

RESEARCH AND EDUCATION

Effect of previous photoactivation of the adhesive system on the color stability and mechanical properties of resin components in ceramic laminate veneer luting



Henrico Badaoui Strazzi Sahyon, DDS, MS,^a Afonso Chimanski, DDS, MS,^b Humberto Naoyuki Yoshimura, DDS, MS, PhD,^c and Paulo Henrique dos Santos, DDS, MS, PhD^d

Adhesively cemented ceramic laminates may be an appropriate choice for esthetic dental restorations because they require little to no tooth preparation¹ and have favorable color match, texture, and shape, all of which promote a natural and symmetrical smile.¹ Lithium disilicate veneers reproduce polychromatism² and translucency³ and are compatible with periodontal tissue.^{1,4} When bonded with a resin cement, excellent replication of dental structure can be achieved.⁵ The resin cement also contributes to the esthetic quality of the restoration and needs to provide adequate mechanical properties.⁶⁻¹⁰

Whether dental adhesive should be photoactivated before the luting agent is applied¹¹ or concomitantly with activation of the resin cement remains unclear.¹² When the resin cement or dental adhesive used is exclusively light activated, it may not be completely polymerized due to light attenuation as a result of restoration thickness and/or opacity.^{13,14}

ABSTRACT

Statement of problem. The color stability and mechanical properties of luting agents influence the esthetics and longevity of ceramic restorations. However, studies evaluating the color changes and mechanical properties of luting agents under ceramic laminates activated by using different methods are lacking.

Purpose. The purpose of this in vitro study was to evaluate the effects of different modes of photoactivation on the nanohardness and elastic modulus of resin cements and dental adhesives and on the color stability of ceramic laminate veneers.

Material and methods. Forty-four lithium disilicate blocks (7×8×0.6 mm) were cemented onto bovine enamel and divided into 4 groups according to the polymerization light (Radii-Cal or Valo) used and the mode of activation of the dental adhesive (no previous photoactivation or previous photoactivation). Single Bond Universal dental adhesive and RelyX Veneer resin cement were used in all experimental groups. Color stability was measured using a UV-2450 ultraviolet-visible spectrophotometer before and after ultraviolet-B artificial accelerated aging (n=8). The nanohardness and the elastic modulus of the adhesive and resin cement were measured using a nanohardness tester (n=3). The color stability and mechanical properties were measured and analyzed using ANOVA and the Tukey least significant difference test ($\alpha=.05$).

Results. No difference in color stability or mechanical properties of the resin cement among the polymerization lights was detected ($P>.05$). Specimens that underwent previous photoactivation of the adhesive using the Valo polywave unit exhibited higher elastic modulus values than those that did not undergo previous photoactivation ($P<.001$).

Conclusions. The Valo polywave polymerization light improved mechanical properties and color stability more than the Radii-Cal unit. Previous activation of the dental adhesive in the dental enamel with the Valo polywave polymerization light yielded more satisfactory results. (J Prosthet Dent 2018;120:631.e1-e6)

Recently introduced polymerization lights include light-emitting diodes (LEDs). The emission spectra of the first dental LED polymerization lights were narrower than those of halogen lights, nearly 1000 mW/cm², very close to

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^aPostgraduate student, Department of Dental Materials and Prosthodontics, Araçatuba School of Dentistry, São Paulo State University (UNESP), Araçatuba, Brazil.

^bPostgraduate student, Center for Engineering, Department of Modeling and Applied Social Science, Federal University of ABC, Santo Andre, Brazil.

^cAssociate Professor, Center for Engineering, Department of Modeling and Applied Social Science, Federal University of ABC, Santo Andre, Brazil.

^dAssociate Professor, Department of Dental Materials and Prosthodontics, Araçatuba School of Dentistry, São Paulo State University (UNESP), Araçatuba, Brazil.

