

Effect of Accelerated Aging on the Color Stability of Dual-Cured Self-Adhesive Resin Cements

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Purpose: The effect of accelerated aging on color stability of various dual-cured self-adhesive resin cements were evaluated in this study.

Materials and Methods: Color stability was examined using three different brands of dual-cured self-adhesive resin cements: G-CEM LinkAce (GC America), MaxCem Elite (Kerr), and PermaCem 2.0 (DMG) with the equivalent color shade. Each resin cement was filled with Teflon mold which has 6 mm diameter and 2 mm thickness. Each specimen was light cured for 20 seconds using light emitting diode (LED) light curing unit. In order to evaluate the effect of accelerated aging on color stability, color parameters (Commission Internationale de l'Eclairage, CIE L*, a*, b*) and color differences (ΔE^*) were measured at three times: immediately, after 24 hours, and after thermocycling. The L*, a*, b* values were analyzed using Friedman test and ΔE^* values on the effect of 24 hours and accelerated aging were analyzed using t-test. These values were compared with the limit value of color difference ($\Delta E^*=3.7$) for dental restoration. One-way ANOVA and Scheff's test ($P<0.05$) were performed to analyze each ΔE^* values between cements at each test period.

Result: There was statistically significant difference in comparison of color specification (L*, a*, b*) values after accelerated aging except L* value of G-CEM LinkAce ($P<0.05$). After 24 hours, color difference (ΔE^*) values were ranged from 2.47 to 3.48 and L* values decreased and b* values increased in all types of cement and MaxCem Elite had high color stability ($P<0.05$). After thermocycling, color change's tendency of cement was varied and color difference (ΔE^*) values were ranged from 0.82 to 2.87 and G-CEM LinkAce had high color stability ($P<0.05$).

Conclusion: Color stability of dual-cured self-adhesive resin cements after accelerated aging was evaluated and statistically significant color changes occurred within clinically acceptable range.

Key Words: Aging; Color; Resin cements

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Introduction

Various types of esthetic prosthesis are currently produced due to high demand for esthetics. For successful esthetic prosthesis, underlying cement color change should be carefully considered beside prosthesis¹⁻⁴. Resin cements have been the first choice in esthetic prosthesis due to their advantages of color stability, stronger adhesion to tooth and better mechanical properties⁵.

Resin cements are classified into three subgroups according to their curing mechanism: the self-cured, light-cured and dual-cured⁶. In previous studies, the effect of amine in self-polymerization on discoloration was determined and light cured resin cement was preferred over self-cured and dual-cured resin cements in esthetic prosthesis^{1,2,7-9}. However, due to the limitation of curing depth of light-polymerization and material itself, dual-curing resin cements have been developed¹⁰. Resin cements are alternatively categorized according to the tooth surface pre-treatment methods: etch-and-rinse, self-etching, and self-adhesive resin cement¹¹. Self-adhesive resin cement is preferred due to its simple procedure, which requires no pre-treatment^{11,12} and uses dual-cured system. There were many studies on color stability of resin cement, yet only a few studies evaluated self-adhesive resin cement and they concluded that self-adhesive resin cements had lower color stability compared to conventional resin cement^{13,14}.

Discoloration of dental cement depends mainly on intrinsic factors rather than extrinsic factors on color stability¹⁵⁻¹⁷. Intrinsic discoloration occurs as a result of discoloration of the resin material itself and it affects the composition of the resin matrix and filler, particle size, activator/initiator components, their ratios and percentage of remaining carbon-carbon double bonds¹⁵⁻¹⁸. A number of accelerated aging has been introduced as a method to evaluate intrinsic discoloration^{1,13,19}. Among these, thermocycling simulates the oral

cavity environment to examine the properties of dental material^{20,21}. According to Wendt et al.²⁰, thermocycle occurs at temperature range between the lowest (4°C~8°C) and the highest (45°C~60°C). And Gale and Darvell²¹ suggested that the 10,000 thermocycles are equivalent to approximately 1 year. In this study, thermocycling (2,000 cycles; 5°C~55°C, 15 seconds dwelling time and 3 seconds waiting time) in thermal circulation water bath demonstrates the use of cements for a period of 3 months. There have been a few studies evaluating the color stability of thermocycled resin cements while no study was conducted to evaluate the color stability of self-adhesive resin cements. This *in vitro* investigation was performed to measure the effect of thermocycling on color stability of dual-cured self-adhesive resin cements.

