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Laboratory Research

Color Stability of Resin Cements Exposed to Aging

NC Ramos • JN Luz • MC Valera • RM Melo • GSFA Saavedra • E Bresciani

Clinical Relevance

In certain preparations for indirect restorations, the cement line is exposed and unprotected by the ceramic. This condition leads to esthetic compromise over time.

SUMMARY

The aim of this study was to evaluate the color stability of light-cured and dual-cured resin cements after artificial accelerated aging. Ten specimens (6-mm diameter and 2-mm thickness) for each of five resin cements were prepared: GC (dual-cured cement, GCem), Vb (light-cured cement, Variolink II only the base), Vbc (dual-cured cement, Variolink II base with catalyst), VV (light-cured cement, Variolink Veneer), and FR (flowable resin composite, light cured). The samples were polished and stored in an accelerated artificial aging machine for 308 hours (160 klx), with cycles of 120 minutes under light and 60 minutes in the dark. All aging was carried out in distilled water at 37°C and light irradiation at 765 W/m^2. The samples were evaluated in a spectrophotometer before and after aging, and results were calculated according to CIEDE2000. The data were statistically analyzed (one-way analysis of variance and Tukey test, 95% confidence). The results of ΔE_{00} were statistically significant for the type of cement ($p<0.001$), with differences among tested groups. Variolink II (base only and base + catalyst) and the flowable resin were the cements with the lowest color variations after the artificial accelerated aging. Considering the values ΔE_{00} of acceptability and perceptibility, none of the tested cements showed acceptable values.

Nathalia C Ramos, DDS, MSc, PhD student, Restorative Dentistry Program (Prosthodontics), Department of Dental Materials and Prosthodontics, Institute of Science and Technology, São Paulo State University (UNESP), São Paulo, Brazil

Julio N Luz, DDS, MSc, PhD student, Restorative Dentistry Program (Prosthodontics), Department of Dental Materials and Prosthodontics, Institute of Science and Technology, São Paulo State University (UNESP), São Paulo, Brazil

Marcia C Valera, DDS, MSc, PhD, professor, Department of Restorative Dentistry, Institute of Science and Technology, São Paulo State University (UNESP), São Paulo, Brazil

*Renata M Melo, DDS, MSc, PhD, researcher, Department of Dental Materials and Prosthodontics, Institute of Science and Technology, São Paulo State University (UNESP), São Paulo, Brazil

Guilherme S F A Saavedra, DDS, MSc, PhD, assistant professor, Department of Dental Materials and Prosthodontics, Institute of Science and Technology, São Paulo State University (UNESP), São Paulo, Brazil

Eduardo Bresciani, DDS, MSc, PhD, associate professor, Department of Restorative Dentistry, Institute of Science and Technology, São Paulo State University (UNESP), São Paulo, Brazil

*Corresponding author: 777 Eng. Francisco José Longo Avenue, 12245-000, São José dos Campos/SP, Brazil; e-mail: renata.marinho@ict.unesp.br

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INTRODUCTION

Ceramic laminates have emerged as an ideal esthetic treatment for anterior teeth. The treatment success depends on (among other factors) the correct adhesive technique to bond the porcelain veneers.[1] Because of improvements in adhesives and resin cements over time, reliable and satisfactory results might be expected.

Cements with different polymerization mechanisms are available, having specific advantages and disadvantages.[2] Resin cements for veneers are normally activated by visible light. The major advantages of these systems are color stability and working time compared with chemically activated and dual-cured systems.[3] Dual-cured resin cements combine activation by light and chemical mechanisms; this type of activation leads to improved mechanical properties, such as increased flexural strength, elastic modulus, and hardness, when compared with other curing systems.[4] Another advantage for both the dual-cured and chemically-cured systems is the initiation of curing in regions where light cannot reach.[5,6] Dual-cured resin cements have a lower color stability compared with cements with only photoinitiators. The main disadvantage of this material might be the lack of color retention over time due to oxidation of tertiary amines in the formulation of dual cements.[7]

When dealing with very thin and/or translucent pieces of ceramic laminates, one should consider light-passing characteristics for choosing the resin cement. The color of employed resin cement influences the final color of the laminate after cementation,[8] although there is no agreement in the literature regarding the color stability of the final restoration. Ghavam and others showed that color changes in resin cement might affect the esthetics of ceramic laminates after aging,[2] while Turgut and Bagis reported no perceptible difference of color change in resin cements when underneath ceramic laminates.[9] Moreover, with minimally invasive preparations or cementing in unprepared surfaces, the termination region is usually exposed and cement is left unprotected by the ceramic, with such cement possibly being esthetically harmed over time.

