

Physical evaluation of the luting systems for veneer ceramic after aging

Avaliação física dos sistemas de cimentação para faceta cerâmica após envelhecimento

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ABSTRACT

The aim of this *in vitro* study was evaluated the amount of color change and degree of conversion of the luting systems for ceramic veneer cementation. Forty-eight human incisors were randomly divided into six groups (n=8) according to the bonding procedure: RelyX Veneer/Single Bond 2; RelyX ARC/Single Bond 2; Filtek Z350 XT Flowable /Single Bond 2; Variolink Veneer/Tetric N-Bond; Variolink II/Tetric N-Bond; Tetric N-Flow/Tetric N-Bond. Ceramic veneers (IPS Empress Esthetic) were cemented using the different bonding procedures. Color differences of the ceramic surface after cementation and 300 h(150kJ/m²) of UV aging test, were examined with a colorimeter. Fourier transform infrared spectroscopy (FT-IR) was used to evaluate the degree of conversion for each luting system. Data were analyzed with one-way ANOVA and Tukey's test ($\alpha=0.05$). All the luting systems demonstrated significant changes in color stability. The ΔE of the systems ranged from 6,11 to 9,33. Tetric N-Flow and RelyX Veneer showed the highest degree of conversion, while lowest values were found for RelyX ARC. All luting systems demonstrated clinically unacceptable ($\Delta E \geq 3.3$) color changes. Despite to the dual cure of the RelyX ARC luting cement, it showed the lowest degree of conversion values, while Tetric N-Flow and RelyX Veneer presented the highest and similar degree of conversion.

Keywords: color, dental veneers, aging, resin cements.

RESUMO

O objetivo deste estudo *in vitro* foi avaliar a quantidade de mudança de cor e o grau de conversão dos sistemas de cimentação para a cimentação de facetas de cerâmica. Quarenta e oito incisivos humanos foram divididos aleatoriamente em seis grupos (n = 8), de acordo com o procedimento de ligação: RelyX Veneer / Single Bond 2; RelyX ARC / ligação simples 2; Filtek Z350 XT Bond / escoamento simples 2; Folheado Variolink / N-Bond Tetric; Ligação N Variolink II / Tetric; N-Flow Tétrico / N-Bond Tétrico. As facetas de cerâmica (IPS Empress Esthetic) foram cimentadas usando os diferentes procedimentos de colagem. As diferenças de cor da superfície cerâmica após cimentação e 300 h (150kJ / m²) do teste de envelhecimento por UV foram examinadas com um colorímetro. A espectroscopia no infravermelho por transformada de Fourier (FT-IR) foi usada para avaliar o grau de conversão de cada sistema de cimentação. Os dados foram analisados com ANOVA one-way e teste de Tukey ($\alpha = 0,05$). Todos os sistemas de cintagem demonstraram mudanças significativas na estabilidade da cor. A ΔE dos sistemas variou de 6,11 a 9,33. O N-Flow Tetric e o RelyX Veneer apresentaram o maior grau de conversão, enquanto os menores valores foram encontrados para o RelyX ARC. Todos os sistemas de cimentação demonstraram alterações de cor clinicamente inaceitáveis ($\Delta E \geq 3,3$). Apesar da cura dupla do cimento de cimentação, RelyX ARC apresentou os menores valores de conversão, enquanto o Tetric N-Flow e o RelyX Veneer apresentaram os maiores e similares graus de conversão.

Palavras-chave: cor, facetas dentárias, envelhecimento, cimentos resinosos.

1 INTRODUCTION

With the development of the adhesive dentistry, resin luting strategies have been widely used to bond indirect restorations to tooth structure. However, the color stability of these materials can be influenced by a variety of extrinsic and intrinsic factors^{1,2}. The extrinsic factors correspond to the absorption of stain from exogenous sources related to hygiene habits, smoking and food³. While the intrinsic factors involve alterations in the percentage of remaining C=C bonds, composition of resin matrix and type of photoinitiator system⁴.

Camphorquinone is the most commonly used photoinitiator in restorative dentistry. Even when it is used in small amounts, it significantly influences the material's color⁵. Other very important components of photoinitiator systems are the tertiary amines, which can be aromatic or aliphatic, and so-called synergists or accelerators⁵. All amines are known to form products during photoreaction, which tend to cause yellow to red/brown discolorations under the influence of light or heat⁶. Because of this color change of the luting systems can become visible, affecting the esthetic appearance of final indirect restorations⁷.

