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RESEARCH ARTICLE

Color Stability and Degree of Conversion of Light-cured Resin Cements

Ana Carolina Andreucci[1,2], Kusai Baroudi[3,4,*], Maiara Rodrigues Freitas[1,2], Marina Amaral[5,2], Flavio Baggio Aguiar[6,7], Rayssa Ferreira Zanatta[8,9] and Priscila Christiane Suzy Liporoni[1,2]

[1]*Department of Restorative Dentistry, University of Taubaté, Taubaté, SP, Brasil*
[2]*Departamento de Odontologia, Universidade de Taubaté, Unitau, Taubaté, Brasil*
[3]*RAK College of Dental Sciences, RAK Medical & Health Sciences University, Ras Al Khaimah, United Arab Emirates*
[4]*School of Dentistry, University of Taubaté, Taubaté, Brazil*
[5]*Department of Prosthodontic Dentistry, School of Dentistry, University of Taubate, Taubate, Brasil*
[6]*Department of Restorative Dentistry, Piracicaba Dental School, University of Campinas, Piracicaba, SP, Brasil*
[7]*Departamento de Odontologia Restauradora, Faculdade de Odontologia de Piracicaba, Universidade de Campinas, Unicamp, Campinas, Brasil*
[8]*Department of Dentistry, School of Health Sciences, University of Brasília (UnB), Brasil*
[9]*Departamento de Odontologia, Faculdade de Ciências da Saúde, Universidade de Brasilia, UnB, Brasilia, Brasil*

Abstract:

Objective:

To evaluate the color stability and degree of conversion of light-cured resin cement with different activator-initiator systems using LED lights with different wavelengths (polywave x monowave).

Materials and Methods:

Sixteen resin cement samples were made using a circular silicone matrix (7 mm diameter, 0.5 mm thickness) for each of the following tested materials: Variolink Esthetic LC (Ivoclar, color Light+); RelyX Veneer (3M ESPE, color A1); Filtek Z350XT flow resin (3M ESPE, color A1); Allcem Veneer APS (FGM, color A1); NX3 Light cure (Kerr, color A1). Half of the samples were photocured with a monowave LED light (Elipar Deep Cure), and the other half with a polywave LED light (Valo Grand). The initial color of each cement was measured using a high translucency ceramic sample simulating ceramic venner. Color measurements were performed with a reflectance colorimetric spectrophotometer and the data was collected according to the CIE L* a* b* system in two steps. The degree of conversion was measured using an infrared spectrometer by Fourier transform (FTIR / ATR) and the absorbance method. Statistical analysis was performed using ANOVA repeated measures and Tukey's *post hoc* tests ($p < 0.05$).

Results:

For the color analysis, there were no significant differences between the cement related to time *versus* light curing ($p = 0.084$) and also related to time versus cement versus light curing ($p = 0.142$). Among the factors, there was only a statistically significant difference for the type of cement ($p < 0.01$). In contrast, for the photocuring device ($p = 0.504$) and the interaction between them ($p = 0.738$), there was no significant difference. For the degree of conversion analysis, it showed a statistically significant difference for both factors, resin cements ($p < 0.01$) and light curing units ($p < 0.01$).

Conclusion:

The color stability of RelyX cement is low compared to other cements, while Variolink cement presented the best degree of the conversion value.

Keywords: Resin cements, Alternative photoinitiators, Ceramic veneers, Color stability, Variolink cement, Patients.

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1. INTRODUCTION

Ceramic restorations, including veneers, onlays, overlays, and full crowns are among the most popular choices in clinical dental practice for patients seeking esthetic restorative treatments [1]. The evolution of ceramics associated with different technologies for their handling allowed their use without metallic structures, becoming an increasingly common option of pure ceramic to the detriment of metal ceramics. In the case of indirect restorative procedures, most of these

* Address correspondence to this author at the RAK College of Dental Sciences, RAK Medical & Health Sciences University, Ras Al Khaimah, United Arab Emirates., Khuzam Road, Seih Al Burairat, QX5J+68W, PO Box 12973, Al Qusaidat, Ras al Khaimah, United Arab Emirates; Tel: +9717222259;
E-mail: d_kusai@yahoo.co.uk.

restorations adhere to the tooth surface using an adhesive technique [2].

