

**Research Article**

## **Evaluation of Color Stability of New Resin Cement after Accelerated Aging**

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### **ABSTRACT:**

**Background:** The color stability of resin cements have a great impact on the beauty of ceramic restorations especially laminate veneers. The purpose of this study was to evaluate the color stability of a new resin cement (RelyX Ultimate).

**Materials and Methods:**In this in vitro study, one light cure (RelyX Veneer) and three dual cure (RelyX Ultimate, RelyX U200 and Panavia F2) resin cements were used. 10 disc shaped samples for each of the resin cement (total of 40) were made. After immersing the specimens in the distilled water (37°C) for 24 hours, the initial color was measured using a spectrophotometer according to the CIE Lab standard system. The specimens were stored in the accelerated aging machine for 100 hours and then the final color measurement was performed and the color difference ( $\Delta E$ ) was calculated. The data were analyzed using one-way ANOVA and Tukey's tests at a  $P < 0.05$  level of significance.

**Results:** The RelyX Veneer cement showed the most discoloration in comparison with others. The color change of three other cements were clinically acceptable and showed no significant differences between each other.

**Conclusion:**The color stability of RelyX Ultimate was clinically acceptable.

**Keywords:**Aging, Color, Dual cure, Resin cements

### **INTRODUCTION:**

All-ceramic crowns and veneers are the most common anterior restorations due to their aesthetic and natural appearance. Since conservative and esthetic approach requires thinner restorations with higher translucency, the cement color stability under these restorations is important for long-term success. Resin cements are commonly used for the cementation of all-ceramic restorations, due to their good aesthetic properties,

low solubility, high bond strength, high mechanical properties and ceramic support.<sup>[1]</sup> Previous studies showed that the shade of luting cements under laying the porcelain restoration can affect the color of final restorations<sup>[2]</sup> and changing color of underneath cement beyond restoration or edges might be visible. Color stability of resin cements is influenced by internal factors such as the

composition of the filler and resin matrix, amount of filler, filler particle distribution, type of initiator and activator and degree of conversion, as well as external factors, including saliva, food, heat and UV irradiation.<sup>[3]</sup>

For cementation of porcelain laminate veneers, light cured resins are usually preferred because of better color stability, polymerization on demand characteristic and longer working time in comparison with chemically or dual cured cements.<sup>[2]</sup> In addition, to ease of use, studies have reported that the proper color stability of these cements is due to the lack of aromatic tertiary amine in their formulation, which could alter the color of cement over time. Moreover, with the use of light cured cements, the removal of excessive material before light curing would be easier and reduce polishing time.

The desirable features of self-cured and light-cured cements are combined in dual cured resin cements. In addition to the advantage of chemical curing in deeper areas where light intensity is low, dual cured cements have higher mechanical properties such as flexural strength, elastic modulus, stiffness and higher degree of conversion compared to light-cured or self-cure cements.<sup>[4]</sup> In chemically-activated cements such as dual cured and self-cured, the oxidation of reactive groups in amine catalyst and inhibitors may cause color change. It is noticeable that these accelerators present in light-curing system with a much lower degree.<sup>[1]</sup>

The color stability can be assessed visually or instrumentally. Color measurement devices such as colorimeter or spectrophotometer are common because of their accuracy, standardization and color numerical expression.<sup>[5]</sup> The color difference ( $\Delta E$ ) indicates whether a change in overall shade can be perceived by a human observer. The color differences greater than 3.3 between veneers and the teeth is considered as clinically unacceptable.