There are three options for luting ceramic veneers: the dual-cured cements that associate some of the desirable characteristics of light- and chemical-cured resin cements⁸ as longer working time and effective polymerization⁹, however, because it contains aromatic tertiary amine in its composition, which can compromise the color stability of indirect restorations over clinical life⁹. Light-cured resin cements have advantages in their color stability and working time⁹, but less effective polymerization as a disadvantage. An alternative material to replace these luting materials is the flowable resin composite¹⁰ that showed the benefits of lower cost despite to the disadvantage of lower range of colors¹⁰.

Thus, the objective of the study is to evaluate the amount of color change and degree of conversion of the luting systems for ceramic veneer cementation after accelerated aging.

2 MATERIALS AND METHODS

Ethics committee

The study was submitted and approved by the review board of Ethics at Piracicaba Dental School- University of Campinas, under the protocol number 078/2012.

Sample preparation and experimental groups

Freshly extracted human incisors (n=48) were cleaned and stored in 0.2% thymol solution at 37°C. 0.6-mm-deep walls were prepared on the labial enamel surfaces of the teeth with a high-speed handpiece and around diamond bur #2135 (KG Sorensen) under water cooling. The diamond bur was discarded after 5 preparations. Ceramics veneers were fabricated with 0.6 mm thickness using IPS Empress Esthetic (Ivoclar Vivadent, Schaan, Liechtenstein) according to the manufacturer's recommendations. On each ceramic veneer, the area designated for contact with the luting system was prepared with 10% hydrofluoric acid gel (FGM Dental Materials, Joinville, SC, Brazil) in gel, applied for 20 seconds, then rinsed with water and gently dried with air. After, the specimens were silanized with a monocomponent silane (RelyX Ceramic Primer- 3M ESPE, St. Paul, MN, USA) for 1 min. The teeth were divided into six experimental groups (n=8) according to the different luting systems: (SB/RVE) Single Bond 2/RelyX Veneer (3M ESPE); (SB/ARC) Single Bond 2/RelyX ARC (3M ESPE); (SB/FFL) Single Bond 2/Filtek Z350 XT Flowable (3M ESPE); (TB/VVE) Tetric N-Bond/Variolink Veneer (Ivoclar-Vivadent); (TB/VAR) Tetric N-Bond/Variolink II (Ivoclar-Vivadent); (TB/TFL) Tetric N-Bond/Tetric N-Flow (Ivoclar-Vivadent).

The prepared teeth were acid-etched with 37% phosphoric acid (Scotchbond Etchant, 3M ESPE) for 15 seconds. The tooth surface were washed and gently dried in preparation for application of the adhesive system. The luting systems were manipulated according to the manufacturer's specifications (Table 1). The ceramic veneers were luted with one of the luting systems and light-cured directly on the ceramic using visible light Demetron LC (sds Kerr, Middleton, USA) for 40 seconds. All the test specimens were stored in isolated receptacles at 37°C under high-humidity and kept in the absence of light for 24 hours until they were submitted to color analysis.

Table 1: Procedures for cementing systems adhesion

Luting Systems	Procedures for cementing
Single Bond 2/ RelyX Veneer (SB/RVE)	1-Acid etching of enamel with 35% phosphoric acid (15s) and washing with distilled water for 15s. 2-Application of two consecutives layer of adhesive system on enamel; 3-Mild air stream 4-Resin cement application in ceramic surface and enamel; 5-Light curing for 40 s.
Single Bond 2/ RelyX ARC (SB/ARC)	1-Acid etching of enamel with 35% phosphoric acid (15s) and washing with distilled water for 15s. 2-Application of two consecutives layer of adhesive system on enamel; 3-Mild air stream 4-Mix of the resin cement (10s); 5- Cement application in ceramic surface and enamel; 6-Light curing for 40 s.
Single Bond 2/ Filtek flowable (SB/FFL)	1-Acid etching of enamel with 35% phosphoric acid (15s) and washing with distilled water for 15s. 2-Application of two consecutives layer of adhesive system on enamel; 3-Mild air stream;4-Application of the flowable composite; 5-Light curing for 40 s.
Tetric N-Bond/ Variolink Veneer(TB/VVE)	1-Acid etching of enamel with 35% phosphoric acid (15s) and washing with distilled water for 15s. 2-Application of two consecutives layer of adhesive system on enamel, mild air stream 3-Resin cement application in ceramic surface and enamel; 4-Light curing for 40 s.
Tetric N-Bond/ Variolink II (TB/VAR)	1-Acid etching of enamel with 35% phosphoric acid (15s) and washing with distilled water for 15s. 2-Application of two consecutives layer of adhesive system on enamel, mild air stream; 3-Mix of the resin cement pastes (base+catalyst for 15s); 4- Resin cement application in ceramic surface and enamel; 5-Light curing for 40 s.