Materials and Methods

1. Specimens Preparation

In this study, three different brands of dual-cured self-adhesive resin cements: G-CEM LinkAce (GC America, Alsip, IL, USA); MaxCem Elite (Kerr, Orange, CA, USA); and PermaCem 2.0 (DMG, Hamburg, Germany) were used which exhibited equivalent transparent shade (Table 1). Ten specimens were prepared per cement to evaluate the color stability of each resin cement. Resin cements were mixed according to the manufacturer's instruction and filled in a Teflon mold which was 6 mm in diameter and 2 mm in thickness¹. The Teflon mold was covered with celluloid strip (URIDENTAL, Seoul, Korea) and slide glass (Marienfeld, Lauda-Königshofen, Germany)²² and lightly pressed with fingers to remove the excess materials. Light emitting diode (LED) curing light with light intensity of 1,000 mW/cm² was used to cure both sides for 20 seconds. Afterward, the specimen was separated from the mold. Color specification (Commission Internationale de l'Eclairage, CIE L*, a*, b*) values

Table 1. The materials used in the study and their composition according to their manufactures

Resin cement (Lot No.)	Manufacture	Resin matrix	Initiator system	Resin shade
G-CEM LinkAce (1309191)	GC America (Alsip, IL, USA)	Urethanedimethacrylate (UDMA), dimethacrylate	Proprietary selfcuring redox-activator, camphorquinone	Clear
MaxCem Elite (5150785)	Kerr (Orange, CA, USA)	Glycerol dimethacrylate dihydrogen phosphate (GPDM), 2 hydroxyethyl-methacrylate (HEMA)	Benzoyl peroxide and amine free redox initiator, camphorquinone	Clear
PermaCem 2.0 (709149)	DMG (Hamburg, Germany)	Bis-phenol-A-bis-(2-hydroxy-3-methacryloxypropyl)ether (Bis-GMA), triethylenglycodimethacrylate (TEGDMA)	Benzoyl peroxide, tertiary amine, camphorquinone	Clear

of specimens were examined after light curing by using Shade Eye-NCC dental chromameter (Shofu Inc., Kyoto, Japan). The colors determined after the specimens preparation were kept in a light-tight chamber at a constant-temperature (37°C) and 100% relative humidity (J-NBT; JISICO, Seoul, Korea) for 24 hours as well as after thermocycling (2,000 cycles; 5°C and 55°C, 15 seconds dwelling time²³ and 3 seconds waiting time), which simulated the use of cements for a period of 3 months²¹.

2. Measurement of Color Parameters

Color specification (CIE L*, a*, b*) values for each specimen were examined three times using Shade Eye-NCC dental chromameter. The effects of external factors were eliminated by measuring color specification of specimen's center in the same place at the same time. Color specification (L*, a*, b*) consists of lightness variable of L* and chromaticity indices a* and b*, represented in two of transverse axis²⁴. L*, a*, b* values were measured to calculate the effect of 24 hours immersion and 2,000 times thermocycling on color change in ΔE^* values using following formula.

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

$$\Delta L^* = L_1 - L_0$$

$$\Delta a^* = a_1 - a_0$$

$$\Delta b^* = b_1 - b_0$$

L₀, a₀, b₀: color values determined before accelerated aging

L₁, a₁, b₁: color values determined after accelerated aging

3. Statistical Analysis

Color stability of each cement was evaluated and compared immediately, 24 hours after curing and after thermocycling using color specification (L*, a*, b*). All statistical analysis was performed using IBM SPSS Statistics version 21.0 (IBM Co., Armonk, NY, USA). The normal distribution was verified using the Kolmogorov-smirnov test. Friedman test was conducted to compare the L*, a*, b* values at each test period and paired sample t-test was used to compare the ΔE^* values before and after 24 hours and thermocycling. Inter-comparison of ΔE^* values between cement at each test period were used to analyze statistically using one-way ANOVA and post hoc Scheff's test (P<0.05).

Result

Each cement was measured three times: immediately, 24 hours after polymerization, and after thermocycling accelerated aging using Shade Eye-NCC dental chromameter. The means and standard deviations of L*, a*, b* values were calculated for each specimen (Table 2, Fig. 1~3).