Clinical Implications

The correct activation of adhesive materials is an essential step when luting ceramic laminate veneers because it can directly affect the quality and longevity of the restoration.

the spectrum of camphorquinone.¹⁵⁻¹⁸ Single-wave and polywave LEDs have higher irradiances than halogen lights, ranging from 2000 to 3200 mW/cm², and single-wave LEDs emit a single wavelength emission peak.¹⁹ Polywave LEDs are equipped with multiple diodes, enabling polymerization of resin materials that contain other types of photoinitiators²⁰ including ivocerin and phenylpropanedione.²¹

The purpose of this *in vitro* study was to evaluate the effect of single-wave and polywave polymerization lights and different modes of activation of the dental adhesive on the color stability of ceramic laminates subjected to ultraviolet (UV) aging and to evaluate the nanohardness and elastic modulus of the dental adhesive and resin cement activated by different polymerization lights. The null hypotheses tested were that the mode of activation of the dental adhesive would not result in differences in the color stability or mechanical properties of the dental adhesive and resin cement; that different aging times would not cause differences in the color stability of the restoration; and different polymerization lights would not result in differences in the color stability of the restoration, or the mechanical properties of the dental adhesive or resin cement.

MATERIAL AND METHODS

The materials used in this study are listed in [Table 1](#). This study was approved by the Ethics Committee of the Araçatuba School of Dentistry, São Paulo State University (#2015-00673).

Forty-four lithium disilicate slices (7×8×0.6 mm) were made from high-translucency lithium disilicate blocks, shade B1 (IPS e.max CAD; Ivoclar Vivadent AG), using a low-speed diamond saw under water cooling (IsoMet 5000; Buehler). The ceramic slices were sintered in the manufacturer's furnace (Programat EP 5000; Ivoclar Vivadent AG) at 780°C for 1 hour.

Forty-four bovine teeth were used in this study; teeth that exhibited cracks or fractures were excluded. The anatomic crowns were removed 1.0 mm above the cementum-enamel junction with a transversal section using a low-speed diamond saw under water cooling to obtain tooth specimens of 7×8×4 mm. The buccal surfaces were abraded with #600 grit silicon carbide paper (Extec Corp), and the teeth were divided into 4 groups (n=11).

The enamel of specimens in the Valo_{WPA} group was etched using 37% phosphoric acid (Dentsply Sirona) for 30 seconds, washed with deionized water, and dried with air jets. An adhesive (Single Bond Universal; 3M ESPE) was actively applied for 20 seconds and dried with an air jet for 5 seconds, without polymerizing. The intaglio surface of the ceramic was conditioned with 10% hydrofluoric acid (Dentsply Sirona) for 20 seconds. Residual acid resulting from etching were removed using air/water spray, and the specimens were dried with air jet. A silane agent (RelyX Ceramic Primer; 3M ESPE) was applied to the surface for 60 seconds and air-dried for 5 seconds. A layer of adhesive (Single Bond Universal; 3M ESPE) was applied, and the solvent was evaporated for 20 seconds using an air jet, without photoactivation. Translucent Shade Resin Cement (RelyX Veneer; 3M ESPE) was applied directly on the intaglio surface of the ceramic laminate, and the restoration was positioned on the dental substrate. A load of 4.9 N was applied to standardize the thickness of the resin cement and subsequently removed. The excess cement was removed using a microbrush; the assembly was polymerized using an LED unit (Valo polywave; Ultradent Products, Inc) for 30 seconds in a black box to prevent external light interference. The luminous intensity of the polymerization light was evaluated by using a radiometer (Ecel RD7; Dabi Atlante) at 1584 mW/cm².

The Valo_{PA} group specimens were treated as described for the Valo_{WPA} group. However, the Single Bond Universal dental adhesive on the enamel surface was previously activated for 10 seconds using the Valo polywave LED polymerization light. The Radii_{WPA} group specimens were treated as described for the Valo_{WPA} group. However, the dental adhesive and resin cement were photoactivated using the Radii-Cal single-wave LED (SDI) polymerization light at a light intensity of 1222 mW/cm². The Radii_{PA} group specimens were treated as the Valo_{PA} group. However, the dental adhesive and resin cement were photoactivated using the Radii-Cal single-wave LED polymerization light.