Resin cements are subject to intrinsic and extrinsic staining factors. The intrinsic factors are those related to the materials composition, filler content, and type of activation. There are reports that triethylene glycol dimethacrylate is more susceptible to staining, with larger filler particles also influencing the discoloration of the material as they detach themselves from the organic matrix, resulting in a rougher surface that is more susceptible to staining.[9-11] Extrinsic factors are the adsorption and absorption of media, including food and beverage dyes.[10]

In addition to those considerations, the survival rate of veneers is about 94.4% after 12 years, with a low clinical failure rate (approximately 5.6%).[1] With few mechanical failures reported, veneer restorations will remain in service for a long time, and thus materials might be susceptible to aging. Marchionatti and others performed a clinical study that showed marginal discolorations after 24 months in 40% and 30% of veneers, cemented with light and dual-curing systems, respectively.[12] Thus, the objective of this study was to evaluate the color stability of light-cured and dual-cured resin cements exposed to accelerated artificial aging. The null hypothesis tested was that no difference in color variations will be detected considering the different types of resin cement tested under artificial aging.

METHODS AND MATERIALS

Five resin cements were used, as shown in Table 1. Ten disc-shaped specimens (diameter: 6 mm; height: 2 mm) were prepared from each material. Metal matrixes with Teflon tape were used for preparing specimens.

The top surfaces of all samples were irradiated for 60 seconds with a light-curing unit (Radii Cal LED, SDI, Victoria, Australia) operating on standard mode and emitting 1200 mW/cm^2 irradiance, measured by a radiometer (Demetron LED Radiometer, Kerr Corporation, Middleton, WI, USA).

The top surfaces of all specimens were polished using the complete sequence of a composite polishing system (Astropol, Ivoclar Vivadent, Schaan, Liechtenstein) from coarse to superfine. Each disk was used for 30 seconds with a handpiece rotating at approximately 10,000 rpm. This step was performed to remove the oxygen-rich surface layer and to flatten and polish the surfaces. The same operator polished all samples in random sequence. After polishing, the specimens were washed to remove any remaining debris.

Color was evaluated before and after accelerated photo-aging periods at 308 hours using specific aging equipment (SunTest CPS+, Atlas, Mount Prospect, IL, USA). The specimens were exposed to a filtered xenon lamp with an illuminance of 160 kilolux and irradiation of 765 W/m^2. Each 180-minute cycle consisted of 120 minutes exposed to a light followed by 60 minutes in the dark. The samples were stored in a container with distilled water at constant room temperature (37°C).[13-15]

Table 1: *Materials Used in the Study*[a]

Resin Cement	Group	Curing	Composition	Manufacturer
GCem	GC	Light-cured	Dimethacrylates, 4-META, phosphoric ester monomer, fluoro-aluminio-silicate glass, camphorquinone	GC Corporation, Tokyo, Japan
Variolink II	Vbc	Dual-curing	Bis-GMA, UDMA, TEGDMA, barium glass, ytterbium trifluoride, Ba-Al-fluorosilicate glass, spheroid mixed oxide, catalysts, stabilizers, and pigments	Ivoclar Vivadent AG, Schaan, Liechtenstein
Variolink II (only base)	Vb	Light-cured	Bis-GMA, UDMA, TEGDMA, ytterbium trifluoride, barium glass, ytterbium trifluoride, Ba-Al-fluorosilicate glass, spheroid mixed oxide, catalysts, stabilizers, and pigments	Ivoclar Vivadent AG
Variolink Veneer	VV	Light-cured	UDMA, TEGDMA, silicon dioxide, initiators, and stabilizers (Value O)	Ivoclar Vivadent AG
Grandio (flowable resin)	FR	Light-cured	BIS-GMA, TEGDMA, HEDMA, functionalized SiO_2 nanoparticles with glass ceramic particles[b]	Voco GmbH, Cuxhaven, Germany

