



Color Stability of Different Resin Cement Systems: A Spectrophotometric Analysis

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ABSTRACT

Aim: To evaluate the color stability of dual cure and light cure resin cements immersed in coffee solution at different time periods (baseline, 3 days and 1 week). **Materials and methods:** The color stability of three light curing (Variolink Esthetic (VLC); Rely X Veneer (RLC) and Mojo Veneer (MLC)) and three dual curing (Variolink Esthetic (VDC); Rely X Ultimate (RDC) and Breeze (MDC)) resin cements were tested. A total of sixty disks (10 mm diameter and 0.5 mm thickness) were prepared using a silicone mold (n=10). The specimens were subjected to baseline color measurement using Commission Internationale de l'Eclairage L*a*b* (CIELAB) color space system with the help of a spectrophotometer (LabScan XE, HunterLab, VA, USA). Following baseline color measurements, the specimens were immersed in a staining coffee solution. The color measurement was repeated after 3 days and 1 week of immersion in coffee solution. All the data collected were statistically analyzed using repeated measures of Analysis of variance (ANOVA, P<0.05). **Result:** After one week of storage in coffee solution, the mean color measurement scores in light cure group were found to be higher for RLC (1.22 ± 1.51) followed by VLC (0.85 ± 0.65) and NLC (0.68 ± 0.36) whereas in dual cure group, the mean color measurement scores were found to be higher for NDC (7.45 ± 4.50) followed by RDC (1.52 ± 1.24) and VDC (0.47 ± 0.34). Repeated measures ANOVA showed a statistically significant difference for light cure group (P=.004) at different time intervals but no significant difference with respect to dual cure group (P=.26). **Conclusion:** There was a significant difference observed between the light- and dual-cured cements over a different period of time. The color changes of both light- and dual-cure resin cements were within clinically acceptable limits except for the dual-cured Breeze cement.

Keywords: Resin cement; Light cured; Dual cured, Ceramics, Luting, Cementation, CIELAB, Staining; Color stability

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Introduction

Ceramic restorations have emerged as an ideal treatment method for patients in quest of superior esthetics in the anterior region (1). However, the optical properties of ceramic restoration could be affected by opalescence, fluorescence, translucency, surface texture, the number of porcelain firings, and also by the intrinsic factors such as color, translucency, and thickness of the underlying resin cement (2, 3). The critical aspect of restorative dentistry is the color stability of the resin cement, which may have a profound effect on the aesthetic appearance of thin ceramic laminate veneers.

The intrinsic factors of ceramic restoration are usually associated with resin cement material properties such as the chemical structure of resin cement (photoinitiator and filler type, the composition of the matrix), polymerization type, conversion rate, and the presence of unreacted monomer (4-6). The currently available resin cements used for luting or cementation are usually activated by visible light. The main advantages of light cured cements are their color stability and longer working time compared to dual- and chemically cured resin cements (7-9). The use of light cured cement facilitates easier removal of any excess material before light-curing and reduces operator's chair time during finishing of the restorations (10). Previous studies have demonstrated excellent color stability of light cured cements which is attributed to the absence of the aromatic tertiary amine as a self-curing catalyst, which is found to affect the color stability of these materials over time (7, 8, 11).

On the contrary, dual-cured resin cements combine some of the desirable advantages of light- and chemically cured resin cements (12). The advantages of dual-cured resin cements are superior mechanical properties including, flexural strength, elastic modulus, hardness and degree of conversion in comparison to the materials activated by a single mode of activation (either light- or chemical cured). Furthermore, they allow chemical curing in

deeper areas where light activation is practically impossible (12-14). However, dual-cured resin cements contain tertiary amine in their formulation, which is found to compromise the color stability of the final ceramic restorations over the long-term use (9).

Since a recent study claims that ceramic veneers' clinical life is about 94.4% even after 12 years' service (15). Moreover, their clinical failure rate is also very low (15). Thus, resin cement's color change during aging is a matter of grave concern. Therefore, the aim of this laboratory study was to evaluate the color stability of dual cure and light cure resin-cements immersed in coloring solution at different time periods (Baseline, 3 days and 1 week) with the help of a spectrophotometer, which has the ability to quantitatively measure the colour change (16). It was postulated that no difference in color variations will be detected considering the different types of resin cement tested under artificial aging.