All specimens were stored at 37°C in distilled water for 24 hours in light-protected containers. Thirty-two specimens were used to evaluate color stability (n=8), and 12 specimens were used to analyze the mechanical properties. Specimens were stored in Hanks solution at 37°C until the color measurement was performed.²² Baseline color was measured according to the Commission Internationale de l'Éclairage using a reflection spectrophotometer (UV-2450; Shimadzu) over a white background and standard illuminant D65. Five analysis readings were made for each specimen, and the arithmetic mean of the values was calculated.

Specimens from each group were aged using the EQUV UV-accelerated aging chamber (Equilam),

Table 1. Materials, classification, composition, and batch number of materials tested

Material	Classification	Composition	Batch
Single Bond Universal (3M ESPE)	Adhesive system	MDP, Bis-GMA, HEMA, photoinitiators, dimethacrylate, water, ethanol, silane.	1523700430
RelyX Veneer (3M ESPE)	Resin cement	Bis-GMA and TEGDMA monomers. Particles of zirconia/silica and colloidal silica. Average particle size of 0.6 mm. Filler loading 66% by weight.	1529200384
RelyX Ceramic Primer (3M ESPE)	Ceramic primer (silane)	MPS, ethanol, water	1400900844

Bis-GMA, bisphenol-A glycidyl methacrylate; HEMA, 2-hydroxyethyl methacrylate; MDP, 10-methacryloyloxydecyl dihydrogen phosphate; MPS, methacryloxypropyltrimethoxysilane (prehydrolyzed silane); TEGDMA, triethylene glycol dimethacrylate.

according to the American Society for Testing and Materials (G154 test). Eight fluorescent lamps (40-W each) were used. The specimens were subjected to alternating periods of UV light and condensation with distilled water saturated with oxygen under conditions of heat and 100% relative humidity. Each aging cycle was 12 hours in duration. In the first 8 hours, UV light was used at 60 ±3°C; in the next 4 hours, a condensation period without light was used at a temperature of 45 ±3°C.^{23,24} This test was performed for 3 periods of aging: 252, 504, and 756 hours. The color change was measured after each aging period (21 cycles), after 84 hours of condensation, and after 168 hours of UV-B with a 313-nm emission peak. The color change was measured again after the aging processes. The color difference was calculated between the color coordinates before (baseline) and after aging treatments applying the following formula:

$$\Delta E = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2},$$

where ΔE is the color difference, ΔL^* is $L_{after\ aging} - L_{baseline}$, Δa^* is $a_{after\ aging} - a_{baseline}$ and Δb^* is $b_{after\ aging} - b_{baseline}$.

Three specimens from each group were sectioned perpendicular to the occluso-apical direction using a low-speed diamond saw (Isomet 5000; Buehler) under water cooling to obtain 3 slices of the central portion. The slices were embedded in acrylic resin (Classico), finished using #320, #600, #800, and #1200 grit silicon carbide paper, and polished with diamond pastes (#6, #3, #1, and #0.25 μm) for a period of 3 minutes. The specimens were cleaned in an ultrasonic unit (Cristofoli) with deionized water for 8 minutes between each step and at the end of the process. The specimens were kept in Hanks solution to prevent cell degradation.²²

The nanohardness (*HIT*) and elastic modulus (*Eit**) were measured using a nanohardness tester (NHT2; Anton Paar). A Berkovich diamond tip was used at a load of 1000 μN and a standard trapezoidal load function of 5-2-5 seconds (Fig. 1).²⁵⁻²⁷ Six measurements were made in 2 different locations at the adhesive interface of each specimen: 3 indentations in

the dental adhesive and 3 indentations in the resin cement. The *HIT* and *Eit** values were calculated based on the load-displacement curves according to the following relationships:

$$HIT = P_{MAX} / A,$$

where P_{MAX} is the maximum load and A is the projected contact area between the indenter tip and the specimen under the maximum load.