Abbreviations: 4-META, 4-methacryloxyethyl trimellitic acid; Bis-GMA, bisphenol A diglycidyl ether dimethacryalate; HEDMA, hexane diol dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate.
[a] *Abbreviations used for group, type of polymerization, composition given by manufacturer, and the manufacturer.*
[b] *Morresi AL, D'Amario M, Monoco A, Rengo C, Grassi FR, & Capogreco M (2005) Effects of critical thermal cycling on the flexural strength of resin composites* Journal of Oral Science ***57(2)*** *137-143.*

The color measurements were performed with a spectrophotometer (CM-2600D, Konica Minolta, Ramsey, NJ, USA) according to the CIE-Lab (Commision Internationale de l'Eclairage, L*, a*, b*) coordinates with a D65 standard light source. The CIE L* parameter corresponds to the degree of lightness and darkness, whereas a* and b* coordinates correspond to red or green (+a* = red, –a* = green) and yellow or blue (+b* = yellow, –b* = blue), respectively. The colorimeter was calibrated with a standard white and black plate every 20 specimens. For the evaluation of the color of the specimens, the reading was performed under reflectance on a white background (L: 84.95; a: –0.38; b: 2.93), with ultraviolet component included.

The color difference was calculated using the CIEDE2000 (ΔE_{00}) formula[16,17]:

$$\Delta E' = \left[\left(\frac{\Delta L'}{K_L S_L}\right)^2 + \left(\frac{\Delta C'}{K_C S_C}\right)^2 + \left(\frac{\Delta H'}{K_H S_H}\right)^2 + R_T\left(\frac{\Delta C'}{K_C S_C}\right)\left(\frac{\Delta H'}{K_H S_H}\right)\right]^{1/2}$$

where $\Delta L'$, $\Delta C'$, and $\Delta H'$ are the differences in lightness, chroma, and hue for the samples (before and after aging) in CIEDE2000 and RT is a function (the so-called rotation function) that accounts for the interaction between chroma and hue differences in the blue region. Weighting functions, SL, SC, and SH, adjust the total color difference for variation in the location of the color difference pair in L, a, and b coordinates, and the parametric factors, KL, KC, and KH, are correction terms for experimental conditions.[16,17]

Statistical treatment was performed by descriptive statistics (mean values and standard deviations), one-way analysis of variance (ANOVA) and two-way repeated-measures ANOVA, and Tukey's test with 95% confidence in Minitab 17 software (Minitab Inc, State College, PA, USA). Data were previously checked and confirmed for normality and homogeneity using Kolmogorov-Smirnov and Levene's tests ($p>0.05$), respectively.

RESULTS

The results of ΔE_{00} are presented in Table 2. One-way ANOVA showed a statistically significant difference for the type of cement ($p<0.001$). Using the Tukey's test (95%), GCem and Variolink Veneer cement presented the highest color changes. Variolink II (only the base and base + catalyst) cement and flowable composite had the least variation in color after the artificial accelerated aging.

Table 3 shows the mean and standard deviation values for each resin cement before and after the artificial aging. Tukey's test was performed for each parameter of L, a and b. All cements showed statistical difference between values, considering the baseline and after aging data within each group. An exception should be mentioned for the flowable resin b parameter and for Variolink Base in a and b parameters. The greatest differences were detected for Variolink Veneer and GCem cement, in agreement with their respective ΔE_{00} results. When a and b parameters were observed, a huge difference was seen in the Variolink Veneer group. All materials increased their b parameter values after aging, except for Variolink Veneer.