1. L*, a*, b* Values

L* values representing brightness, had a tendency

Table 2. L*, a*, b* value of self-adhesive resin cements according to testing time

Resin cement		Immediate	24 hours	Thermocycling	P-value
G-CEM LinkAce	L*	99.68±0.58 ^b	99.56±0.63 ^b	99.88±0.10 ^a	0.196
	a*	-0.12±0.19 ^a	-0.98±0.21 ^b	-1.26±0.22 ^c	0.000
	b*	-15.21±0.75 ^b	-12.45±0.53 ^a	-12.26±0.31 ^a	0.000
MaxCem Elite	L*	99.90±0.00 ^a	99.02±0.53 ^c	99.77±0.41 ^b	0.000
	a*	0.33±0.12 ^a	0.38±0.11 ^a	-0.23±0.06 ^b	0.000
	b*	-16.43±0.65 ^b	-14.21±0.59 ^a	-16.71±0.77 ^b	0.000
PermaCem 2.0	L*	99.69±0.55 ^a	97.63±1.29 ^b	95.67±0.70 ^c	0.000
	a*	-3.76±0.45 ^c	-2.59±0.35 ^b	-1.75±0.15 ^a	0.000
	b*	1.62±0.68 ^c	3.57±0.41 ^b	5.00±0.45 ^a	0.000

Values are presented as mean±standard deviation.

Means with different upper-case letters in each row showed significant differences (P<0.05).

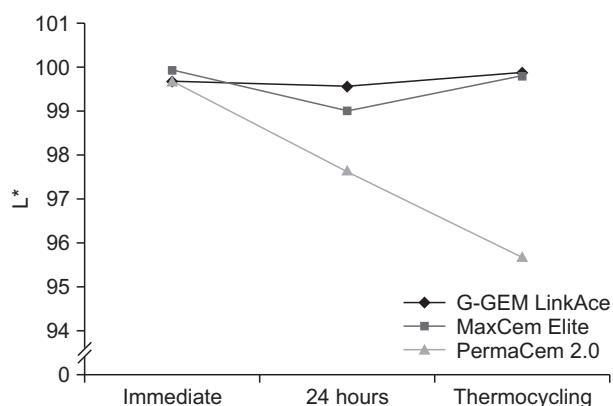


Fig. 1. L* of self-adhesive resin cements according to testing time.

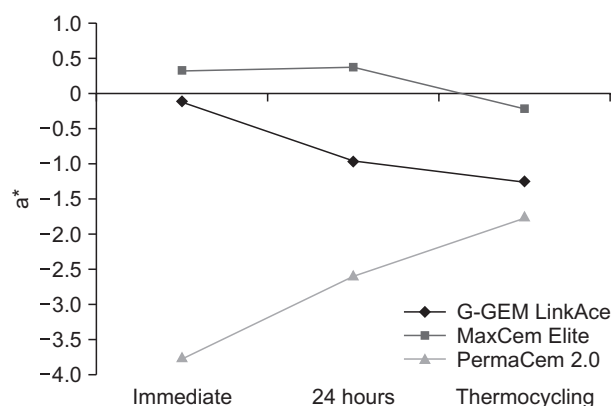


Fig. 2. a* of self-adhesive resin cements according to testing time.

to decline in all cements and statistically significant difference were detected in MaxCem Elite and PermaCem 2.0 (P<0.05). After the thermocycling accelerated aging, brightness of G-CEM LinkAce and MaxElite increased. In contrast, PermaCem 2.0 decreased in brightness and statistically significant difference was noted (P<0.05).

a* values, representing redness index, had a tendency to decline towards color green in G-CEM LinkAce and incline towards color red in both MaxCem Elite and PermaCem 2.0. Except MaxCem Elite, statistically significant difference was resulted in G-CEM LinkAce and PermaCem 2.0 (P<0.05). After thermocycling accelerated aging, a* values had a tendency to decline toward color green in G-CEM LinkAce and MaxCem Elite although

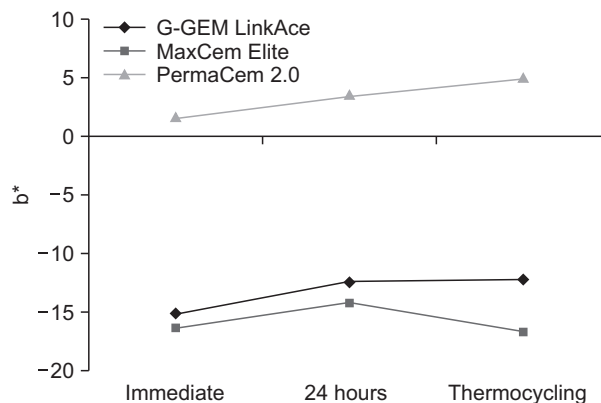


Fig. 3. b* of self-adhesive resin cements according to testing time.

