

ملاحظات	صور 4 لون	صور 1 لون	ص الألوان	عدد الصفحات	الترقيم	رقم المقالة
						146

SPECTROPHOTOMETRIC ANALYSIS OF THE EFFECT OF POLYMERIZATION ON COLOR STABILITY OF FIVE RESIN CEMENTS

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ABSTRACT

Objective: To examine the effect of polymerization on color stability of five different resin cements.

Materials and Methods: A silicon mold was used to prepare 50 cement resin annuli (2 mm thick and 18mm diameter with a central hole of 8 mm) of five groups (n=10). Group A: Smart Cem 2 resin cement; Group B: RelyX U200 resin cement; Group C: RelyX Ultimate resin cement; Group D: Calibera Esthetic veneer resin cement; Group E: RelyX Veneer resin cement. Colorimetric values of the specimens before and after polymerization were measured using a spectrophotometer. The CIE L*a*b color system was used for the determination of color difference. Kruskal-Wallis test was used to analyze the data for significant differences. ($P \geq 0.05$).

Results: There were noticeable color changes in all groups ($\Delta E^*ab \geq 3.3$). Group C (RelyX Ultimate) showed the highest color change (6.66 ± 0.24) while Group D (Calibera Esthetic veneer) had the lowest color change (3.78 ± 0.18). Kruskal-Wallis test revealed high significant difference between four tested groups $p < 0.05$.

Conclusions: Tested resin cements showed color changes above the clinically accepted value in all study groups ($\Delta E^*ab \geq 3.3$).

Key Words: Resin Cement, Color Stability, Spectrophotometer, and ΔE .

INTRODUCTION

The final ceramic veneer color may be influenced by the thickness and translucency of the ceramic.¹ In addition to, the color of underlying tooth and luting agent which may also influence the final color of veneer restorations.²⁻⁸

Ceramic laminate veneer thickness has been reported to range between 0.3 and 1.5 mm.⁹ It has been shown that with a decrease in veneer thickness, light transmission was significantly increased.¹⁰ Previous studies have shown that changes in thickness of translucent feldspathic porcelain as

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well as the underlying cement may dramatically influence the final shade of the restoration.³

Resin cements are considered the best luting material for ceramic veneers. They are supplied in different shades by the manufacturers to allow for final color enhancement of porcelain/cement restorations. They may be light activated or dual-cured.

The main advantages of light activated cements are their longer working time, compared to chemically and dual-cured resin cements.^{11,12} The use of this type of cement makes it easier to remove any excess material before light-curing and reduces the finishing time required after cementation of the restorations.⁶ Dual-cured resin cements combine some of the desirable characteristics of light- and chemically cured resin cements.¹³ Besides the advantage of allowing further chemical curing in deeper areas where the light is attenuated,¹⁴⁻¹⁶ dual-cured resin cements have also shown superior mechanical properties, such as flexural strength, elastic modulus, and hardness.¹³⁻¹⁷ However, dual-cured resin cements also contain aromatic tertiary amine in their formulation, which could compromise the color stability of the cemented restorations over the long-term.¹²

The color stability and optical properties of resin composites have long been studied.¹⁸ Color change and loss of shade match with surrounding tooth structure is a major reason for replacement of esthetic restorations.¹⁹ The color stability of composites has been studied by artificial aging in different conditions and for different durations.^{20,21}

Light cured resin cements are sometimes supplied with try-in gels that should match the polymerized cement color. It helps the practitioner in final resin cement shade selection by giving him an idea of the final shade of veneer in combination with cement and tooth color. The question is; does this try-in gel really matches the polymerized cement color? A few reported clinical cases suffer from veneer final shade change immediately after polymerization

of the cement. In 2013, Karaarslan ES, et al, reported color changes of light-cured composite resin material after various polymerization methods above the clinically acceptable value.²²

Presently, electronic shade-matching instruments have been used for dental practice.¹⁵ In order to determine objectively the color changes on resin cements, some methods have been experienced, among them the spectrophotometry. In this method reflected wavelength by a body is changed in values expressed in ΔE^* units. Instrumental color analysis offers a potential advantage over visual color determination, as instrumental readings are objective, quantifiable, and more rapidly obtainable.²³

The Commission Internationale de l'Eclairage Lab (CIE L^*a^*b) color system has been used for determination of color difference.²⁴⁻²⁶ Chroma is calculated as $C^*ab = (a^{*2} + b^{*2})^{1/2}$.¹⁹ The total color difference ΔE^*ab between two color stimuli, each given in terms of L^* , a^* , b^* is calculated from:²⁷ $\Delta E^*ab = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$. The L^* coordinate is a measure of the lightness- darkness of the specimen. The greater the L^* is, the lighter the specimen. The a^* coordinate is a measure along the red-green axis. A positive a^* relates to the amount of redness, and a negative a^* relates to greenness of a specimen. The b^* coordinate is a measure along the yellow-blue axis, that is a positive b^* relates to the amount of yellowness; a negative b^* relates to the amount of blueness of the specimen. ΔL^* , Δa^* and Δb^* are the differences in the CIE color-space parameters of the 2 colors.²⁸