Table 2: *Mean and Standard Deviation of ΔE_{00} for Each Group, Coefficient of Variation, and 95% Confidence Interval*[a]

ΔE_{00}	Mean ± SD	Coefficient of Variation	Confidence Interval
GC	5.051 ± 0.594[A]	23.37	4.458; 5.644
Vb	2.013 ± 0.504[B]	75.99	1.420; 2.606
VV	4.458 ± 1.521[A]	41.72	3.865; 5.051
Vbc	2.770 ± 0.970[B]	44.56	2.177; 3.363
FR	1.991 ± 0.687[B]	54.90	1.398; 2.584

Abbreviations: FR, Grandio (flowable resin); GC, GCem; Vb, Variolink II (only base); Vbc, Variolink II; VV, Variolink Veneer.
[a] Tukey's test is represented by uppercase letters within the mean column; different letters represent a statistically significant difference among the groups.

DISCUSSION

According to the results obtained from the experimental groups, the null hypothesis that groups would not present statistical differences for color change was rejected. Groups with samples made from Variolink II cement (only the base and base + catalyst) and flowable composite presented the least variation in color after being subjected to artificial accelerated aging.

Most studies use the CIELab color difference to evaluate their results, but CIEDE2000 was developed to correct the CIELab and to better determine color acceptability and perceptibility. This correction was determined between the computed or measured color and the perceived visual color for better application in a clinical context.[12,18]

The color difference $\Delta E_{00} = 1.8$ refers to 50:50% acceptability, and $\Delta E_{00} = 0.8$ refers to 50:50% perceptibility, according to Paravina and others.[19] Taking that into consideration, none of the cements used in this study presented results within that range. On the other hand, color variations should also be evaluated for the other parameters, such as ΔL, Δa, and Δb, as seen in Table 3. The variations in L, a, and b parameters agreed with the ΔE_{00} results. The a parameter shows the color variation between green (negative) and red (positive), indicating that Variolink Veneer was susceptible to a change in red color after aging. The b parameter represents the variation between blue (negative) and yellow (positive). As the Δb value for Variolink Veneer was smaller after aging, a decrease in yellow tone was observed. Kilinc and others found similar results when they used Nexus 2 (bisphenol A diglycidyl ether dimethacryalate and dimethacrylate) in dual-cured mode.[20] The authors blamed the bleaching effect of the ceramic veneer for the cement lightening but did not further explain why this occurred. In fact, the material itself suffered a bleaching effect; therefore, the highest color variation ΔE_{00} for Variolink Veneer should not be considered without a comprehensive examination of the other parameters. The bleaching effect may be related to a continuously delayed dark curing with further consumption of initiators. Moreover, residual molecules may be further excited under intense light of the aging procedure, which could intensify the bleaching effect. In general, overall color variation is not expected, but it is better when this variation does not lead to yellowing. This was the case in the present study and also in the study by Kilinc and others. Further studies need to address the reasons why this occurred.

Despite the observed different behavior in color variation, the compositions of Variolink II and Variolink Veneer are close, namely, inorganic filler, dimethacrylate, catalysts, stabilizers, and pig-

Table 3: *Mean and Standard Deviation of L, a, and b Parameters Before and After Aging*[a]

Group	Color Reading	L*	a*	b*
GC	Baseline	68.39 (0.90)c	6.36 (0.44)g	21.62 (0.58)b
	After aging	69.88 (1.13)a	2.72 (0.50)a	27.06 (2.39)a
VB	Baseline	71.80 (0.41)b	3.74 (0.33)ab	25.33 (1.45)a
	After aging	69.84 (0.72)a	3.35 (0.34)bc	26.22 (1.87)a
VV	Baseline	76.67 (1.23)f	−2.25 (0.56)e	21.76 (1.75)b
	After aging	74.68 (0.71)e	0.05 (0.32)f	15.65 (1.50)e
VBC	Baseline	71.54 (0.82)b	4.36 (0.45)cd	23.41 (1.06)b
	After aging	69.40 (1.03)a	4.91 (0.62)d	27.26 (2.13)a
FR	Baseline	71.46 (1.03)b	3.72 (0.49)bc	18.10 (1.13)c
	After aging	70.25 (0.54)a	2.95 (0.51)a	18.62 (1.86)c