In this context, there are three types of resin cement available for the cementation of ceramics, and their classification is according to the polymerization mode: self-curing, light-cured or dual [3]. Ceramic veneers are restorations that require careful planning because they are thin-thickness restorations that could reflect the color of cement [1]. Color instability of resin cement is a common problem and can be caused by extrinsic or intrinsic factors. Extrinsic factors include staining caused by infiltration of food, drink, habit, and addictions; intrinsic factors are directly related to the restorative material, chemical composition, type of photoinitiator, type and quality of polymerization [1, 4, 5]. Resin cement that have camphorquinone as a photoinitiator in the composition react with an intermediate molecule (tertiary amine), which over time, can decompose and change the color of the cement, directly interfering with the aesthetic quality of ceramic restorations [6]. Due to esthetic failures related to long-term color change, there is an indication not to use dual-curing cement with a tertiary amine in relatively thin ceramic restorations that reflect the color of cement [1, 6, 7]. Light-cured resin cement uses the photoinitiator camphorquinone to induce the formation of free radicals, resulting in the polymerization reaction. Furthermore, the degree of polymerization for light-cured cement depends on light penetration, which is influenced by the thickness and translucency of indirect restorations [6, 8].

It has been observed in the clinical routine that color instability of light-cured cement in a short period of time and try-in pastes as a guide for choosing the appropriate color do not always reproduce an exact match of colors with the cement before and after their polymerization [9]. Color prediction in ceramic veneers remains a challenge for dentists, given the numerous factors that influence the esthetic outcome of restorations [10, 11].

Due to the color change of cements with camphorquinone photoinitiator and tertiary amine co-initiator, companies are producing methods to replace it or reduce its concentration and combine it with other alternative photoinitiators [1, 6, 7] which may require LED devices with different wavelengths [1, 4, 6].

Therefore, this study aimed to evaluate the color stability and degree of conversion of light-cured resin cement with different activator-initiator systems using LED lights with different wavelengths (polywave x monowave). The first null hypothesis is that there is no difference in color between light-cured cements using different types of LED lights. The second null hypothesis is that there is no difference in the degree of conversion (DC) between light-cured cement using different types of LED lights.

2. MATERIALS AND METHODS

The sample size was calculated based on the study of Pissaia *et al*. [12]. using the software G Power 3.1 (Heinrich-Heine-Universität, Dusseldorf, Germany) and the following factors were considered: the values for color alteration, using the level of significance of 95% and the power of the test of 80%. The sample size was determined as n = 8.

The specimens were made using a silicon matrix with 7mm diameter and 0.5mm thickness, which was placed over a glass plate and filled with the respective cementing agent: Variolink Esthetic LC, color Light+ (Ivoclar Vivadent); RelyX Veneer shade A1 (3M ESPE); Filtek Z350XT flow shade A1 (3M ESPE); Allcem Veneer APS shade color A1 (FGM Dental Products); NX3 Veneer shade A1 (Kerr). The composition of the cements tested is described in Table **1**.

After placement of the cementation agent inside the mold, a polyester strip was placed over it, followed by another glass plate and compression was made to guarantee uniformity and remove bubbles. Then, a standard translucent lithium disilicate ceramic (IPS e.max CAD, Ivoclar Vivadent) specimen with 1mm thickness was placed over the cement and then light cured for 40 secs with either a monowave (1470 mW/cm^2 - Elipar Deep Cure, 3M ESPE) or polywave (1000 mW/cm^2 - Valo Cordless Grand, Ultradent Products) LED light.

The initial color measurement of each sample was obtained using a spectrophotometer (CM-2600d, Konica Minolta, Japan) following the CIE Lab. The initial color coordinates were obtained immediately after light curing the samples with the equipment set at D65 light, 100% ultraviolet light, specular reflection included (SCI), observe angle at 2º, and reading area of 25 mm^2 [13]. After the initial color reading, the samples were stored in Eppendorf Tubes, and final measurements were performed after 7 days. The same parameters adopted for the initial readings were used, and color alteration was calculated by means of the formula: $\Delta E^* = (\Delta L^2 + \Delta a^2 + \Delta b2)^{½}$.

Table 1. Composition of the cementing agents tested.