$$Eit^* = E * (1 - \nu_s^2)$$

$$E^* = 1 / [(1/ER) - (1 - \nu_i^2)] / E_i,$$

where E_r is the reduced modulus, E_i is the modulus of elasticity of the diamond indenter tip, ν_i is the Poisson ratio of the diamond tip, and ν_s is the Poisson ratio of the analyzed material. The *HIT* and *Eit** values were calculated automatically by the software program installed with the tester.

Color stability was analyzed using a 3-way repeated measures ANOVA; the mechanical properties were analyzed using a 2-way ANOVA. The Tukey least significant difference test ($\alpha=.05$) was also used for both analyses.

RESULTS

Table 2 shows the analysis of the color stability, which revealed no differences between the polymerization lights under the same experimental conditions ($P>.05$). After 252 hours of aging, the group with no previous photoactivation exhibited more chromatic alteration of the dental enamel than the group that was previously polymerized using Valo polywave ($P=.023$). There was more chromatic alteration after 756 hours than after 252 hours in both protocols for the dental adhesive when the Valo polywave was used ($P<.05$) (Table 2).

Table 3 shows the mechanical properties values of the resin cement, with no differences in the *HIT* and *Eit** values between the polymerization lights in both protocols for the dental adhesive ($P>.05$). However, when the adhesive activation protocols were compared, the

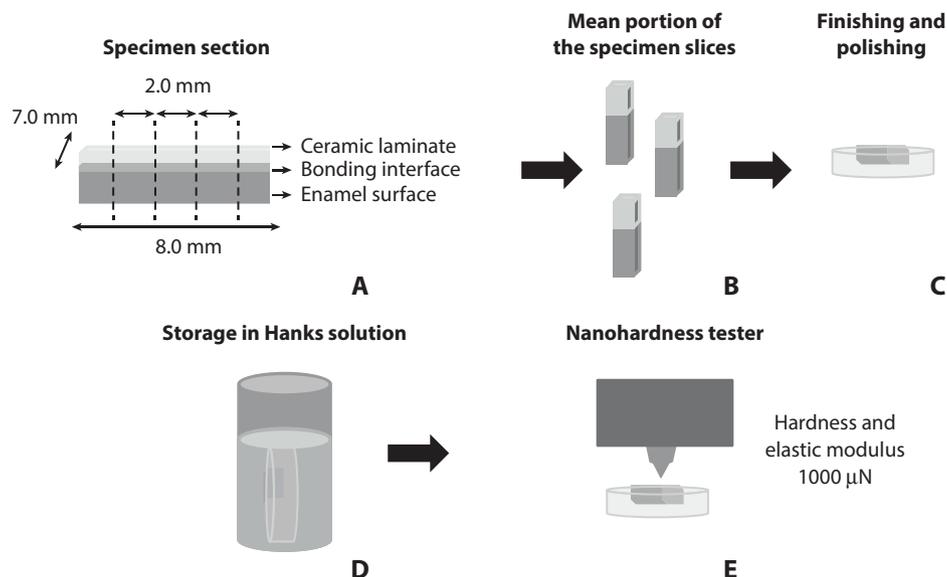


Figure 1. Specimen preparation for analysis of mechanical properties. A, Specimens sectioned perpendicularly to adhesive interface. B, Three slices approximately 2.0 mm in thickness obtained for each specimen. C, Specimens finished with silicon carbide abrasive papers and polished with diamond pastes. D, Specimens kept immersed in Hanks solution. E, Nanohardness (*HIT*) and elastic modulus (*Eit**) measurements with load of 1000 μN .

