Assessment of color stability of different resin cements having different modes of polymerization before and after aging

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Abstract: Objectives: This the aim of this in vitro study was to evaluate the effect of aging on the color change of three different resin cements of light and dual polymerization mechanisms and compare between the change in their color content after the aging process to determine which mode of polymerization has better color stability. **Methods:** 30 ceramic discs of diameter of 10 mm and thickness 0.5 mm were prepared from IPS Empress CAD blocks and three different resin cements, (Rely X Unicem, Rely X Ultimate and Rely X Veneer) were applied to the surface treated ceramic discs (each cement was applied on 10 discs) with a thickness of 0.1 mm through a custom made Teflon mould of internal thickness of 0.6 mm and then light curied, after that the samples have undergone an aging process (Thermocycling) in which the shade was measured by a digital spectrophotometer before aging and after 1000, 2000 and 3000 thermal cycling to determine the change in the color content of the resin cements. **Results:** Rely X Ultimate showed the highest color stability followed by Rely X Veneer while Rely X Unicem showed the lowest color stability of all the resin cements used. The results showed that within all the cements there was an increase in ΔE with increasing the number of thermal cycles and there was a significant difference ($p < 0.05$) between 24 hours and (1000 cycles, 2000 cycles and 3000 cycles), for the Rely X Ultimate and the Rely X Unicem there was a significant difference ($p < 0.05$) between 1000 cycles and (2000 cycles and 3000 cycles) as for the Rely X Veneer there was a significant difference ($p < 0.05$) between 1000 cycles and 3000 cycles, although all the final results of E among all the cements were clinically acceptable. **Conclusions:** The studied cements behaved acceptably according to E, but they became Darker after aging, and that the Rely X Ultimate (dual cured) resin cement could be used to cement either crowns or veneers with a high final esthetic outcome with a high degree of color stability like that of the light cured resin cements. Clinical Implications: The studied cements both using the dual cured and light cured mode of polymerization can ensure color stability when used to cement porcelain laminate veneers.

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1. Introduction

Porcelain laminate veneers have become an interesting treatment option for patients seeking better esthetics in the anterior region[(1)]. Patients usually prefer porcelain veneers because they require minimally invasive preparation of the tooth structure compared with other treatment options [(2)]. As for their biocompatibility and translucency they guarantee not only healthy margins but also superior esthetics [(3)].

The final color of a ceramic restoration could be influenced by translucency, opalescence, fluorescence, surface texture, the number of porcelain firings, and the condensation technique, and also by the color, translucency, and thickness of the underlying resin luting agent [(4-6)]. Resin cements are the luting agent of choice for the cementation of tooth-colored restorations, including both porcelain and indirect resin composite [(7)]. The effect of cement shades on the final color of ceramic restorations remains controversial; some investigations have shown that resin cements can produce perceptible color differences with particular combinations of background shades, cements, and ceramic restorations [(8, 9)]. Other studies identified that resin cements presented no significant effects on the final color of the ceramic material [(10-12)]. However, the thickness of restorative materials plays a major role on the final color of restorations.

One feature that is fairly consistent among various resin cements is the ability to use the material as a light-activated resin only or to mix with a chemical accelerator, making the resin both photo-initiated and dual-polymerized [(13)]. Research has shown that to obtain more complete polymerization under a restoration 2 mm or more thick, a dual-polymerized resin is necessary [(14,15)]. It has also been shown that opacity of the porcelain restoration does not affect polymerization as much as the thickness of the porcelain [(16-18)]. Therefore, in most bonding situations, it is preferable to use a dual-polymerized resin cement to insure an adequate bond [(13)]. The dual-polymerized reaction is initiated with a peroxide

initiator and an amine accelerator [15]. For photo initiation to occur, visible light in the range of 460 nm (blue) is necessary to activate the camphoroquinone initiator [16]. Some studies found that with accelerated aging the color of dual-polymerized cements is somewhat unstable [19] and that the additional chemicals necessary for dual-polymerization can cause the color of the cement to change over time, although little research has been completed on this topic [13].

The purpose of this study was to measure the color stability of both the light- and dual-polymerized versions of one shade of three commercial resin cements following accelerated aging.