Cementing Agents	Initiator	Fillers	Organic Composition
Variolink Esthetic LC Ivoclar Vivadent	Ivocerin	Ytterbium Trifluoride and Zirconia Oxide Beads	UDMA
Relyx Veneer 3M	Camphorquinone, Photoinitiator Diphenyl Iodonium Hexafluorophosphate (PDIH)	Zirconia, silica and fumed silica	BisGMA and TEGDMA
Filtek flow Z350XT 3M	camphorquinone	zirconia and silica	Bis-GMA, TEGDMA and Bis-EMA.
Allcem Veneer APS FGM	Camphorquinone and co-initiators	Silanized barium, aluminum and silicate glass particles and silicon dioxide 63%	BisGMA and TEGDMA
NX3 Veneer Kerr	Tertiary amine and benzoyl peroxide free redox system	Silica and barium aluminum silicate glass particle	HEMA and titanium dioxide pigments

Besides color, the degree of conversion (DC%) was also measured for each cementing agent, either with the monowave and the polywave LED lights using a spectrophotometer by means of FTIR (Spectrum 100 FTIR/ATR; Perkin Elmer, USA). The uncured spectra of all agents were obtained by placing a portion of it over the equipment crystal. The parameters used were absorbance, 32 readings at 4cm^{-1} resolution, from 650 to 4000cm^{-1}. For the cured spectra it was used, the samples immediately after the final color readings by placing them over the equipment crystal, and final readings were made under slight pressure made by the arm for load application of the equipment itself. The conversion degree (%DC) was calculated using the formula:

$DC\% = 100x[1-(R_{cured}/R_{uncured})]$,

in which R represents the ratio between the absorbance peaks 1640cm^{-1} and 1610 cm^{-1}.

2.1. Statistical Analysis

Data were checked for normal distribution and then subjected to analysis of variance in two factors (Two-ways ANOVA). Microhardness (KMH) and roughness (Ra) were analyzed by repeated measures ANOVA, followed by the Tukey test. Color data (ΔE) and EDX values were submitted to ANOVA two-way, followed by the Tukey test. All evaluations were made considering $p < 0.05$.

3. RESULTS

For the color alteration, the ANOVA test showed a significant difference in the time factor ($p < 0.01$) and time *vs*. cement interaction ($p < 0.01$). For the interactions time *vs*. light curing ($p = 0.084$) and time *vs*. cement *vs*. light curing ($p = 0.142$), there were no differences. Among the factors, there was a significant difference only in the type of cement ($p < 0.01$), while for the light curing unit ($p = 0504$) and interaction between them ($p = 0.738$) there was no difference.

The Tukey test showed that for Elipar Deep Cure (Table **2**) the Allcem values increased, while for RelyX, they decreased and for the others, there was no significant difference. In the immediate time, Allcem presented the lowest value of ΔE compared to Variolink, with no difference between the others. Interestingly, after 7 days, only RelyX had its value reduced, while the others had similar values. In the general analysis, for cement, only Variolink presented higher values of ΔE, being similar only to Allcem.

For Valo Grand (Table **3**), the Tukey test also showed an increase in the value of ΔE for flowable resin, Allcem cement and Variolink, while for RelyX, there was also a decrease in value. In the immediate period, the cements RelyX, NX3 and Variolink presented the highest values. In contrast, for the final period (7 days) all values were similar, with the exception of RelyX, which showed a significant reduction. In the general analysis, also for the Valo Grand, only the Variolink presented higher values of ΔE, being similar only to the NX3.

For the analysis of the degree of conversion DC%, the ANOVA test showed significant difference for the two factors under study, cement ($p = 0.0002$) and light curing ($p = 0.0001$), as well as for the interaction of light curing factors *vs*. cement ($p < 0.0001$). Tukey test showed that there was a statistical difference between Elipar Deep Cure and Valo Grand LED, and also showed statistical differences between the cement tested in both light sources (Table **4**).

Table 2. Mean values and standard deviation of ΔE with elipar deep.

ΔE – Elipar Deep Cure					
-	Immediate		7 days		Cement Factor
-	Mean	SD	Mean	SD	
Filtek Flow	2.29	(±1.13) Bca	3.41	(±0.96) Ba	A
Allcem Venner	1.71	(±0.54) Aba	4.63	(±2.07) Bb	A
RelyX Veneer	3.84	(±1.11) Bca	1.47	(±0.25) Ab	A
NX3 Ligth Cure	3.73	(±1.30) Bca	3.79	(±0.66) Ba	AB
Variolink Veneer	3.89	(±1.07) Ca	5.27	(±1.00) Ba	B
Time factor	a		b		-

Note: Capital letters show difference in columns for cement factor and lower case letters show difference in rows for time factor.