To simulate testing of the long-term intraoral color stability of composites, several in-vitro methods are presented including exposure to different food staining solution, visible light, UV radiation, Xenon rays and storage in water and dry

conditions.<sup>[6]</sup> In this study, we used an accelerated aging machine. Accelerated aging is a method that simulates the oral conditions, allowing the detection of color changes of materials over time, by exposing them to UV or Xenon light, temperature changes and humidity.<sup>[7]</sup>

According to the claim of manufacturer, compared to other advanced cements, RelyX Ultimate cement (3M ESPE, St. Paul, MN, USA) as a dual cured cement has excellent color stability due to the lack of aromatic amine components.<sup>[8]</sup> The aim of this study was to evaluate the color stability of RelyX Ultimate resin cement along with three other resin cements after accelerated aging. The null hypothesis of this study was 1) color parameters ( $\Delta E$ ) of Rely X Ultimate resin cement and other cements after accelerated aging will remain at an acceptable clinical level and 2) between tested cements after accelerated aging, there will be no considerable difference in color.

**MATERIALS AND METHODS:**

In this in vitro study, a light cured resin cement, RelyX Veneer (3M ESPE, St. Paul, MN, USA) and three dual cured resin cements including RelyX Ultimate (3M ESPE, St. Paul, MN, USA), RelyX U200 (3M ESPE, St. Paul, MN, USA) and Panavia F2 (Kuraray, Okayama, Japan) were used (Table 1).

**Table 1:** Description of the materials used in this study

Material	Composition	Manufacturer
Relyx Veneer	BisGMA, TEGDMA, Zirconia/silica and fumed silica, Pigments, Photoinitiator	3M ESPE, St. Paul, MN, USA
Relyx U200	Silane treated glass powder, substituted dimethylacrylate, 1-benzyl-5-phenyl-barbic-acid, calcium salt, 1,12-dodecanedimethacrylate, sodium p-toluenesulfonate, silane treated silica, calcium hydroxide	3M ESPE, St. Paul, MN, USA
Relyx Ultimate	10-Methacryloxydecyl dihydrogen phosphate (MDP) Dimethacrylate resins. HEMA. Vitrebond™ copolymer Filler. Ethanol. Water. Initiators. Silane	3M ESPE, Seefeld, Germany
Panavia F2	Paste A: BPEDEMA, MDP, DMA, silica, barium sulfate, dibenzoylperoxide Paste B: N,N-Diethanol-p-toluidine, silica sodium fluoride	Kuraray Noritake Dental Inc., Okayama, Japan

10 disc shaped samples (diameter of 14 mm and thickness of 1 mm) for each of the resin cement (total of 40) were made using a split metal mold. After the manipulation according to the manufacturer's specifications, each material was inserted on to a mold. The mold was placed on a glass plate with a black background to avoid light reflection. Then, another thin slide glass is placed on the upper surface of the mold and with a relatively uniform pressure of putting two fingers up to it, to remove excessive cement and create a uniform cement thickness. The cements were light cured directly on the upper slide using halogen curing unit (Optilux 501 Demetron, Kerr, USA) for 40s at 3 equidistant points of discs and confirmed for all groups at 600mw/cm<sup>2</sup>. The intensity of the light curing device was monitored periodically by its radiometer.

Afterwards, the specimens were wet polished sequentially using a series of silicon carbide grinding sheets (#600, #800 and #1200 grit) (Matador, Germany). The grinding and polishing process were performed on one side of the samples to make 1 millimeter thick specimens. The final thickness was checked with a digital micrometer (Mitutoyo Corp, Tokyo, Japan). Then all samples were stored in a lightproof container at 37°C distilled water for 24h. After this period, the initial color measurements were determined using a spectrophotometer (ColorEye 7000A XTH, Gretagmacbeth, USA) after calibration using a white standard. Measurements were performed according to the CIE Lab standard system.