Materials and methods

The resin cements used and the nomenclatures of the groups are presented in Fig.1. The complete lists of materials used in this study are presented in Table 1. Ten disk-shaped specimens (10 mm in diameter and 0.5 mm in thickness) from each study groups were prepared using a silicone mold (n=10, N=60). The upper surface of the mold was covered by mylar sheet to obtain a smooth surface. To obtain even distribution of cements and expel excess adhesive from the mold, a 300 g weight was applied for 20 seconds. The light- and dual-polymerizing resins were polymerized with a light-curing unit (Elipar, 3M ESPE, Seefeld, Germany) with a light intensity of 800 mW/cm². The light intensity was confirmed by a radiometer (Sybron, New Port Beach, CA, USA). The tip of the light-curing unit was applied on four overlapping surfaces of the specimens for 30 seconds each to ensure optimal polymerization. After polymerization, specimens' dimensions were confirmed using a digital caliper (Mitutoyo Corp., Tokyo, Japan) at four

different points on the disk, and the specimens were numbered from 1 to 10 on the opposite side of color measurement surface. The numbered specimens were stored in distilled water for 24

hours at room temperature before subjecting to color measurement and immersion in staining solution.

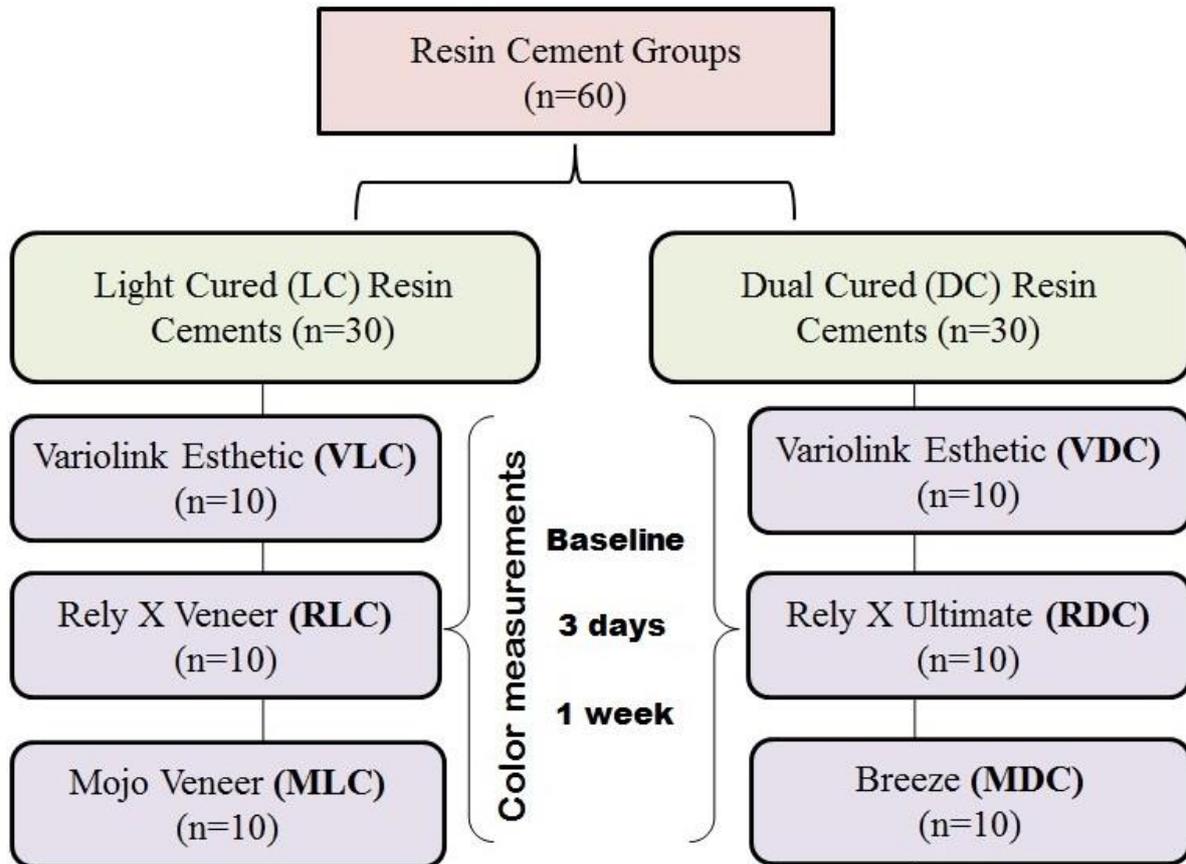


Fig. 1: Graphical representation of the study design.

