

Original Article

Effect of Resin Cement Type on the Final Color of High Translucent Zirconia Before and After Thermocycling: An In Vitro Study

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Abstract

Aim: Advancements in dental cements were made to enhance bonding to zirconia. However, their effect on the color stability of the restoration is still unclear. The purpose of this study was to compare the effect of amine-free MDP-containing dual-cured self-adhesive resin cement versus light-cured resin cement on the final color of monolithic high translucent zirconia before and after thermocycling. **Subjects and methods:** Twenty high translucent zirconia discs, shade A2 (0.5 mm thickness, 10 mm diameter) were obtained, air abraded and randomly divided in two groups (n=10) according to the cement type used; Group CH: cemented by choice 2 (light-cured resin cement; control), Group ZC: cemented by ZR-Cem (amine-free MDP-containing dual-cured self-adhesive resin cement; intervention). Specimens were cemented to PMMA discs and subjected to thermocycling. Color was assessed after cementation and after thermocycling using laboratory spectrophotometer. After testing for normality, collected data revealed parametric distribution and were expressed as mean and standard deviation. Data were statistically analyzed at a level of significance ($P \leq 0.05$). **Results:** After cementation, color change was significantly lower in Group ZC ($\Delta E = 2$) than in Group CH ($\Delta E = 3.8$). However, after thermocycling, it was significantly higher in Group ZC ($\Delta E = 3.63$) than Group CH ($\Delta E = 2.47$), being within the clinically accepted range. **Conclusion:** Resin cement type and aging affect the final color of thin high translucent zirconia restorations. Light-cured resin cement has more resistance to artificial aging compared to amine-free MDP-containing dual-cured resin cement.

Keywords: Color; MDP; resin cement; thermocycling; high translucent zirconia.

I. INTRODUCTION

High-translucent (3Y-TZP) zirconia has been used as monolithic restorations in esthetic areas. It offered high flexure strength, durability and good optical properties, allowing more conservative preparation while eliminating the unwanted complication of veneer chipping.¹ Its translucency allowed its use as laminate veneers, permitting adequate resin-cements polymerization.²

Its high translucency was achieved by reducing the grain size to nanoscale (400-700 nm i.e. less than visible wavelength), which decreased light scattering.^{2,3} Additionally, reducing alumina

concentration (<0.05wt %) and size, increasing lanthanum-oxide to 0.2% mol., alumina particles (scattering centers) repositioning on the grain boundaries, reducing residual pores and impurities, increasing the sintering temperature and decreasing the sintering time enhanced its translucency.^{1,2,4,5}

However, being chemically inert, bonding to zirconia still presents a challenge.⁶ Light-cured resin-cements were recommended for luting thin esthetic restorations due to their color stability and controllable working time.⁷ Despite the color change, dual-cured resin-cements were desirable, due to their high mechanical properties and

insurance of complete polymerization in inaccessible areas.⁸

Amine-free MDP-containing dual-cured resin-cement (ZR-Cem) achieved strong bond to zirconia, dentin and enamel ensuring superior retention and marginal integrity.⁹ Being amine-free offered long lasting color stability.⁹ The presence of phosphate ester monomer; 10-methacryloyloxydecyl-dihydrogenphosphate (MDP), which can chemically react with ZrO₂ was also beneficial.¹⁰ MDP presents a terminal functional group, which reacts with Zr and forms P-O-Zr bonds.¹⁰ Researchers found that adhesion between 10-MDP and zirconia was not only ionic but also hydrogen bonding.¹¹

Color of thin esthetic restorations are affected by color stability of resin-cements.^{12,13} Perceptibility and acceptability are both important factors. Perceptibility refers to the patient's ability to see color difference between their natural teeth and restorations.¹⁴ Acceptability is the degree to which patients are satisfied with their restoration color.¹⁴ Color change threshold ranged from 1.0 to 3.7 for perceptibility and from 1.7 to 6.8 for acceptability.⁶

To evaluate color stability in-vitro, various methods were introduced to simulate clinical aging. Thermocycling is a widely used, controlled and reliable method that simulates intra-oral thermal changes.¹⁵ It was suggested that 10,000 thermocycles corresponded to one clinical service year,¹⁶ hence; a minimum of 5,000 cycles is recommended.¹⁷ For color assessment, instrumental techniques, such as laboratory spectrophotometer, provide highly accurate, objective and reproducible quantitative results.¹⁸

Esthetic outcome of newly introduced translucent zirconia cemented with recent resin-cements is still unclear. Thus, the aim of the present study was to assess the effect of resin-cement type; either amine-free MDP-containing dual-cured resin-cement or light-cured resin-cement, on the color of thin monolithic high-translucent zirconia before and after thermocycling. The null hypothesis was that the type of resin-cement would have no effect on zirconia color before and after thermocycling.