Color measurement

CIEL*a*b* co-ordinates (L*, a*, b*) (CIE, 2004) were recorded for specimen pre- and immediately post-aged by means of a spectrophotometer (Easyshade, VITA Zahnfabrik, BadSackingen, Germany) with a D65 illuminant. A white (L* = 92.38, a* = 0.48, b* = 2.89) ceramic tile was used as background for the color analysis. Differences in each inherent color parameter were determined as ΔL^* , Δa^* , and Δb^* subtracting each pre- and immediately post-aged co-ordinate parameter values¹¹. Color stability was evaluated by color variation (ΔE) using the formula: $\Delta E = [(L_f - L_i)^2 + (a_f - a_i)^2 + (b_f - b_i)^2]^{0.5}$. Values of ΔE values below 3,7 were considered clinically acceptable¹².

Accelerated artificial aging

The specimens were submitted to accelerated artificial aging (AAA) using an UV-

accelerated aging chamber (EQUV, Equilam Ind Com Ltda, São Paulo, SP, Brazil). The standardized conditions of AAA consisted of 4h of exposure to UV-B light at 50°C and 4 h of water storage at 50°C for a period of 300h, which corresponded to six months of clinical use¹³.

Degree of conversion

The degree of conversion (DC) was determined using a FT-IR spectrometer (Spectrum 100, Perkin-Elmer Corp, Norwalk, CT, USA). The uncured resin cement was placed between two disks and the spectrum were recorded with 32 scans at a resolution of 4cm⁻¹ in the 1590 a 1660cm⁻¹ band. After the IR spectral scan, the material was cured between the transparent disks for 40 seconds with a Demetron LC (SDS Kerr, Middleton, USA).

Remaining unconverted double bonds were calculated using the standard method for monitoring the change in the ratio of aliphatic C=C absorption at 1638cm⁻¹ to aromatic carbon-carbon absorption at 1608cm⁻¹ between cured and uncured specimens according to the following equation: $DC = 100 \times [1 - (C/U)]$, where C and U are reasons of intensity between aliphatic and aromatic C=C absorptions, after and before the polymerization, respectively. Thus, the percentage of carbonic double linking that did not react during the polymerization reaction is determined. The DC is determined by the subtraction of the residual percentage of aliphatic C = C from 100%.

Statistics analysis

Statistical analysis was performed using one-way ANOVA and Tukey's test to both tests. In the present study, $\alpha=0.05$ was considered as the level of significance.

3 RESULTS

The results of ANOVA showed no significant differences for color stability among the tested luting systems ($p=0.4494$). Table 2 shows the Tukey's test for color stability values. All the luting systems demonstrated significant changes in color stability.

Table 2: Mean values of the color stability (ΔE) of the materials evaluated.

Luting Systems	N	Means (SD)	Tukey
TB/VVE	8	9.338 (3.504)	a
SB/ARC	8	8.313 (3.622)	a
TB/TFL	8	7.875 (2.763)	a
TB/VAR	8	7.438 (2.644)	a
SB/RVE	8	6.863 (3.052)	a
SB/FFL	8	6.113 (3.779)	a

Data transformation was performed before the analysis of variance test. ANOVA showed significant statistically differences among the materials tested. Table 3 shows the Tukey's test for degree of conversion (%). Tetric N-Flow and RelyX Veneer showed the highest degree of conversion, while lowest values were found for RelyX ARC.

Table 3: Values of the Degree of conversion (DC) of the materials evaluated.