2. Materials and Methods

2.1 Specimen Preparation

Thirty ceramic slices were machined to a uniform standard thickness of 0.5 mm from low translucency (LT) **IPS Empress CAD** (Ivoclar Vivadent AG, Schaan, Liechtenstein) blocks of shade A1, using a sawing machine ; **Metkon micracut 150 precision cutter** (Metkon metallography, Metkon Instruments ltd. Turkey), cutting at a speed of 350 rpm with a water cooling unit attached to the cutter, the discs were machined, thickness was verified by a digital caliper (Tresna; Guilin Guanglu Measuring Instrument Co., Ltd; Guangxi Province, China), then smoothened by silicon carbide sand paper of grit #600 then after that with grit #1200 from both sides for 30 seconds by light finger pressure along a line 5 cm long, then were ground into circular discs of a uniform diameter of 10mm by hand grinding and finally were polished by rubber wheels of different grits ; **Optrafine** (Ivoclar Vivadent AG, Schaan, Liechtenstein)**. IPS Universal Glazing Paste** (Ivoclar Vivadent AG, Schaan, Liechtenstein) was used to glaze the thirty discs on one surface only (to become the shade measurement surface later on) according to the manufacturer's directions of use to mimic the highly glazed and polished ceramic veneers cemented intraorally and the glaze firing was conducted in a compatible ceramic furnace e.g. **Programat P300** (Ivoclar Vivadent AG, Schaan, Liechtenstein)**.** Teflon Cylindrical sample moulds with external dimensions of 20mm diameter and 3mm thickness and internal dimensions of 10 mm diameter and 0.6 mm depth of the internal stopper which was included inside the mould In order to assure a 0.1mm uniform resin cement space as well as proper disc positioning [31]. The two open ends of the mould facilitated light curing by direct contact with the sample (from the glazed surface) and easy removal of the samples from the mould after each one is prepared respectively. Before application of the resin cements to the unglazed surfaces of the samples were treated with **Dentobond porcelain fix** (Laboratoire ITENA, PARIS , France) a Porcelain etch gel and a Porcelain Silane coupling agent, the dual-cured resin cements **Rely X Ultimate**, **Rely X Unicem** and the light cured resin cement **Rely X Veneer** (3M Deutschland GmbH, Seefeld)were applied each on the unglazed treated surface of ten ceramic discs and cured by a high intensity LED curing unit ; **Bluephase** (Ivoclar Vivadent AG, Schaan, Liechtenstein) through the glazed surface of the ceramic for 20 seconds for both the Rely X Ultimate and RelyX Unicem [21,22] and 30 seconds for the RelyX Veneer[23]. Resin\Ceramic sample thickness 0.6 mm was verified by a digital caliper after removal from the mould.

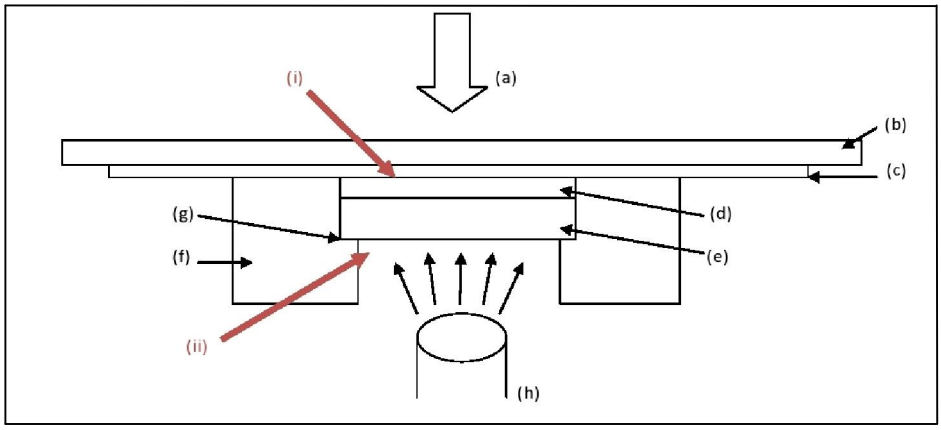

Figure 1: The resin ceramic sample

a) Finger pressure on the outer surface of the glass slab, b) Glass slab, c) Mylar strip, d) Resin cement with a thickness of 0.1 mm, e) IPS Empress CAD disc with a thickness of 0.5 mm, f) Teflon custom made mould, g) Internal shoulder at a depth of 0.6mm, h) Tip of the light curing device, i) The upper opening of a diameter 10 mm of used for the placement of the ceramic disc and resin cement both with a thickness of 0.6 mm (0.6 mm from the outer surface until the internal shoulder), ii) The lower opening used for curing of the resin cement underneath the ceramic disc and for removing of the sample from the mould after the resin cement curing.