II. MATERIALS AND METHODS

Sample size calculation was performed using G-power software ver-3.1.9.2 (Heinrich-Heine-Universität Düsseldorf, Düsseldorf, Germany) with 0.05 alpha and 80% power, rendering 10 samples in each group.

• Zirconia Discs Preparation

Twenty uniform disc-shaped specimens (12-mm diameter, 0.6-mm thickness) were prepared from high-translucent zirconia (KATANA Zirconia HT13-T14, Kuraray Noritake Dental, Japan) with shade corresponding to A2 to adhere to one of the most prevalent tooth shades.¹⁹

For standardization, zirconia cylinders were milled then sliced using linear precision cutting machine (IsoMet 4000, Buehler, USA) at low-speed (2350 rpm) under copious water to avoid heat generation. The discs were 25% larger than the desired final size to compensate for sintering shrinkage. Sintering was then proceeded (Zirconia Sintering Furnace, WEISSEN, China) according to the manufacturer's instructions to avoid any deviation in the properties or color of zirconia.²⁰

For better smoothness, the outer surface of all specimens was polished (**Figure 1**) in one direction, using diamond polishing kit (DIASYNT PLUS & DIACERA polishing kit, EVE, Germany). After polishing, the specimens were checked using the digital caliper (APT TM52000 LCD, China) to verify the final 0.5-mm thickness and 9.5-mm diameter.

To clean contaminants, increase surface roughness and enhance bonding, the disc's unpolished surface was air-abraded (BEGO Protempomatic machine, China) using 50- μ m Al₂O₃ at 0.2-MPa pressure for 20 seconds at 10-mm distance.^{21,22}

Polishing and air-abrasion were performed manually to simulate the clinical practice, by the same operator to ensure standardization. Specimens were cleaned in distilled water in digital ultrasonic cleaner (Codyson Ultrasonic Cleaner CD-4830, China) for 10 minutes and air-dried with oil-free air spray for 1 minute to ensure the removal of any residues that might affect the outcome.¹³

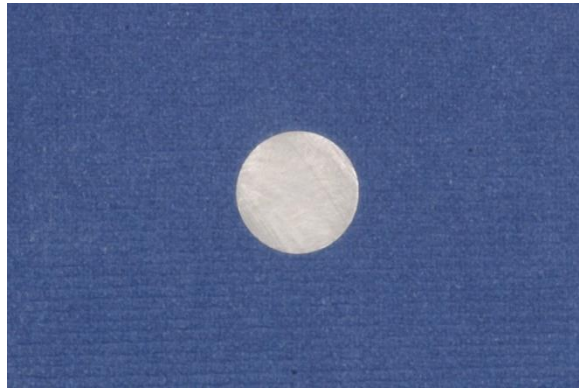


Figure 1: Sintered zirconia disc

- **PMMA substrates preparation**

Twenty standardized PMMA acrylic resin (Yamahachi PMMA Discs, Japan) cylinders (shade A2) were sliced as previously mentioned at 5-mm thickness to eliminate any background effect that might adversely affect the outcome.²³ Their diameter (10 mm) was slightly larger than zirconia discs to facilitate color measurement and excess cement removal.²⁴

To enhance bonding, all substrates were air abraded (BEGO Protempomatic machine, China) as previously mentioned for 10 seconds,²⁴ then ultrasonically cleaned in distilled water for 10 minutes and air-dried.²⁵

- **Randomization**

Discs and substrates were individually placed in a numbered plastic bag to protect their surfaces from scratching and retain the sandblasted surface clean and grease-free. A random sequence was generated for zirconia discs using (<http://www.random.org>)

software to allocate the specimens to the tested groups (1:1 ratio). Another randomized sequence was generated to distribute the substrates. Allocation concealment was employed to eliminate bias.

- **Specimen-holder**

Custom-made split cylindrical Polytetrafluoroethylene (PTFE; Teflon) specimen-holder was fabricated to hold the zirconia disc over its complementary substrate during pre-cementation color assessment and during cementation. The holder comprised a central hole to accommodate the zirconia-substrate assembly, with an external metallic ring to hold and secure the split halves together. The mold thickness (5.6 mm) was made to accommodate the thicknesses of zirconia disc (0.5 mm), substrate (5 mm) and cement layer to help standardize the cement thickness at 0.1 mm as recommended for optimum fit of thin restorations (**Figure 2**).

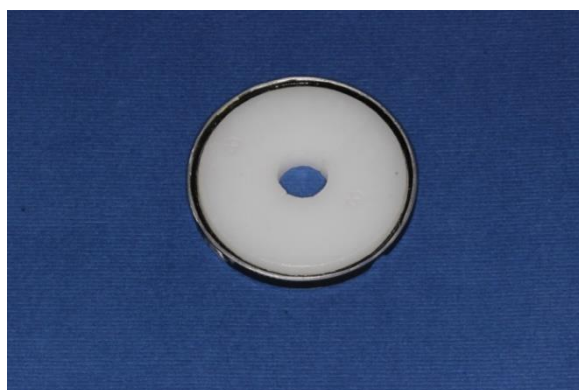


Figure 2: The assembled specimen-holder

- **Pre-cementation assessment**

Color was measured before cementation to serve as baseline reference, where each zirconia specimen was secured over its substrate in the specimen-holder, then placed in laboratory spectrophotometer (Cary 5000 UV-Vis-NIR, Agilent, USA), which was calibrated before each measurement. Three measurements were taken for each specimen and were expressed according to CIELAB System.