Table 3. Mean and standard deviation values of ΔE with valo grand.

ΔE – Valo Grand					
-	Immediate		7 days		Cement Factor
-	Mean	SD	Mean	SD	
Filtek Flow	1.19	(±0.34) BCa	4.35	(±0.74) Bb	A
Allcem Venner	2.13	(±0.81) ABa	4.71	(±0.45) Bb	A
RelyX Veneer	3.39	(±0.65) Ca	1.84	(±0.52) Ab	A
NX3 Ligth Cure	3.43	(±1.20) Ca	3.44	(±0.72) Ba	AB
Variolink Veneer	3.46	(±0.96) Ca	5.11	(±0.93) Bb	B
Time factor	a		b		-

Note: Capital letters show difference in columns for cement factor and lower case letters show difference in rows for time factor.

Table 4. Mean values and standard deviation of DC of the cementing agents.

Cementing Agents	LED Light Unit	
	Elipar Deep Cure	Valo Grand
Filtek Flow	48.68 (±0.77) Bb	54.73 (±0.84) Aa
Allcem Venner	50.85 (±1.30) Aa	54.70 (±0.95) Aa
RelyX Veneer	50.94 (±1.53) Aa	49.68 (±1.21) Ab
NX3 Ligth Cure	50.73 (±1.31) Aab	51.47 (±1.18) Ab
Variolink Veneer	49.48 (±1.44) Bab	54.75 (±0.73) Aa

Note: Different letters (capital letters compare the LED light unit and lowercase letters compare resin cement) indicate a statistically significant difference ($p < 0.05$).

For flowable composite resin and Variolink light-cured resin cement, the Valo Grand poly wave light curing agent promoted higher DC% compared to mono wave LED. For Elipar Deep Cure, there was no significant difference in DC% between the light curing agents tested for the other resin cements.

When light-cured with the Valo Grand LED, the flow composite resin, the Variolink and Allcem cement presented the highest DC% values compared to the RelyX and NX3 cement, with no significant difference between them. When light-cured with the Elipar Deep Cure LED, RelyX and Allcem cement showed higher DC% when compared to flowable composite resin. The resin cements NX3 and Variolink presented intermediate values, with no significant difference.

4. DISCUSSION

All cements had a significant increase in the ΔL value, which represents a positive result. The higher the ΔL value, the clearer and the more lower, the darker value, which means that no cement showed darkening within the individual analysis of ΔL. RelyX and NX3 cements had the lowest ΔL value and Variolink, Allcem and Flow cements had the highest ΔL value. Thus, the first null hypothesis was denied.

Among the agents compared in the present study, Variolink and NX3 had the lowest value of Δb*, the axis that represents the variation between yellow and blue, the higher the value of Δb*, the closer to the yellow color scale and the lower the value of Δb*, the closer to the blue color scale. These results are consistent with previous findings [12, 14] and can be explained by the fact that Variolink has a photoinitiator derived from dibenzoyl germanium (Ivocerin) and NX3 has a redox-free initiator system, tertiary amine and benzoyl peroxide. The inclusion of an initiating system free of tertiary amines and benzoyl peroxide, such as NX3, makes the cement more stable in relation to color change. This result can be attributed to the fact that the NX3 contains an amine-free redox initiator system and an optimized resin matrix [1].

The lower Δb* value for the Variolink and NX3 cements can also be explained by the fact that they are the only luting agents tested that do not have camphorquinone as a photoinitiator. The presence of camphor quinone in the composition of light-cured cements is responsible for the initiation of the polymerization reaction; however, when this yellowish compound is not completely consumed, it degrades, causing a change in the color of cement [6, 12, 15].

Previous studies reported that ΔE<1 is not detectable by the human eye; ΔE between 1 and 3.3 can be perceived by specialists but is considered clinically acceptable; while ΔE >3.3 is perceived by laypersons as being clinically unacceptable [16]. Thus, the present study and most studies consider that ΔE≤3.3 is clinically acceptable [14, 17]. In the immediate period, Allcem and Flow had the lowest ΔE value, the Variolink, RelyX and NX3 cements had ΔE≥3.3 values, considered clinically unacceptable.