Preparation of staining solution

The specimens stored in distilled water were dried using a blotting paper. Next, a coffee solution was used as a staining agent. The coffee solution was prepared according to previously published literature (17). A 500 ml of boiled distilled water was poured over 15 g of coffee powder (Nescafe, Nestle Brazil, Brazil) in a coffee cup. The prepared solution was stirred every 30 min for 10 s until it cooled down to oral temperature (37° C). The coffee was filtered using a sieve to remove any contaminants and stored in an airtight amber colored bottle. The coffee solution was prepared freshly every day until the conclusion of the immersion procedure. In addition, the prepared coffee solution was stirred daily for one minute to reduce the precipitation of the particles.

Specimen immersion procedure

The dried specimens were subjected to color measurement at baseline and later immersed in color-coded plastic containers filled with staining coffee solution (Figure 3.5). The color-coded containers allowed easy identification of the resin groups. The containers were closed with airtight cap to avoid any undue contamination of the specimens. The color measurement was repeated after 72 hours and after 1 week of immersion. For each color measurement, the resin cement specimens were removed from the container, rinsed with distilled water for 5 min to remove any surface impurities and later dried using a blotting paper.

Color measurement

The color measurement was made using Commission Internationale de l'Eclairege L*a*b*

(CIELAB) color space using bench top color spectrophotometer (LabScan XE, HunterLab, VA, USA). Three color measurements were made for each specimen and the total color (ΔE) were calculated using the below formula. $\Delta E = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2} \dots (1)$

The specimens were initially placed on a black background, with the unnumbered surface always facing the measurement site in order to prevent any potential effects of absorption from any other color parameters being measured.

National bureau of standards interpretation of CIE-LAB measurements

The values obtained by $L^*a^*b^*$ color space were converted to National Bureau of Standards (NBS) units to relate the changes to a clinical environment. The NBS interpretation of the data is presented in Table 3.2. The NBS value was calculated using the below formula (18).

$NBS \text{ unit} = 0.92 \times \Delta E \dots (2)$

Table 1: Materials used in the present study with their composition.

Name	Composition	Manufacturer
Variolink Esthetic (VLC)	Urethane dimethacrylate Methacrylate (UDMA) monomers, Inorganic fillers, initiators (Ivocerin and thiocarbamide hydroperoxide), stabilizers and pigments	Ivoclar Vivadent, Schaan, Liechtenstein
Variolink Esthetic (VDC)	Bisphenol A glycidyl methacrylate (BISGMA), barium glass filler, Dimethacrylates, silica, initiators (Tertiary amine and camphorquinone), stabilizers and pigments	
Rely X Veneer (RLC)	Bisphenol A glycidyl methacrylate (BISGMA), Triethylene glycol dimethacrylate (TEGDMA) Zirconia/silica and fumed silica, initiators (Tertiary amine and camphorquinone), stabilizers and pigments	3M ESPE, St. Paul, Minneapolis, MN, USA
RelyX Ultimate (RDC)	Methacrylate monomers Radiopaque alkaline (basic) fillers initiator components, initiators (Tertiary amine and benzoyl peroxide), stabilizers, rheological additives, fluorescence dye	
Mojo Veneer (MLC)	Poly(oxy-1,2-ethanediyl), α, α' -[(1-methylethylidene) di-4,1-phenylene]bis[ω -[(2-methyl-1-oxo-2-propen-1-yl)oxy]-1,6-hexanediyl bismethacrylate	
Breeze (MDC)	BISGMA, UDMA, TEGDMA, Hydroxyethyl Methacrylate (HEMA), 4-MET resins, silane-treated barium borosilicate glasses, silica with initiators, stabilizers and UV absorber, organic and/or inorganic pigments, opacifiers	Pentron, Orange CA, USA

Table 2: National bureau standards (NBS) interpretation of color changes (Judd, 1967)

NBS unit	Clinical interpretation	
0.0–0.5	Trace	Extremely slight change
0.5–1.5	Slight	Slight change
1.5–3.0	Noticeable	Perceivable
3.0–6.0	Appreciable	Marked change
6.0–12.0	Much	Extremely marked change
12.0 or more	Very much	Change to another color

Statistical analysis

All the data collected were statistically analyzed using the Statistical Package for Social Sciences ver. 18.0 (SPSS, Chicago, IL). The data were analyzed using repeated measures of Analysis of variance (ANOVA). The *post hoc* Bonferroni test and independent sample 't' test was applied to find out the statistical difference with respect to color measurements among the groups at different time periods. In all test, a p-value < .05 was considered as statistically significant.