2.2 Artificial aging process:

Thermocycling was the method of choice for conducting the artificial aging process (MPM instruments s.r.i , Bernareggio, Italia), each sample was aged for 3000 cycles, the time elapsed by each cycle was 90 seconds, each cycle consists of a first step of 30 seconds dwell time under a hot temperature water bath of $55C^0$, a second step of 30 seconds dwell time in a water bath with a temperature corresponding to that of the room temperature $25C^0$, a third step of 30 seconds dwell time under a cold temperature water bath of $5C^0$.

Measurements were repeated after each 1000 cycles (which is equivalent to one year of clinical service and the samples where measured for the change in color content. Then data was collected and analyzed statistically to reach final results.

2.3 Color measurment

Vita EasyShade Compact (VITA Zahnfabrik H.Rauter GmbH- Germany) was used to measure the change in the color content of the Ceramic\Resin samples immediately after curing of the cements, after 24 hours, after 1000 thermocycles, after 2000 thermocycles and finally after 3000 thermocycles and were stored in 37 C^0 distilled water and in between all the aging stages and measurements. Where measurements were taken in the middle part of the glazed surface of the sample and against white backgrounds where in each time, CIE Lab values were calculated and the a*, b* and L* values were recorded in order to compare the color parameters in the different phases of the aging process. The Easy Shade device was calibrated before every group measurement in order to standardize the reproducibility.

Color difference between different groups was then calculated according to the following equation:

$$\Delta E = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{½}$$

Then the ΔE of the each cement after each step was calculated where the values of a_1, b_1 and L_1 were constant in the equation above and corresponding to the mean value of the immediate measurement of each cement after curing, and the a_2, b_2 and L_2 were variable in the equation of each cement were they represented the after 24 hours, 1000 cycles, 2000 cycles and 3000 cycles respectively, then all the resin cements were compared for color stability according to the resultant values of the ΔE after each step of the procedure.

Quantitative data were presented as mean and standard deviation values. Data were explored for normality by checking the data distribution, calculating the mean and median values and using Kolmogorov-Smirnov and Shapiro-Wilk tests. Mixed anova was used to assess effect of cement type and aging over change in color ΔE followed by pair wise comparison tukey post hock test and simple main effect with Bonferroni correction The significance level was set at $P \leq 0.05$. Statistical analysis was performed with IBM® SPSS® Statistics Version 20 for Windows.

3. Results

3.1 The effect of aging on the change of color

After 24hours Rely X Unicem showed highest value of ΔE followed by Rely X Veneer followed by Rely X Ultimate and there was a significant difference between Rely X Ultimate and Rely X Unicem, and after 1000 cycles Rely X Unicem showed highest value of ΔE followed by Rely X Veneer followed by Rely X Ultimate and there was significant difference between Rely X Ultimate and other groups; however after 2000 cycles and 3000 cycles there was no significant difference between groups.

2.4 Effect of cement type

The results showed that within all the cements there was an increase in ΔE with increasing the number of thermal cycles and there was a significant difference between 24 hours and (1000 cycles, 2000 cycles and 3000 cycles), for the Rely X Ultimate and the Rely X Unicem there was a significant difference between 1000 cycles and (2000 cycles and 3000 cycles) as for the Rely X Veneer there was a significant difference between 1000 cycles and 3000 cycles.

Table 1: The mean and standard deviation of different materials at different aging stage

	Material	Mean	Std. Deviation
ΔE 24	ultimate	0.7885	.27214
	veneer	1.1488	.40948
	unicem	1.4357	.53456
ΔE 1000	ultimate	1.7155	.39535
	veneer	2.3305	.58156
	unicem	2.3974	.27613
ΔE 2000	ultimate	2.5845	.42915
	veneer	2.8066	.42033
	unicem	2.6643	.31121
ΔE 3000	ultimate	2.6749	.51906
	veneer	2.9026	.54808
	unicem	3.0606	.37337

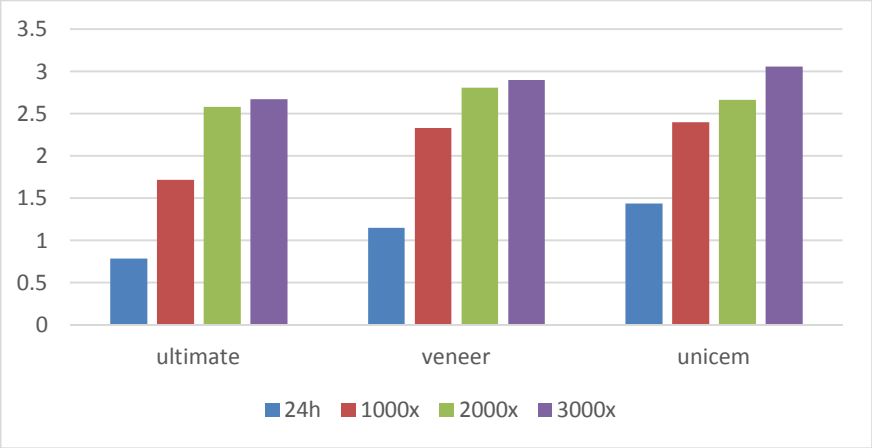

Figure 2: Mean value of different resin cements at different aging stage.