Color measurements were repeated 3 times for each sample and the average was recorded as a color value. After color examination at baseline, samples were artificially aged in a weathering

machine (XenotestBeta+, Atlas, USA) for 100 hours under the following conditions: Irradiance: 50 w/m<sup>2</sup>, 40 minutes only xenon radiation, 20 minutes Xenon radiation and water precipitation, temperature 37°C, humidity 95% .<sup>[1,6]</sup> After aging process, color analysis were repeated for samples, as previously described. The color changes were calculated according to the following formula:

$$\Delta E = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2}$$

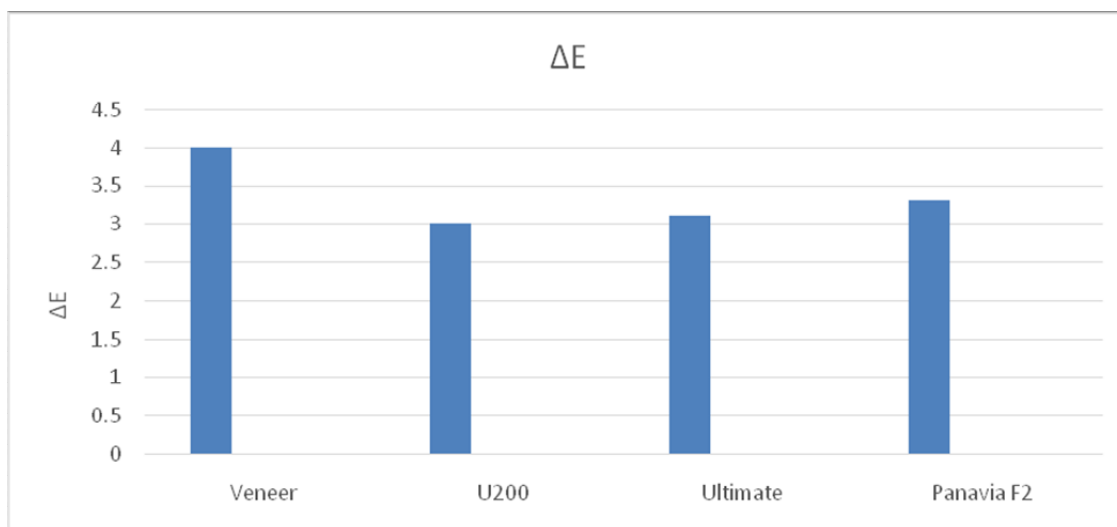
$\Delta L^*$  describes the differences in brightness,  $\Delta a^*$  stands for the changes on the red-green axis and  $\Delta b^*$  depicts the shifting on the yellow-blue axis. Data were analyzed using the one-way ANOVA and Tukey's tests were used to compare the groups. To determine the normal distribution of data, Kolmogorov Smirnov test was conducted as well. The level of significance was set at  $P < 0.05$ .

**RESULTS:**

Results of color change ( $\Delta E$ ) for the resin cements evaluated in this study are shown in Table 2. RelyX Veneer cement showed the greatest change in color whilst other 3 cements showed no significant differences in color changes between each other ( $P > 0.05$ ). RelyX U200, RelyX Ultimate and Panavia F2 had clinically subtle color change ( $\Delta E < 3.3$ ). Only the veneer cement showed color change over 3.3 that was not clinically acceptable. Also according to the recorded values of  $\Delta L$ ,  $\Delta a$  and  $\Delta b$  showed in Table 2, all four groups showed decreasing in brightness (L). Groups of Panavia and RelyX Veneer cements exhibited a red shift (+a), but U200 and Ultimate groups showed a green shift (-a). Yellow shift (+b) was observed in all groups except Panavia F2 cement that it had changed to blue (-b).