Table 2. Mean color change ($\Delta E \pm$ standard deviation) as function of aging period, polymerization light, and mode of activation of dental adhesive

Polymerization Light	Aging Period	Without Previous Photoactivation	With Previous Photoactivation
Valo	252 h	6.4 \pm 3.3 B a	3.1 \pm 1.0 B b
	504 h	7.9 \pm 3.8 AB a	5.8 \pm 3.4 A a
	756 h	10.9 \pm 5.3 A a	7.4 \pm 3.3 A a
Radii-Cal	252 h	5.3 \pm 2.3 A a	4.1 \pm 1.8 A a
	504 h	6.7 \pm 4.1 A a	5.2 \pm 2.1 A a
	756 h	8.2 \pm 2.9 A a	5.9 \pm 3.4 A a

Different letters (uppercase in polymerization light column, lowercase in row for same experimental condition) indicate statistically significant differences ($P < .05$).

material without previous polymerization had higher *Eit** values than that previously polymerized when the Radii-Cal single-wave polymerization light was used ($P = .005$) (Table 3).

Table 4 shows the mechanical properties of the dental adhesive. A comparison of the modes of activation of the dental adhesive revealed no difference in the *HIT* values between the groups evaluated for both polymerization lights ($P > .05$). In general, specimens polymerized with Valo polywave had better mechanical properties than those polymerized with the Radii-Cal single wave for both methods ($P < .05$). When the Valo polywave was used, the previously polymerized dental adhesive had higher *Eit** values than that with no previous photoactivation ($P < .001$). When the Radii-Cal was used, the adhesive without previous polymerization had higher *Eit** values than that with previous photoactivation ($P < .001$).

Table 3. Mean \pm standard deviation nanohardness (*HIT*) (MPa) and elastic modulus (*Eit**) (GPa) of resin cement as function of polymerization light and mode of activation of dental adhesive

Mechanical Properties	Polymerization Light	Without Previous Photoactivation	With Previous Photoactivation
<i>HIT</i>	Valo	726 \pm 74 A a	763 \pm 223 A a
	Radii-Cal	663 \pm 115 A a	642 \pm 147 A a
<i>Eit*</i>	Valo	12.3 \pm 1.4 A a	11.2 \pm 1.8 A a
	Radii-Cal	12.5 \pm 1.4 A a	10.9 \pm 1.4 A b

Different letters, uppercase in column and lowercase in row, indicate statistically significant differences for each mechanical property analyzed ($P < .05$).

Table 4. Mean (\pm standard deviation) nanohardness (*HIT*) (MPa) and elastic modulus (*Eit**) (GPa) of dental adhesive in enamel as function of polymerization light and mode of activation of dental adhesive

Mechanical Properties	Polymerization Light	Without Previous Photoactivation	With Previous Photoactivation
<i>HIT</i>	Valo	192 \pm 33 A a	194 \pm 38 A a
	Radii-Cal	138 \pm 55 B a	148 \pm 13 B a
<i>Eit*</i>	Valo	3.3 \pm 0.2 B b	3.9 \pm 0.2 A a
	Radii-Cal	5.5 \pm 1.9 A a	3.0 \pm 0.1 B b

Different letters, uppercase in column and lowercase in row, indicate statistically significant differences for each mechanical property analyzed ($P < .05$).

DISCUSSION

The mode of activation of the dental adhesive influenced the color stability and mechanical properties of the dental adhesive and the resin cement, and so the first null hypothesis was rejected. The use of different polymerization lights resulted in differences in color stability, *HIT*, and *Eit** values, thus the third null hypothesis was rejected. Finally, analysis of the effects of different aging times on the color stability of ceramic laminates led to a rejection of the second null hypothesis.

The composition of Single Bond Universal includes water and ethanol as solvents (Table 1). Dental adhesives containing alcohol or acetone solvents mixed with water result in minor chromatic changes.²⁸ When there was no prior polymerization of the dental adhesive, the water in the system may have been incorporated by hydrophilic groups in the resin cement at the time of luting of the laminate ceramic,²⁹ thus promoting hydrolytic degradation of the resin materials.³⁰ This hydrolytic degradation could explain the increased chromatic alteration of the dental adhesive not previously polymerized using the Valo polywave (Table 2).