Threshold color difference levels based on instrumental color measurements that can be visually perceivable or clinically acceptable have been discussed. However, the clinically acceptable value for color difference in restorative materials is assumed to be $\Delta E^* \leq 3.3$.²⁹

The purpose of this in vitro study was to evaluate the amount of change in color of five different resin

cements after polymerization. The null hypothesis of the present study was that; there is no color difference between unpolymerized and polymerized resin cement.

MATERIALS AND METHODS

Fabrication of annulus prototype:

An annulus of 2 mm thickness and 18 mm diameter having a central hole of 8 mm diameter was drawn on AutoCad 2014 for mac software (Autodesk, San Rafael, CA, USA) and exported to Micro SLA 3D printer (ProJet 1200, 3D systems, South Carolina, USA). A castable liquid plastic cartridge (VisiJet FTX Green UV Curable Plastic, 3D systems, South Carolina, USA) was inserted into the 3D printer. Resin annulus was printed according to the same dimension. It was then separated from base and supporting material was cut off using round-end taper diamond bur (Komet, Brasseler, Germany). The annulus was then dipped in isopropanol alcohol solution for cleaning printed discs. (I.P.A, 3D systems, South Carolina, USA)

Fabrication of polymerized resin samples:

A silicon index for the resin annulus was constructed using industrial silicon (Body Double, Smooth-On, Inc.) (Fig. 1).



Fig. (1) Silicon index for the resin annulus.

A total of 50 annuli were identically fabricated and grouped into five groups ($n = 10$) (Table 1) according to material used, following the next steps:

- A) For dual-cured cements, the cement was auto-mixed and injected into the silicon index until completely filled. A glass microscope slide was placed on top of the silicon index under a fixed weight of 200 grams until complete setting of the cement. Each sample was then removed from the index and inspected for accuracy. (Fig. 2)

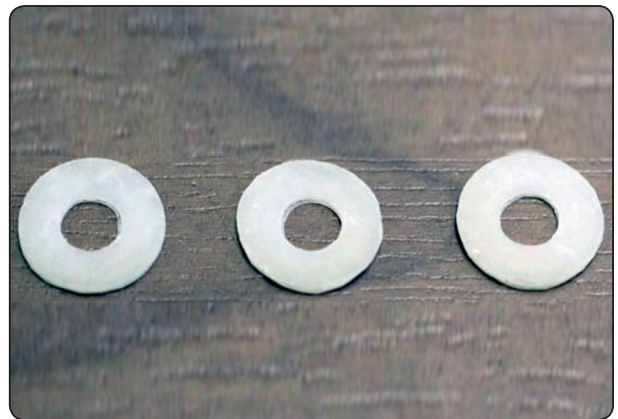


Fig. (2) Polymerized resin samples.

- B) For light-cured cements, the previous process was duplicated except for the polymerization, where a LED curing light (Elipar S10, 3M ESPE, USA) was applied for 10 sec. above the glass microscope slide until curing.

Spectrophotometric analysis of polymerized resin samples:

Colorimetric values of the resin cement specimens after polymerization were measured using a spectrophotometer (Vita Easy- Shade, Vident, No: 502744, USA). The spectrophotometer comprises a base unit and a hand-piece. In this system, the specimen was exposed to an emission of light, and the reflected light was analyzed with a spectrophotometer. Instrument calibration was evaluated after

TABLE (1) Sample grouping and properties of materials used.

Group	Material	Manufacturer	Type	Composition	Filler
A (n=10)	Smart Cem 2 Automix (SC)	DENTSPLY, Caulk, 38 West Clarke Avenue , Milford DE 19963	Dual cure resin cement	UDMA Resin, EBPADMA Urethane Fluoride Glass filler	69 wt%
B (n=10)	RelyX U200 Automix (R2)	3M-ESPE, St. Paul, MN, USA	Dual cure resin cement	TEGDMA, DMA Glass filler	45-55 wt%
C (n=10)	RelyX Ultimate Automix (RU)	3M-ESPE, St. Paul, MN, USA	Dual cure resin cement	TEGDMA, DMA zirconia/silica filler.	50-60 wt%
D (n=10)	Calibera Esthetic veneer Automix (CE)	DENTSPLY, Caulk, 38 West Clarke Avenue, Milford DE 19963	Dual cure resin cement	Bis-GMA, DMA Barium boron fluoro alumino silicate glass	>70 wt%
E (n=10)	RelyX Veneer (RV)	3M-ESPE, St. Paul, MN, USA	Light cured resin cement	Bis-GMA, TEGDMA, zirconia/silica filler.	66 wt%

