang et al. [22] concluded that ceramic thickness greatly influences polymerization quality, while other authors found that only a thickness of more than 2 mm drastically reduces the degree of conversion either of dual-curing or light-curing resins [9,23,24]. Thus, to reach proper polymerization, curing time should be prolonged beyond the manufacturer's recommendation when a 2 mm thick indirect restoration is cemented. The lithium disilicate thicknesses tested in the present study intended to simulate a ceramic veneer with a thickness between 0.6 and 1.5 mm; the results showed that such a thick layer of lithium disilicate did not significantly attenuate the curing light.

Our findings also support the hypothesis that immediate photo-activation of the dual-cure resin based material may compromise the final degree of conversion, as recently reported in a study conducted by Pereira et al. [14]. The authors also reported that dual-curing resin cements have different polymerization kinetics, and that the extent of polymerization changes considerably among different cements. In particular, the moment of light activation determines the formation of the polymer structure, and consequently, determines the structural integrity of the materials. These results agree with those of a study conducted by Faria-e-Silva et al. [25] who also hypothesized that light activation may negatively affect the self-curing mechanism. The rationale is that the rapid formation of a cross-linked polymer after light exposure would lead to entrapment of the reactive species, including activators and initiators needed for the self-cure reaction. Conversely, the findings of the present study contrasted with those of a previous report that showed that only the thickness of the indirect restoration affected the DC of the luting materials (two dual-cure cements and a conventional microhybrid resin composite) [22]. Such controversial findings compared to the present paper could be attributed to the different light-curing materials tested. Moreover, the different thickness and nature of the material employed as indirect restoration could have strongly influenced the DC [25], mostly for light-curing resin composites. In addition, Acquaviva et al. [23] evaluated DC through Raman Spectroscopy 24 h after light curing activation, whereas in the present study, the DC was assessed 10 min after the start of light source irradiance. In addition, in the present study, curing started when cementation procedures were completed and the cement excesses were removed, as takes place *in vivo* during ceramic veneers luting procedures [24].

Deficient polymerization of the resin cement negatively affects its physical and

mechanical properties [12]. However, longevity of a resin cement is influenced, not only by the polymerization degree, but also by the chemical composition of the material itself.

Surface microhardness of a restorative resin is one of the most important parameters for assessing physical properties of dental materials, and is defined as the resistance of a material to indentation or penetration. In the literature, microhardness is commonly used as a simple and reliable method for indirectly estimating the degree of conversion of resin-based cements [26, 27] Although it is generally thought that hardness is directly related to DC percentages [10], the findings of the current study confirmed that other variables also influence the surface hardness of a material. Our statistical analysis revealed significant influence on the results, either by the material or by the interaction between the material and thickness variables. These results are in accordance with those of a study by Tantbirojn et al. [28] who stated that microhardness data are comparable only within the same resin system, since they are not linearly correlated with the degree of cure if compared across different materials.

However, within the same cement, the evaluation of thickness influence on MH was taken into consideration and lead to partially refuse the null hypothesis since only light-cure cement MH is influenced by lithium disilicate thickness. The generally significantly better Vickers hardness values of the light-cure cement regard the dual-cement could be explained by an intrinsic characteristic of the material such as its filler load, filler type, resin matrix, or formulation [24, 25, 29, 30]. The filler particles incorporated into the matrix, influence the mechanical properties more than the matrix itself. Therefore, up to a certain limit, a higher filler load may be expected to improve the mechanical properties [10]. These results are partially in contrast with those of an *in vitro* study conducted by Hofmann et al. [10] in

which dual-cure materials showed better mechanical properties than photo-activated ones, particularly when irradiated through 2.5 mm of leucite-reinforced glass-ceramic. Several studies have shown that the critical thickness of ceramic for a proper curing process is 2 mm or more [9, 23, 24], whereas the present study tested lower thicknesses. Moreover, Hoffmann et al. [10] tested dual-curing cements in both a dual-activation and light-activation mode without mixing the base and the catalyst paste, thus altering the curing process and the intrinsic nature of the dual curing material. In the present study, light-curing cement was compared to a dual-curing one. On the other hand, a study published in 1995 by El-Badrawy & El-Mowafy [30] studied the setting of three dual-cured cements under resin composite inlays, and reported that chemical curing did not completely harden the cements when light was attenuated by tooth and restoration material, which could account for the better performance of the light-curing cement. Furthermore, the composition of the two luting resins tested in the current study suggested that the dual-curing cement (NX3, Kerr Co, USA) has a lower filler content (about 60%) than the light-curing one (about 78%) (Choice2, Bisco, Inc), which could have strongly influenced the material's hardness. Moreover, the light activation of a material with a dual mechanism of conversion may have influenced the polymeric network cross-link density and, consequently, the material mechanical properties.

Conclusion

Within the limitations of this *in vitro* study, both the light cure and the dual cure cement used to lute lithium disilicate veneers with a thickness between 0.6 and 1.5 mm yielded a sufficient polymerization level. Further, the light-cure and the dual cure resins reach a comparable DC%.

Higher MH values obtained with the light-curing resin and the influence of disilicate thickness only in this group of samples may have been the results of a different structure and composition of the two tested materials.

Further studies are needed to validate these results, especially considering the great variability among cements' chemical formulations.

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