PermaCem had a tendency to increase. All cements showed statistically significant difference (P<0.05).

b* values, representing yellowness index, had a tendency to incline towards color yellow in

Table 3. ΔE^* value of self-adhesive resin cements according to testing time

Resin cement	After 24 hours	After thermocycling	P-value
G-CEM LinkAce	3.06±0.53 ^{b,c}	0.82±0.60 ^a	0.000
MaxCem Elite	2.47±0.61 ^{a,b}	2.87±0.63 ^b	0.014
PermaCem 2.0	3.48±0.92 ^c	2.81±1.06 ^b	0.073
P-value	0.000	0.000	

Values are presented as mean±standard deviation.

Means with different upper-case letters in each column showed significant differences ($P < 0.05$).

all tested cements after 24 hours immersion and statistically significant difference was found. After thermocycling accelerated aging, b^* values had a tendency to decline towards color blue in MaxCem Elite and had a tendency to incline towards color yellow in G-CEM LinkAce and PermaCem 2.0. Both MaxCem Elite and PermaCem 2.0 showed statistically significant differences after the aging.

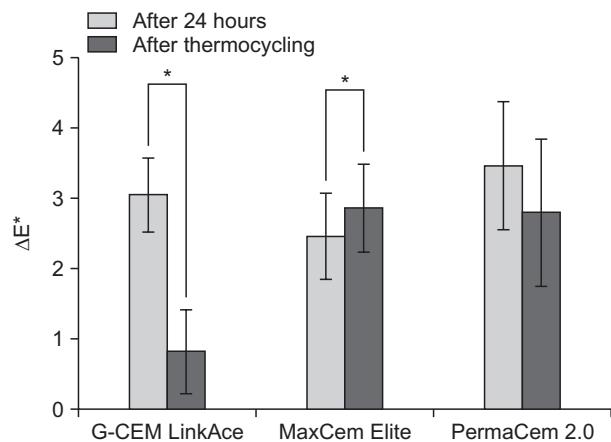
2. Color Difference (ΔE^* Values)

L^* , a^* , b^* values were measured and substituted into formula to calculate ΔE^* values, color difference (Table 3, Fig. 4).

Color difference (ΔE^*) values were resulted from 0.82 to 3.48. After 24 hours immersion, MaxCem Elite had the lowest ΔE^* values and G-CEM LinkAce was the second lowest followed by PermaCem 2.0. No statistically significant difference was found between MaxCem Elite and G-CEM LinkAce and between G-CEM LinkAce and PermaCem 2.0. After thermocycling accelerated aging, G-CEM LinkAce had the lowest ΔE^* values and Maxcem Elite was the second lowest followed by PermaCem 2.0. No statistically significant difference was found between MaxCem Elite and PermaCem 2.0.

Discussion

Color of abutment, types and thickness of prosthesis and color stability of resin cement require

**Fig. 4.** Color difference (ΔE^*) of self-adhesive resin cements according to testing time. * $P < 0.05$.

thorough consideration for esthetic dental prosthesis¹⁻⁴). In this study, CIE L^* , a^* , b^* values which represent color specification values were established by Commission Internationale de l'Eclairage (CIE), were used to measure color stability of resin cements. Color specification (L^* , a^* , b^*) consists of lightness variable of L^* and chromaticity indices a^* and b^* , represented in two of transverse axis²⁴). Brightness is measured on a black (0) to white (100) scale. Positive a^* values represent red and negative a^* values represent green while positive b^* values represent yellow and negative b^* values represent blue²⁴). Color differences were calculated using measured L^* , a^* , b^* values and substituted to ΔE^* calculating equation. As the color difference value increases, color stability decreases and it is distinguishable with eyes if the color difference is 1~2²⁵). Clinically acceptable limit of color difference (ΔE^*) for use of tooth restoration is approximately 3.3~3.7 and the opinions vary among authors^{8,26,27}).