Results

Table 3 presents the descriptive statistics of the color measurement values at different time-points. Among baseline, 3 days and 7 days groups, the mean value was found to be higher for VLC (1.34 ± 0.75), MDC (3.62 ± 2.20) and MDC (7.45 ± 4.50), respectively. Whereas, the RLC (0.4 ± 0.3), VDC (0.4 ± 0.3) and VDC (0.5 ± 0.3) showed lower color measurement values in the baseline, 3 days and 7 days' time-point, respectively.

Table 3: Mean distribution of the color measurements values at different time-points

Storage	Light cure groups			Dual cure groups		
	VLC	RLC	MLC	VDC	VDC	VDC
Baseline	1.3±0.7	0.4±0.3	0.9±0.4	0.7±0.3	0.9±0.8	1.0±0.5
3 days	1.0±0.6	0.7±0.7	0.8±0.3	0.4±0.3	1.1±0.7	3.6±2.2
7 days	0.8±0.6	1.2±1.5	0.7±0.3	0.5±0.3	1.5±1.2	7.5±4.6

Repeated measures ANOVA were applied to compare the color measurements of light cure and dual-cure group at different time intervals. Statistically significant difference was observed for light cure group ($p=0.004$) at different time intervals. However, no statistical difference was observed among the dual cure group ($p=0.26$). The *Post hoc* Bonferroni test was applied for pairwise comparison. Statistically significant difference was seen between baseline and 3 days ($p=0.012$), and between 3 days and 1 week ($p=0.001$) among light cure groups. Whereas, statistically significant difference was seen

between baseline and 1 week ($p=0.03$) among dual cure group.

Independent sample *t*-test was applied to compare the groups at different time intervals. At baseline, there was a significant difference observed between VLC and VDC ($p=0.034$); at 3 days, a significant difference was observed between VLC and VDC ($P=0.05$) and between MLC and MDC ($p=0.00$); at 1 week, a significant difference was observed between MLC and MDC ($p=0.00$) (Table 4).

Table 4: Comparison of the groups using an independent sample *t*-test

Time interval	Subgroups	Mean difference	p-value
Baseline	VLC * VDC	0.67	0.034*
	RLC * RDC	-0.60	0.07
	MLC * MDC	-0.04	0.20
3 days	VLC * VDC	0.54	0.05*
	RLC * RDC	-0.43	0.61
	MLC * MDC	-2.83	0.00*
1 Week	VLC * VDC	0.38	0.11
	RLC * RDC	-0.29	0.87
	MLC * MDC	-6.76	0.00*

N.B * means significant

Table 5 presents the National Bureau of Standards (NBS) interpretation of the color measurement values. The NBS units were clinically interpreted as extremely slight change (0.0–0.5), slight change (0.5–1.5), perceivable (1.5–3.0), marked change (3.0–6.0), extremely marked change (6.0–12.0) and change to another color (12.0 or more).

Table 5: Mean values of color change (ΔE) and the National Bureau of Standards (NBS) interpretation of values.

		Groups	Color measurement values	NBS units	Clinical interpretation
Baseline	Light cure	VLC	1.34	1.23	Slight change
		RLC	0.36	0.33	Extremely slight change
		MLC	0.96	0.88	Slight change
	Dual cure	VDC	0.67	0.61	Slight change
		RDC	0.96	0.88	Slight change
		MDC	1.0	0.92	Slight change
3 Days	Light cure	VLC	1.00	0.92	Slight change
		RLC	0.71	0.65	Slight change
		MLC	0.79	0.72	Slight change
	Dual cure	VDC	0.46	0.42	Extremely slight change
		RDC	1.15	1.05	Slight change
		MDC	3.62	3.33	Marked change
1 Week	Light cure	VLC	0.85	0.78	Slight change
		RLC	1.22	1.12	Slight change
		MLC	0.68	0.62	Slight change
	Dual cure	VDC	0.47	0.43	Extremely slight change
		RDC	1.52	1.39	Slight change
		MDC	7.45	6.85	Extremely marked change