Bis-GMA: bisphenol-A glycidyl dimethacrylate, TEGDMA: triethyleneglycol dimethacrylate; UDMA: urethane dimethacrylate; Bis-EMA: bisphenol-A ethoxylated dimethacrylate; DMA, dimethacrylate.

measurement of each group (n=10) and recalibration. The tip of the hand-piece was placed firmly into the calibration port and was held steadily in place until the instrument sounded a beep to indicate that calibration was complete. Specimens were positioned at the same place on different occasions to ensure consistency of the repeated measurements. A white plate was used for the background color in this study.^{27,30} All specimens were chromatically measured three times, and the average values were calculated. (Fig. 3)

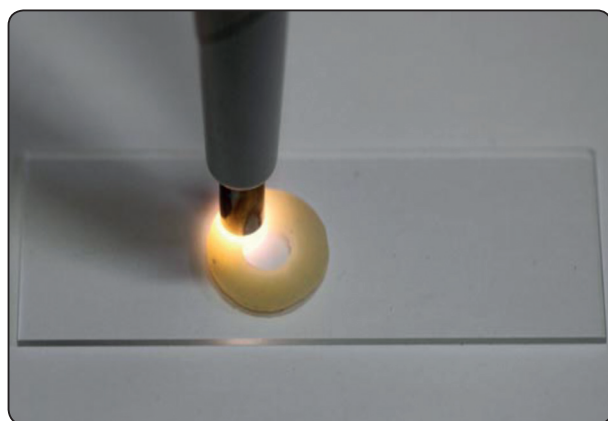


Fig. (3) Spectrophotometric analysis of polymerized resin samples.

Spectrophotometric analysis of unpolymerized resin:

On a white background plate color, each polymerized resin sample was placed. Fresh unpolymerized material of the resin sample was injected in its central hole of the sample (Fig. 4) and immediately placed under glass microscope slide. Using a spectrophotometer shade was recorded exactly like the previous procedure. CIE L*a*b color system was used for determination of color difference between polymerized and

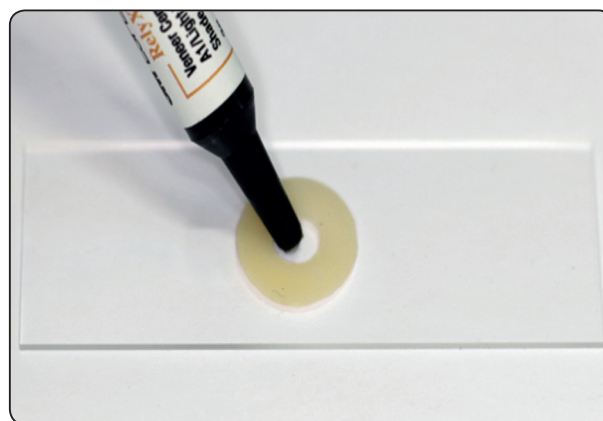


Fig. (4) Application of unpolymerized resin samples.

unpolymerized resin cement. Changes in color coordinates (ΔL^* , Δa^* and Δb^*) were calculated as 'the value after polymerization – the value before polymerization'. The formula of ΔE^*_{ab} : $\Delta E^*_{ab} = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$.

Statistical analysis

All data were entered into a spreadsheet (Excel; version 4.0, Microsoft, Seattle, WA, USA) for calculation of descriptive statistics (mean, standard deviation). Data were then entered the statistical package for social sciences SPSS version 20. Shapiro-Wilk test³¹ revealed data were not normally distributed. Statistical differences between groups were tested using Nonparametric Kruskal-Wallis test. Confidence level of the study was kept as 95.5% therefore, P- values less than or equal to 0.05 were considered statistically significant.

RESULTS

A total of fifty samples were tested for color stability after polymerization. Descriptive results of mean and standard deviation of delta E values of resin cements before and after polymerization between five groups are presented in Table 2 and chart 1. There were noticeable color changes in all groups ($\Delta E^*_{ab} \geq 3.3$). Group C (RelyX Ultimate) showed the highest color change (6.66 ± 0.24) while Group D (Calibera Esthetic veneer) had the lowest color change (3.78 ± 0.18).