4. Discussion

This invitro study evaluated different light and dual-cure resin cements in terms of internal color stability. The focus was on the possible effect of this phenomenon on the appearance of ceramics. The translucent ceramic material is considered color stable but it may not be able to mask the discoloration of the underlying resin cement. [20]. So the resin cement plays a very important role in the indirect esthetics of the porcelain laminate veneer and in most of the time it can determine the success or failure of the esthetic rehabilitations, if used improperly discolorations won't be masked and it can affect the shade and translucency of the final esthetic restorations especially of thinner thicknesses.

Rely x ultimate is a dual cure, self-etching adhesive resin cement developed for glass ceramic and also polycrystalline ceramics cementation in mind. It was used in this study to measure its change in color content as it is a dual cured resin cement that utilizes a non-aromatic tertiary amine initiator which is claimed by the manufacturer to be more color stable than the aromatic tertiary amine that usually causes a change in the color due to its high reactivity with oxygen and other components, it enables dual curing while at the same time guaranteeing the long term color stability, so it is suggested by the manufacturer to be used in highly esthetic restorations [21].

RelyX Unicem is a *Self-Adhesive Universal* encapsulated, dual-cured resin cement. It was used in this study to compare its change in color content (as it utilizes an aromatic tertiary amine initiator) with the RelyX ultimate resin cement [22].

Rely X veneer cement is a permanent, light-cure only resin luting material. It was used in this study to compare its change in color content with the RelyX ultimate as it is light cured and contains a photosensitive aliphatic tertiary amine initiator that is much less reactive than the aromatic. RelyX veneer cement is very efficient in its light-curing ability, It contains a high efficiency photo-initiator system., This allows for excellent color stability of the cured cement. This feature is vitally important for highly esthetic veneer restorations. [23].

The thickness of cement was controlled at 0.1 mm in the study to imitate the maximum accepted cement thickness under porcelain laminate veneers clinically, because different thicknesses of cement might influence the final color of the veneer restoration, and this thickness was suitable for internal fit of the veneer restoration, as recommended by some studies for analyzing the stress distribution of the interface between ceramic and resin cement [24,25]. **Chang et al. (2009)**[9], and **Magalhães et al, (2013)**[26] used resin cement of 0.1 mm thickness..

This study used only one ceramic material, one ceramic color and one ceramic thickness for standardization. Ceramic samples were constructed from IPS Empress CAD blocks of shade A1. Leucite-reinforced ceramics are recommended when esthetics is the primary objective, so they are mostly used in porcelain laminate veneers restorations. [27]. As showed by **Al- Qahtani et al., (2012)**[28]**,** who concluded that a highly significant differences were demonstrated among the translucency of three ceramic materials, IPS Empress Esthetic Press, IPS Emax Press, and IPS ZirPress. These values were higher for IPS Empress Esthetic Press in comparison with the other two ceramic materials. The thickness of the samples was 0.5 mm (+- 0.05 mm) to allow the increase light transmission through the thin translucent ceramic to obtain a higher degree of conversion of the underlying resin cements [29].

Ivoclar Vivadent's (Bluephase) curing unit was used to cure the three resin cementswith a light intensity of a minimum value of 1,000 mW/cm^2 (and can reach to 1200 mW/cm^2) considered to be ideal. This value allows short exposure times of 10 seconds

to be consistently applied even if the conditions are not ideal [30].

The teflon mould was made similar to that used by **Klinic, et al. (2011)**[31] in order to obtain a uniform thickness of 0.1 mm for the resin cements in all the samples.

The Vita Easyshade compact digital spectrophotometer was used as a standard quantifying device i.e. a color spectrophotometer wasdevice which uses the ΔE from the Commission International d'Eclairage CIE (L*a*b*) color system This system inherits the advantage of being repeatable, sensitive, objective, universally accepted, and can measure small color differences[32].It was used by **Magalhaes et al (2014)**[33], to measure the color content of the resin\ceramic sample and by **Nuaimi et al. (2014)**[34] to investigate the color stability of 2 types of composite resin.