Luting Systems	N	Means (SD)	Tukey
TTFL	10	0.02053 (0.0003)	a
VVE	10	0.02040 (0.0005)	a
FFL	10	0.01937 (0.0008)	b
RVE	10	0.01853 (0.0006)	c
VAR	10	0.01667 (0.0003)	d
ARC	10	0.01234 (0.0006)	e

Means followed by different letters represent significantly different.

4 DISCUSSION

The study showed discoloration of the materials after accelerated aging in a weathering machine, the color changes in luting systems induced by UV irradiation have been related to chemical alterations in the activators, initiator systems, and the resin cement itself. Degradation of residual amine and oxidation of residual unreacted carbon-carbon double bonds also result in the change of color¹⁴. In addition, the monomers contained in the resin matrix may influence in the staining¹⁴. The presence of UDMA can contribute to a reduction in the amount of TEGDMA, which is the monomer that release higher quantities of monomers into aqueous environments¹⁵, added to this is the monomer responsible for higher rates of water sorption in resin-based materials due to its hydrophilic ether linkages¹⁶. Therefore, materials that replace part of TEGDMA for UDMA may have less color change¹². Compared with BIS-GMA, UDMA appears to be less susceptible to staining¹⁷.

As reported, RelyX ARC and RelyX Veneer contains BIS-GMA and TEGDMA, Variolink II is composed of BIS-GMA, TEGDMA and urethane dimethacrylate,

Variolink Veneer contains UDMA and decamethylene dimethacrylate, Tetric N-Flow contains BIS-GMA and TEGDMA and Filtek Z350 Flow is basically composed of BIS-GMA, TEGDMA and BIS-EMA. The results showed that the monomers contained in the resin matrix might not influence in the staining. All the materials tested showed clinically visible color changes, the ΔE values ranging between 6,11 to 9,33 greater than 3,3 were considerable clinically perceptible, based on the previous reported¹².

Another possible explication is after the accelerated aging procedures, that combining UV light exposure with cycles of humidity and light can cause oxidation of the amine, which is necessary for initiating the polymerization process. The similar results of all materials tested although different results were expected are the formulation. As dual-cured resin cement, the base paste of Variolink II and RelyX ARC contains both aliphatic and aromatic tertiary amines, and the catalyst paste contains benzoyl peroxide. The light-cured resin Variolink Veneer and RelyX Veneer were made of base paste only, while the dual cured resins were prepared by mixing base and catalyst, so that the aromatic tertiary amine remained intact in the light-cured materials¹⁷. The inclusion of two kinds of amine in both materials can explain why the ΔE of the different groups did not significantly differ in the current study.

Previous investigations have shown that light-polymerizing resin, Variolink Veneer and RelyX Veneers, claims to be amine reduced, thus, it is expected to be more color stable. However, its ΔE was in the range of others cements. The light-cured resin cements should receive enough light energy to polymerize sufficiently¹⁸, the efficiency of this polymerization can also influence discoloration, because it determines the amount of residual monomer available to form colored degradation products¹⁷, this can explain the highest values of light-cured materials that showed no difference statistical of the dual-cured resin cement.

A high level of conversion for the dual-cured resin cements are expected because of the both curing mode (chemical and light), although because all dual-cured resin cements must be mixed, the possibility of the incorporation of air bubbles during the mixing process occur¹⁹ this can explained the significantly lower conversion values were obtained to both dual-cured resin cements Variolink II and RelyX ARC.

The conversion values for materials noted in this study were higher for Tetric N-Flow and Variolink Veneer; this finding may be due to the low viscosity of the materials which 64% and 60% respectively of filler concentration. Decreased viscosity allows a greater diffusion of free radicals in the polymerization process and higher conversion

potential¹⁹ this can explain the better results for these materials.

However, methodological limitations for *in vitro* studies are inherent in the assessment of clinical performance of materials thus more *in vivo* studies are necessary to help clarify the better luting systems indicated to laminate veneers.

5 CONCLUSIONS

Within the limitations of this *in vitro* study, the following conclusion can be drawn: Light- and dual-cured demonstrated unacceptable color change after aging. Despite to the dual cure of the RelyX ARC luting cement, it showed the lowest degree of conversion values, while Tetric N-Flow and RelyX Veneer presented the highest and similar degree of conversion.

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