Abbreviations: FR, Grandio (flowable resin); GC, GCem; Vb, Variolink II (only base); Vbc, Variolink II; VV, Variolink Veneer.
[a] Tukey's test of each parameter is represented by lowercase letters in columns; different letters represent a statistically significant difference among the values.

ments. A slight difference is presented in the amount of inorganic filler, with Variolink Veneer presenting approximately 65% compared with 73% in Variolink II.[21] Variolink II (only the base) can also be used in the light-activated mode. According to Nathanson and Banasr, less color variation is expected when only the base is used compared with the regular activating mode (base + catalyst).[22] This may be related to low concentrations of chemical components attached while using in light-sensitive mode only. The composition of the cement base presents aliphatic and aromatic amines, whereas the catalyst contains benzoyl peroxide, which reacts with the aromatic amines for chemical polymerization. Ghavam and others did not find significant differences between these two groups (only the base and catalyst + base),[2] because although tertiary amines do not come into contact with the benzoyl peroxide of the catalyst, the base contains both amine components.[12]

Chemically cured cements also have a tertiary amine that aids in their curing. The degradation of residual amines and oxidation of residual carbon double bonds culminate in forming yellow components.[9] Variolink Veneer has a reduced amount of amine in its formulation according to its manufacturer, which should generate better chemical and color stability, which is different from what was seen in this study.

The GCem cement is not indicated for cementation of ceramic laminates according to the manufacturer. This cement contains phosphoric acid ester monomer, and all the self-adhesive cements that contain hydrophilic acidic monomers, such as carboxylic or phosphoric acid, can result in high sorption and solubility of their polymers.[23] This fact can be related to the results of improper color stability found in this study.

Turgut and Bagis reported the size of the filler particles of composites with discoloration.[9] Most composites have large filler particles and are more susceptible to staining. However, this study used a nanoparticle resin with 80% inorganic filler (nanoparticles functionalized SiO_2 with particles of glass ceramics),[24] and this fact might explain its better behavior in terms of color stability. Large particles may debond from the resinous matrix, leaving a rough surface exposed to the oral media and increasing the susceptibility to staining. When smaller particles (nanoparticles) are used, even if they debond from the matrix, superficial flaws would be smaller.[10]

Specimen dimension does not relate to clinical characteristics when considering a possible cement line exposed to the oral environment. On the other hand, the specimen finishing protocol was defined as being the closest to a clinical sequence. The samples were polished with specific tips for clinical use with a handpiece. It was decided that the cement should be evaluated without a ceramic layer because color change would depend on many factors related to the ceramic, such as ceramic color, translucency, thickness of the restoration, sintering, number of firings, polishing, and so forth.[25] Moreover, the intention was to evaluate the color change for the cement alone, to simulate the aging of the cement line, since a ceramic laminate veneer would protect the resin cement and substrate from the influence of artificial accelerated aging.[26] The present results might overestimate the clinical results, since the surface exposed to aging was greater than clinical conditions. The present results are directly related to the behavior of the tested materials under the aging protocol.

There is no way to predict whether these results fully reflect clinical practice, since the aging was accelerated and the entire cement was exposed to light irradiation, and these are limitations of this study. The protocol used for aging puts the materials under conditions of higher temperatures, humidity, and irradiation by light; therefore, oxidation of the amino group used as an initiator in resin materials can occur and lead to some kind of color change in the resin cements used.[2,3] Artificial accelerated aging does not represent the behavior in the oral environment, but efforts are being made to standardize this method in research and to cause accelerated degradation in effective time to find answers about the studied materials.[26]

CONCLUSION

Although there are limitations in an *in vitro* study, flowable composite resin and Variolink II (only the base or base + catalyst) showed the lowest variation in color after being exposed to artificial accelerated aging. Considering the acceptability and perceptibility levels, none of the resin cement used showed acceptable values.

Conflict of Interest

The authors of this article certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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