Accelerated Thermal aging was adopted in our study as the artificial accelerated ageing protocol used which although still cannot reliably simulate the clinical situation exactly but is considered as a harsh environment for testing conditions so it is frequently used as an aging procedure [35-37], A fact to be considered in the above is that it did not link comparative values between numbers of thermal cycles and time in oral environment so, The work was based on **Leibrock et al (1999)** [38] where he suggested that 1500 thermal cycles would be similar in physiologic normal conditions to the period of 1 and a half of year for resins. Also **Botega et al (2008)**[39]; simulated the use of temporary restorations for a period of three years, where each 1000 thermocycles represent the use of a fixed prosthesis for one year and **Stewardson et al. (2010)**[40]**,** claimed that the 500 cycles would only correspond to the number of cycles estimated to occur in less than 2 months in the mouth.

For the thermocycling process the temperature regime of 5^0–55^0C proposed in ISO 11405 recommendations (International Standards Organization, 1994) was used in this study [41], Dwell time is the period of time that the specimen is immersed in a bath of a particular temperature [40], It corresponds to a latency period, which is required by the oral capacity to reach its normal temperature again, after consuming hot or cold food and drink. Unfortunately, the choice of dwell times in experimental studies appears arbitrary and no effect of dwell time on results has been clearly established [42,43] inthis study a dwell time of 30 seconds was chosen simillar to other studies [44].

Thermocycling has been used by many researchers as a means of accelerated aging for measuring the color stability of resin based materials, **Kim et al., (2012)**[45] conducted a study on resin cements and thermocycling was the procedure of choice for the process of artificial accelerated aging.

The Effect of aging on the change of color Our results are explained by **Ghavam el al., (2010)**[46]**,** in his study he found that Aromatic tertiary amines used in self- cured resins are more likely to oxidize than aliphatic amines used in light-cured materials; therefore, light- cure cements are expected to have more color stability. They evaluated the effect of accelerated aging on the color and opacity of resin cements. The hypothesis was that the auto-polymerizing cements would show less color stability. They discovered that none of the groups showed significant differences in ΔE before and after aging (p>.05); ΔE remained in the range of clinical acceptance (ΔE<3.3), also all of the resin cements in our study resulted in a ΔE that remained in the range of clinical acceptance. As a clinical finding it was found that the studied cements can ensure color stability when used to cement porcelain laminate veneers. Also **Magalhaes et al (2014)**[33], had also similar results and statistical findings to the ones in our study, where the color content of the resin\ceramic specimens was measured, and a conclusion that there were no significant differences (after artificial accelerated aging) between dual- and light-cured modes of both cements like in our study where all of the resin cements showed a ΔE that remained in the range of clinical acceptance, although there was a significant statistical difference between the dual and light cured groups.

As for the **Effect of aging on the change of color**, **Magalhaes et al (2013)**[26]**,** she found that the yellowing of a material over time could be related to the exposition of Bis-GMA-based material to ultraviolet light and heat. As composite materials age, the water sorption characteristics of the resin monomers may contribute to differences in the degree of color stability. Also the study done by **Kim et al (2012)**[45]. Where Porcelain laminate disk samples were luted with resin cements. After thermocycling of each 1000, 2000 and 3000 cycle, specimens were measured by spectrophotometers and concluded that after thermocycling, color difference (ΔE) of all specimens were increased, but were not statically significant differences and that after 3000 thermocycles. All are within clinically acceptable ranges and that Color stability of different resin cements was comparatively excellent. **Klinic et al (2011)**[31] found similar results obtained by our study where he concluded that artificial accelerated aging of all resin cements showed statistically significant results and all of the resin cements showed varying degrees of discoloration after aging significantly, he also concluded that dual cured resin cements discolored more than light cured resin cements which

showed better color stability, however the actual color change was partially masked by the ceramic in the veneered group and were clinically acceptable.

The differences in the color change (ΔE) between all of the cements after artificial accelerated aging showed to be clinically acceptable although there was a statistical significance between the dual – cured (Rely X Ultimate) resin cement and the other cements.

5. Conclusions

Within the limitations of this study, the following conclusion could be drawn:

- The color change of the resin cements immediately after curing didn't showed no difference between the three resin cements regarding color change.
- The color measurement of the resin cements after 3000 thermal cycles showed that the three cements are clinically acceptable but the Rely X Ultimate had the best color stability (ΔE).

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