After 7 days, the only cement that had a decrease in ΔE was RelyX, the other cement tested had the ΔE value increased above ΔE≥3.3 - clinically unacceptable. The thickness of resin cement used in this study (0.5 mm) was greater than the thickness of the cement line used under acceptable clinical conditions with values close to 120 µm [2]. Therefore, it was the minimum possible for evaluation in the equipment, which could be considered a limitation of this study, and this may justify the accentuated values of ΔE, characterizing them as outside the clinically acceptable limits [1, 12, 16].

The shade of resin cement is a factor that influences the color stability of resin cements. Lighter shades tend to have less color stability than darker shades, producing more visible changes and higher ΔE values [3, 12], which may also justify the accentuated values of ΔE in the present study.

This study compared two photoactivation devices, monowave and polywave, but showed no significant difference in ΔE change between the devices tested. Despite the limitations of the pioneering study, the results corroborate the general belief that the light-activatable cementing agents present better color stability and the flow resin tested can be used as a cementing agent, as it presents similar results to the other tested cements [12, 14, 16 - 18].

But photoactivation devices presented a difference regarding DC%. The DC% results showed a statistical difference between Valo Grand and Elipar Deep Cure. Filtek flow and Variolink luting agents had higher conversion values with Valo Grand compared to Elipar. Difference in the composition of systems, differences in charges and opacities and also in the different photoinitiators and co-initiators, as well as characteristics of each light source, such as power, emission spectrum, beam collimation, light homogeneity, among others, are directly related to the color change and DC of these agents [6, 19].

Variolink contains thiocarbamide photoinitiator derived from dibenzoyl germanium (Ivocerin) with violet light absorption at 385 - 400nm, which justifies the decrease in conversion with Elipar, which has a longer wavelength [14].

When light-cured with the monowave LED Elipar, the cement RelyX Veneer and Allcem Veneer showed the highest DC values, followed by NX3, Variolink and Filtek flow, but without significant difference with the former, as well as when compared to flow resin, which showed the lowest DC.

The highest DC% value for RelyX is probably due to diphenyl iodonium hexafluorophosphate (DPIH) photoinitiator in its formulation, which has higher DC% when polymerized with a blue LED in the wavelength range between 430 - 480nm [20]. AllCem contains small amounts of camphorquinone and several co-initiators in its composition, which may have influenced its conversion values. However, the composition of Filtek flow's organic matrix contains ethoxylated bisphenol A dimethacrylate, as well as Bis-GMA and triethylene glycol dimethacrylate. Such a component can decrease the material's mechanical properties because it is particularly sensitive to the conditions of photoactivation and could have contributed to a lower degree of conversion [16].

For cements polymerized with Valo Grand, the highest DC values obtained were from Variolink, Filtek flow, Allcem and they showed statistical differences compared to RelyX Veneer and NX3 cements. Valo Grand is a polywave LED with a wavelength between 385 – 515 nm, which can improve the DC performance of materials with alternative initiators or co-initiators that require violet light [6, 19]. Variolink presented the highest conversion degree values that can be explained by the presence of Ivocerin in the composition, which was designed to promote improvement in DC and polymerizes properly with violet light [14, 15].

Inadequate polymerization decreases the physical properties of the resin material, adhesion strength and rigidity, in addition to increasing water absorption and affecting color stability [21]. There is no total conversion of monomers, and an acceptable DC% ranges from 42 to 72% [21]. The present study showed statistical differences between the cement tested in both light sources, but none had DC% lower than acceptable values in other studies [21, 22]. Besides color stability, it should be highlighted that the longevity of the cemented ceramic restorations also depends on the correct cementation technique, with proper control of humidity using dental isolation [23, 24], control of gingival bleeding during cementation [25], and proper removal of resin cements excesses [26].

CONCLUSION

1. Relyx Veneer cement showed the lowest ΔE value at 7 days, all other cements showed clinically visible color change values after 7 days.

2. Variolink Veneer cement light cured with Valo Grand showed the best DC% in immediate time.

3. The LEDs evaluated showed effectiveness in polymerization through 1 mm thickness of lithium disilicate ceramic for most of the cement studied.

ETHICS APPROVAL AND CONSENT TO PARTICIPATE

Not applicable.

HUMAN AND ANIMAL RIGHTS

No animals/humans were used in this research.

CONSENT FOR PUBLICATION

Not applicable.

AVAILABILITY OF DATA AND MATERIALS

The data supporting the findings of the article is available from the corresponding author [K.B] upon request.

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CONFLICT OF INTEREST

The authors declare no conflict of interest, financial or otherwise.

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