TABLE (2) Mean Delta E $\pm$ SD between tested groups

Group	$\Delta E \pm SD$
A	6.32 ± 0.52
B	5.81 ± 0.48
C	6.66 ± 0.24
D	3.78 ± 0.18
E	6.03 ± 1.32

SD: standard deviation

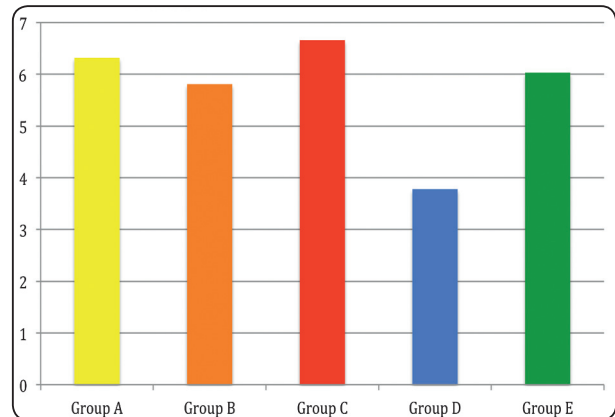


Chart (1) Delta E between tested groups.

For qualitative results Kruskal-Wallis test revealed high significant difference between four tested groups $p < 0.05$, as presented in table 3.

TABLE (3) Kruskal-Wallis test used to evaluate level of significance between tested groups

adjusted H:	29.962
df:	4
P value:	$4.98E^{-6}$

df: degree of freedom, P value: level of significance

DISCUSSION

This invitro study evaluated the color stability of five resin luting materials which may affect the final shade of ceramic veneers.

A number of studies³²⁻³⁵ measured color stability of resin cement using only 0.2 mm thickness cemented to ceramic. Samples were placed on a white plate background and spectrophotometer measured the color change of resin from below ceramic. This setting may negatively influence their results, as the white plate background may affect the 0.2 cement thickness and act as opaque cement. Clinically the cement is backed by translucent tooth structure not a white back ground. In the present study, 2mm thickness cement was used to intensify and isolate the effect of resin cement color alone.³⁶⁻³⁸

Other studies used the accelerated ageing protocol involving a weathering chamber where samples were submitted to increased temperature (63°C), humidity and ultraviolet light, for times ranging from 300 h to 900 h.^{11,34,38,39} This protocol is far apart from what actually happens intraorally and definitely doesn't represent clinical conditions. Extreme temperatures and ultraviolet light may be far extreme than the actual environment.

Spectrophotometers are today amongst the most accurate, useful and flexible instruments for color matching in Dentistry.⁴⁰ The data obtained from spectrophotometers are manipulated and translated into a form useful for dental professionals.⁴¹ The advantages of spectrophotometric analysis with the CIE L*a*b* system are the detection of color changes that are not visible to the human eye and the ability to express color differences in units that may be related to visual perception and clinical significance.⁴² There is some controversy in literature regarding the values of clinically noticeable color changes.⁴³ Vichi et al.⁴⁴ used three different ranges for distinguishing color differences: DE values lower than 1.0 were considered undetectable by the human eye, values between 1.0 and 3.3 were considered visible by skilled operators, but clinically acceptable, and DE values greater than 3.3 were considerable appreciable also by non-skilled persons and for that reason clinically not acceptable. Chang et al.⁶ reported the gold standard threshold of 2.0, which was considered a perceptible color change able to determine the optical effect of resin cements. In a recent study, a DE = 1.6 represented the color difference that could not be detected by the human eye.⁴⁸ However, most studies report DE + 3.3 as clinically acceptable.^{38,45} The color changes in the present study ranged from 0.41 to 2.40, regardless of the type of material, which would be within the previously mentioned conditions.

Results of this study showed noticeable color change ($\Delta E^* > 3.3$) between unpolymerized and polymerized resin cement. Which implies that the

try-in gels supplied by some veneer cementing kits are ineffective. These gels represent the color of the unpolymerized resin, which may change after polymerization affecting the final color of the ceramic veneer and jeopardizing esthetics. The results of current study are in accordance with Karaarslan, et al,²² who in 2013 concluded that, the color changes of composite resin material in all groups were above the clinically acceptable value.

Color changes may be related to the changes in the resin matrix and in the silanization process of the filler particles, causing higher water sorption.⁴⁶ Another explanation may be the size and number of particles that may influence the values of DE, DL*, Da* and Db*, as well as the translucency of composite resins.⁴⁷

The null hypothesis that was proposed for this study was rejected, since the resin cements changed in color.

CONCLUSIONS

Within the limitations of this in vitro study, the following could be concluded:

- The color changes of resin cement materials in all groups were above the clinically acceptable value ($\Delta E^*_{ab} \geq 3.3$).
- Calibera Esthetic veneer showed higher color stability than the other tested materials.
- RelyX Ultimate showed the lowest color stability between tested groups.

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