**Table 2:** The results of  $\Delta E$ ,  $\Delta L$ ,  $\Delta b$  and  $\Delta a$

	$\Delta E$ (SD)	$\Delta L$ (SD)	$\Delta b$ (SD)	$\Delta a$ (SD)
Relyx Veneer	4.0 (.66)	-1.67 (.4)	2.53 (.82)	2.52 (.18)
Relyx U200	3.0 (.80)	-.77 (.96)	.35 (1.67)	-3.22 (.24)
Relyx v	3.1 (.51)	-1.00 (.49)	-.72 (.52)	2.63 (.52)
Panavia F2	3.3 (.55)	-1.05 (1.55)	-.50 (.66)	1.96 (.2)



**Chart 1:** The results of  $\Delta E$

### DISCUSSION:

Many studies have examined the color stability of resin cements.<sup>[9-11]</sup> Most of these have only evaluated external factors and despite the known effect of resin color on final restorations, a small number of studies have focused on resin cement material itself.<sup>[12-14]</sup> In this study the color stability of three dual cured and one light cured cements after accelerated aging were evaluated. According to the results of this study, both hypotheses were approximately verified. The Panavia F2, Ultimate and U200 cements showed clinically acceptable values of  $\Delta E$  ( $<3.3$ ). Although there is controversy in the literature regarding the acceptable clinical value of color change ( $\Delta E$ ). The reported values are changed between 0.4 to 3.7. However in the most studies it considered as  $\Delta E < 3.3$ .<sup>[6]</sup>

In the current study a quantitative standard device, spectrophotometer, was used to determine the color value that is preferable rather than visual examination. Spectrophotometers are today amongst the most accurate, useful and flexible instruments for color matching in dentistry.<sup>[4]</sup> The resin cement thickness in this study was one millimeter that is much more than the actual clinical film thickness. This thickness of the samples was chosen because of the limitations of the *in vitro* testing methods. The spectrophotometer and ISO standards<sup>[15]</sup> require a

certain thickness of the samples for achieving reliable results.

Many color stability studies have used accelerated aging procedures combining the xenon light irradiation with cycles of light and moisture in order to better simulate the oral environment.<sup>[7,12,16-18]</sup> Water has a key role in unreacted monomer leaching, molecular displacement, degradation of the silane intermediate layer of filler-matrix and filler particles, so it will affect on color parameters of restorations.<sup>[6]</sup> The accelerated aging carried out in the present study using a weathering machine (Xenotest Beta+, Atlas, USA) submitted the samples to increased temperature, humidity and UV light. The manufacturer have estimated that around 300 hours of aging is equivalent to one year of clinical service.<sup>[19]</sup> Kokhar and colleagues reported that the most color change occurs within 100 hours of accelerated aging.<sup>[20]</sup> Therefore, in this study, 100 hours of accelerated aging was used.

According to the results of this study, all resin cements showed color change after accelerated aging, but it is clinically unacceptable only for RelyX Veneer cement ( $\Delta E > 3.3$ ) (Table 2). Also In this study, all groups exhibited yellow shift in color except Panavia F2. This result is in line with previous studies as it has been shown that accelerated photoaging led to a significant increase

in  $\Delta b$  in indirect and direct light-cured composite restorations.<sup>[6]</sup> Remaining camphorquinone (photoinitiator) is considered as a responsible agent for the yellow color change over time.<sup>[21]</sup> Moreover, tertiary aromatic amines (accelerators) form photoreactive byproducts that tends to cause yellow to red/brown color shift under the influence of light or heat.<sup>[17,22]</sup> The presence of unreacted benzoyl peroxide in dual cured materials may also lead to greater color change, which will jeopardize the long-term esthetic appearance of the resin materials. So the difference in concentration and structure of light initiators and reducing amines may explain the wide range of  $\Delta a$  and  $\Delta b$  values that obtained among tested materials. Moreover, the resin composition, type and size of fillers have an influence on color stability of tooth-colored restorative materials.<sup>[23]</sup> Since the size of filler particles is implicated in color stability, larger size of the filler is more susceptible to discoloration by wateraging. A possible explanation may be the direct relationship between perception and dispersion of color due to the fact that the hydrolytic degradation of the resin matrix-particle interface can change the path that light is scattered by particles. However, according to two studies (Dietschi et al<sup>[24]</sup> and Vichi et al<sup>[25]</sup>), differences in the proportion of resin-filler, resulting variations in water absorption, cannot explain the large  $\Delta E$  quantities of composite materials alone. Internal color change is accelerated by ultraviolet radiation and temperature changes.<sup>[26]</sup> The dual cured resins containing benzoyl peroxide and tertiary amine have more susceptibility to be darkening over time. In contrast, the color of camphorquinone that mainly used in light curing cement is more stable. Although the camphorquinone that is not polymerized enough gradually turn color to yellow.<sup>[27-29]</sup>