The incorporation of water into the composition of the restorative material is directly related to solubility³¹ and significantly impacts the clinical behavior of dental materials. A high concentration of water could result in irreversible alterations such as cracks and hydrolytic degradation of the chemical components of the resin matrix.¹ RelyX Veneer resin cement includes triethylene glycol dimethacrylate (TEGMA), which contributes to color alteration. Materials based on this component release large quantities of monomers in an aqueous medium.¹ When associated with bisphenol-A glycidyl methacrylate (Bis-GMA)-based materials, water sorption is directly proportional to the concentration of the TEGMA present.³ The higher values of chromatic alteration in the specimens where the dental adhesive was not previously polymerized may have been due to the protocol used (Table 2). Previous activation of the adhesive in the dental enamel could have caused volatilization of the solvents because of the gradual increase in temperature from the light-polymerization process,¹⁷ thereby preventing water sorption by the Bis-GMA monomers in the resin cement.

An increase in the chromatic alteration values for both protocols activated using the Valo polywave was observed after 756 hours compared with 252 hours (Table 2). These results are consistent with previous studies in which chromatic alteration occurred in the first 300 hours of aging process because of a decrease in water sorption of the resin matrix and stabilization of polymer chains.^{4,11} Catelan et al.²⁴ reported that after 300 hours of aging, composite resins had undergone color parameter changes similar to those observed in a 24-month clinical study. Accelerated artificial aging promoted clinically perceptible chromatic changes. The exception was the group that underwent previous polymerization of the adhesive using the Valo polywave (Table 2) because the ΔE value was less than 3.3, which is considered imperceptible to human eyes.¹⁴ For these luting materials, the prior polymerization of the dental adhesive in the enamel appears to have contributed to improving color stability of the luting system.

The results of the chromatic stability analysis (Table 2) and mechanical properties of the resin cement (Table 3) indicated no differences between the polymerization

lights in all analyzed groups. Although they have distinct emission peaks (single wave, 430 to 480 nm; polywave, 385 to 515 nm) and irradiance ranges (single wave, 1222 mW/cm²; polywave, 1584 mW/cm²), there were no differences in the *HIT* and *Eit** values or in the chromatic stability analysis of the resin cement. It is speculated that the photoinitiators/coinitiators in the resin material were activated by the wavelengths emitted by both polymerization lights.^{15,16,18}

The mechanical properties of the adhesive were influenced by the polymerization lights used (Table 4). The higher light intensity emitted by the Valo polywave may have contributed to these results because some factors can affect light transmission through the ceramic, including the thickness and shade of the ceramic and resin cement, the microstructure of the ceramic, and the presence of defects and porosities, promoting light attenuation and decreased polymerization of light-activated resin materials.^{1,10} As the nanoindentation test enables the indirect evaluation of the quality of the conversion of monomers into polymers; therefore, the higher hardness values improve the mechanical behavior of the luting material.³²

Some limiting factors of this study should be considered, including the sensitivity of the nanoindentation test and the use of only 1 color of resin cement and 1 translucency of ceramic. Future variables examining the adhesion of ceramic laminates are warranted, such as marginal adaptation analysis, bonding strength, and permeability.

CONCLUSIONS

Based on the findings of this in vitro study, the following conclusions were drawn:

1. Nanohardness and elastic modulus were influenced by different polymerization lights, and the Valo polywave improved mechanical properties compared with the Radii-Cal single-wave polymerization light.
2. The polymerization light had no effect on color stability.
3. Previous photoactivation of dental adhesive in enamel using the Valo polywave yielded more satisfactory results.

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Corresponding author:

Dr Paulo Henrique dos Santos
 Araçatuba School of Dentistry (UNESP)
 Rua José Bonifácio, 1193
 Araçatuba, São Paulo 16015050
 BRAZIL
 Email: paulosantos@foa.unesp.br

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