Discussion

The CIE-LAB color analysis showed significant changes in the color measurement of the evaluated resin cement groups at different time period and hence the null hypothesis stated in the present study was rejected. The color measurement values in the present study ranged from 0.36 to 7.45 over a period of 1 week, regardless of the type of material. Controversies exist in the previous literature regarding the values of clinically noticeable color changes (19). According to (20), color values < 1.0 were considered undetectable by the human eye, values between 1.0 - 3.3 were considered visible by clinicians, but clinically acceptable, and color values > 3.3 were considered visible also by non-clinicians and for that reason, those values were not clinically acceptable. According to (21), the color values = 2.0, is perceptible

color change able to determine the optical effect of resin cements and is considered as the gold standard threshold. However, most studies report color values ≤ 3.3 as clinically acceptable (19).

Significant differences were observed between dual and light-activated cements, which were in agreement with the outcome of the previous study (22). However, the resin cements in the previous study were aged in distilled water and were not visually perceptible. In the present study, the studied cements had color values <3.3 which was acceptable clinically except for the dual-polymerizing resin cement, MDC which showed an increased color value of 3.62 at 3 days and 7.45 after 1 week. This is due to the presence of tertiary amines in their composition that acts as chemical initiators for the polymerization reaction. However, for

polymerization reaction, oxidation of the amine reactive groups is necessary and this may cause color changes (23).

Interestingly, VDC resin material showed lower color difference values compared to their light cured counterpart, VLC. The base paste of VDC contains both aliphatic and aromatic tertiary amines, and the catalyst paste contains benzoyl peroxide. The light-cured VLC is comprised of base paste only, while the dual-cured VDC are prepared by mixing base paste and catalyst. The aromatic tertiary amine remains intact in VLC group. The inclusion of two kinds of amine in dual-cured VDC could be the reason of lower color values of the dual-cured resin cement compared to light cured VLC. This finding was in agreement with the findings of other previous studies (24, 25).

Among the light cured cements, RLC showed lower values. RLC is a resin luting material containing a photosensitive aliphatic tertiary amine initiator that is much less reactive than the aromatic. Another reason is that RLC is efficient in its light-curing ability because of the photoinitiator system. On the contrary, the dual-cured RDC utilizes a non-aromatic tertiary amine initiator which is claimed by the manufacturer to be more color stable than the aromatic tertiary amine. This result was in agreement with the outcome of the previous study (26).

Another important finding of the present study was that the Variolink group and Mojo Veneer Cement showed lower color values from baseline to 1 week compared to other tested resin cements. However, the color values of MLC were less compared to VLC. According to the manufacturer, Mojo Veneer Cement is manufactured using Tru Kolor™ technology and remains color stable even after long period of use. (Elbieh *et al.*, 2012) evaluated the stain masking effect of Rely-X veneer cement and Mojo veneer cement. The authors found that both the resin cements were capable of just masking the color of lightly stained teeth. Furthermore, both the cements showed ΔE values above the critical limit. This finding was in

disagreement with the outcome of the present study.

Previous studies used different criteria for perceptibility of color values. However, the current study adopted the NBS system to determine the degree of color difference which offers absolute criteria by which color values can be converted to definitions with clinical significance (27). Although different beverages have been used to study the color stability of dental materials, however, the coffee is the most chromogenic agent in comparison to other staining substances (28, 29). The resin cement discs were immersed in the coffee solution for 3 days and 1 week which equaled to 4 years and 9.3 years, respectively in the oral cavity (30). The uniqueness of the oral environment could affect the color result significantly. Most of the color stability studies have investigated the *in-vitro* effect of cements. Here it becomes necessary to consider the patients' opinions on the aesthetic result. Future studies should focus on *in-vivo* investigations and patients' opinions.

Conclusion

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

- a) The color changes of both light- and dual-cure resin cements were within clinically acceptable limits except for the dual-cured MDC cement.
- b) There was a significant difference observed between the tested light- and dual-cured cements over different period of time.
- c) Variolink group (VLC) and Mojo Veneer Cement (MLC) showed lower colour difference values over a different period of time compared with other tested material.
- d) Dual cured MDC group demonstrated extremely marked change values

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