The color change values in the present study for dual cured cements were below clinically acceptable limit ( $\Delta E < 3.3$ ). Ghavam et al<sup>[30]</sup> and Archegas et al<sup>[4]</sup> showed in their studies that  $\Delta E$  values of dual cure cements were below 3.3 that are in consistent with our study. Dual cured resin cements combine some of the favorable

characteristics of light and self cured resin cements. In addition to the advantage of allowing further chemical curing in deeper areas where the light is reduced, dual cured resin cements have higher degree of polymerization and mechanical properties in comparison to the light or chemical ones.<sup>[4]</sup>

Most self-cure or dual-cure resin cements use benzoyl peroxide and tertiary amines to initiate the polymerization and curing, and this chemical combination tends to discolor with time<sup>[31]</sup> (however in this study the color change of dual cure cements were clinically acceptable). For this reason and to prevent discoloration (to combine good mechanical and optical properties), some resin cements were made without tertiary amine accelerators. Limited studies have done on new non-amine resin cements. It was recently reported by Smith et al (2011) that resin cements without a primer system of benzoyl peroxide/amine redox, showed better color stability.<sup>[3]</sup> Also, Ural and colleagues (2016) investigated the effect of non-amine systems and type of polymerization on color stability of resin cements and reported that new resin cements without tertiary amine and benzoyl peroxide have less color change than the Light-cure and dual-cure cements.<sup>[31]</sup> In this study the  $\Delta E$  values of Relyx Ultimate was clinically acceptable.

Several studies showed better color stability of light cure resin cements compared with dual-cure and self-cure cements. As they mentioned, this issue is related to the lack of aromatic amines in their composition.<sup>[32]</sup> In the current study, the RelyX Veneer as light-cure resin cement, showed values of  $\Delta E$  substantially higher than clinically indicated. A possible cause of this problem returns to mode of curing of these cements. Several studies have shown that light-cure resins have less degree of conversion compared to other dual-cure type; when the degree of conversion becomes less, resin is more susceptible to color change and will be unstable over time.<sup>[12,33]</sup> Papazoglou et al (2006) showed that dual-cure systems will polymerize better than light-cure types.<sup>[34]</sup> Di Francescantonio and colleagues evaluated the effect of viscosity

and curing type on resin cements and proved that the degree of conversion was higher for cements with lower viscosity.<sup>[35]</sup> Oliviera et al showed higher degrees of conversion by increasing the temperature in Dual-cure cements.<sup>[36]</sup> According to Beun and colleagues study, with decreasing filler particle size, the viscosity of resins increases.<sup>[37]</sup> Because of the smaller size of the filler particles in RelyX Veneer (0.2-3 Micrometer) compared with RelyX Ultimate cements (13 Micrometer) and RelyX U200 (12.5 mm), viscosity is high, so the higher viscosity decreases the cement fluidity and cause trapping the bubble and porosity inside the cement mass. This matter leads to increasing water absorption and the subsequent increase is likely to change color of cement.

#### CONCLUSION:

Considering the limitations of this study, the following conclusions were made:

1. After accelerated aging, the color stability of all cements was clinically acceptable ( $\Delta E < 3.3$ ) except Relyx Veneer ( $\Delta E > 3.3$ ).
2. Considering  $\Delta E$ , there was significant difference between Relyx Veneer and other groups. There was no substantial difference between Relyx Ultimate, Relyx U200 and Panavia F2.

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