In the present study, the color differences were measured for three different brands of dual-cured self-adhesive resin cements with equivalent transparent shade after 24 hours and after thermocycling. The obtained values ranged from 0.82 to 3.48, and were considered clinically acceptable²⁷). This value may decrease in the event of

measurement performed beneath a restoration and when thickness of sample decreases. According to the previous studies on color difference of cement beneath a restoration³⁾, color differences decreased when resin cements were adhesive to porcelain disk and 1 mm thick porcelain disk provided a masking effect to decrease the color differences^{3,9)}. In contrast, 0.5 mm thick porcelain disk may be affected by underlying tooth color and cement color. Therefore the cement and underlying tooth color in adhesive dentistry must be taken into consideration. In addition, thickness of resin cement samples used in the present study were thicker than those used in clinical. Cement space in porcelain restoration is approximately $150 \mu\text{m}$ ^{28,29)} while 2 mm thick specimens were used to conduct *in vitro* experiment can be stated as a limitation of this study. As the thickness of specimen increases, greater color change occurs³⁰⁾. Thus, it is acknowledged that the color differences measured in the present study may have been less effective than the actual color change.

In the present study, intrinsic discoloration of resin cement was evaluated using thermocycling, which stimulates the use of cements for a period of 3 months. In previous study about color stability of accelerated aging of resin cement by Turgut and Bagis¹³⁾, specimens were exposed to ultraviolet light and water spray after accelerated ageing and L^* and a^* values decreased. Although b^* value increased and statistically significant differences were observed, these were within clinically acceptable range. Hekimoğlu et al.¹⁾ found that color stability after accelerated aging was not depended on exposure time and cement type and no significant difference was observed. In addition, it was found that light-cured resin cement had better color stability than self-cured and dual-cured cement. Kim et al.³¹⁾ reported that color change after thermocycling was clinically acceptable range and no significant difference was noted between number of cycle (1,000, 2,000, 3,000 cycle) of thermocycling

treatment in resin cement. In the present study, after 24 hour immersion and thermocycling, color changes were evident. Color difference of MaxCem Elite was greater after thermocycling than after 24 hours and G-CEM LinkAce was greater in 24 hours after than after thermocycling. These results were similar to findings in previous study which states that color difference was not depended on time¹⁾ and each resin cement resulted in different pattern³²⁾. L^* values of PermaCem 2.0 had a tendency to decrease as application time passes ($P < 0.05$). In all resin cements, L^* values decreased after 24 hours immersion and this result pattern was similar to previous study³⁾. b^* values were increased and had a tendency to turn yellow with statistically significant different, except MaxCem Elite. According to Ferracane et al.³³⁾, b^* values increased to turn yellow in the bis-phenol-A-bis-(2-hydroxy-3-methacryloxypropyl) ether (Bis-GMA) resin and this tendency seems to be accelerated by ultraviolet radiation exposure, decomposition of camphorquinone and reaction of untreated methacrylate. In previous study⁷⁾, b^* values increased differently depending on the resin cement.

Color differences between cements, which were measured at the same period, were compared and color stability was higher for MaxCem Elite in after 24 hours and G-CEM LinkAce in after thermocycling. MaxCem Elite had significantly better color stability and it was patented for benzoyl peroxide (BPO) and amine free redox initiator technology. G-CEM LinkAec was also stabled after thermocycling due to HEMA, hydrophilic monomer, free technology. Also, it was used urethanedimethacrylate (UDMA) as monomer and was inferred to have better color stability^{13,16)}. The color difference of PermaCem 2.0 was higher since it is a Bis-GMA based matrix and it contains BPO and tertiary amine as self-cured initiator. Chemically cured resin with BPO initiator is unstable, especially at elevated temperatures. This instability leads to a polymerization of resin pastes

during storage, even in the absence of tertiary amine activators, and discoloration may occur³³. Oxidation of tertiary amine accelerators also has an effect on discoloration¹⁵⁻¹⁸. Especially tertiary amine related to self-curing is aromatic amine, which has a higher chance of oxidation than amines inducing light curing reaction⁴. In addition, there are various factors influencing discoloration of resin cements. In the previous studies^{16-18,33}, oxidation of reacted carbon-carbon double bonds was one of the factor causing discoloration^{16-18,33} and colored peroxide compounds tend to absorb light in the violet range of the visible light spectrum, which is interpreted as yellow color by human observers². As a number of unreacted monomer increases, it results in increased discoloration³⁴. This study cannot find the direct effect of low initial degree of polymerization on discoloration of resin cement. For that reason, additional experiment is proposed to examine its effect.

Conclusion

Three self-adhesive resin cements were evaluated for color change followed by accelerated aging. Within the limitations of this study, the accelerated aging significantly influences the color stability of self-adhesive resin cements, the magnitudes of the mean color differences were at an acceptable perception level and were considered clinically acceptable. All samples became slightly darker and turned more yellow from baseline to 24 hours after polymerization.

Conflict of Interest

No potential conflict of interest relevant to this article was reported.

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