- **Cementation**

Group CH: The substrate was secured in the specimen-holder resting on a glass slab. All-Bond Universal adhesive (Bisco, Chicago, USA) was applied to the substrate and zirconia disc according to manufacturer's instructions, then air-dried and light-cured for 10 seconds (Elipar deep cure L curing device, 3M ESPE, USA) using 1470 mw/cm light intensity.

Choice2 veneer cement - Translucent shade (Bisco, Chicago, USA) was applied to the substrate then zirconia disc was seated with gentle pressure till the disc flushed with the specimen-holder's upper edge. Tack curing was done and excess cement was removed gently using an explorer. Final light-cure (40 seconds) was done according to the manufacturer's instructions. Any excess cement remnants at the zirconia-substrate boundaries were removed by using diamond polishing kit (DIASYNT PLUS & DIACERA polishing kit, EVE, Germany) (**Figure 3**).

Group ZC: ZR-Cem - Translucent shade (Premier, USA) was applied similar to Group CH, but without adhesive layer application.

All cemented specimens were checked using digital caliper to verify their final thickness ($5.6 \pm 0.01\text{mm}$) and stored in distilled water for 24 hours to allow maximum degree of cements conversion.²⁶



Figure 3: Final cemented specimen

- **Post-cementation assessment**

Color assessment was performed as previously mentioned. The color change (ΔE) of each specimen was obtained,

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

Where L^* represents lightness-darkness, while a^* and b^* represent redness-greenness and yellowness-blueness, respectively.

- **Thermocycling**

All specimens were thermo-cycled (Robota, BILGE, Turkey) for 5000 cycles in distilled water between 5 and 55 °C with 25 seconds dwell time, to simulate 6-months intraoral conditions.²⁷

- **Post-thermocycling assessment**

Final color assessment was done as previously discussed.

- **Statistical analysis**

The collected data were analyzed by an expert statistician who was blinded to the groups. Statistical analysis was performed using SPSS 2011 (Statistical Package for Social Science, IBM, USA). After testing for normality, using Shapiro Wilk and Kolmogorov tests, data were found to be normally distributed and were expressed as mean and standard deviation (SD). One-Way ANOVA was used to compare between different assessments, followed by Tukey's Post-Hoc test for multiple comparisons.

Independent t-test was also used to compare between both groups. The level of significance was set to ($P \leq 0.05$).

III. RESULTS

After cementation, the color change (ΔE) was significantly lower in Group ZC, however, after

thermocycling, it was significantly lower in Group CH (**Table 1**). Additionally, statistically significant color changes were seen in all parameters of both cements after thermocycling.

Table 1: Color change as affected by cement type

	Parameters	Group (CH)		Group (ZC)		Difference				
		M	SD	M	SD	MD	SEM	95% CI		P-value
								L	U	
After cementation (baseline-cementation)	ΔL	-3.24	0.17	-1.68	0.13	1.55	0.06	1.41	1.69	<0.0001*
	Δa	0.13	0.04	0.15	0.02	0.02	0.01	-0.005	0.050	0.054
	Δb	1.98	0.12	1.06	0.10	0.92	0.04	-1.02	-0.81	<0.0001*
	ΔE	3.80	0.17	2.00	0.12	1.8	0.065	-1.93	-1.66	<0.0001*
After Thermocycling (cementation-thermocycling)	ΔL	-1.18	0.11	-1.65	0.12	0.47	0.05	-0.58	-0.35	<0.0001*
	Δa	0.66	0.10	1.03	0.08	0.37	0.039	0.28	0.45	<0.0001*
	Δb	2.07	0.16	3.06	0.19	1.19	0.22	0.73	1.65	<0.0001*
	ΔE	2.47	0.16	3.63	0.17	1.15	0.07	0.99	1.30	<0.0001*

M: Mean, SD: Standard deviation, MD: Mean difference, SEM: Standard error mean

*: Significant difference as $P \leq 0.05$, CI: Confidence interval, L: lower arm, U: Upper arm

IV. DISCUSSION

The null hypothesis was rejected, as there was a statistically significant effect of the cement type on color change before and after thermocycling.

Zirconia discs thickness was set to simulate laminate veneers. Both tested cements were translucent for standardization and to minimize their influence on the outcome.⁷

CIEL*a*b* system was used due to its accuracy and high reproducibility.¹⁸ It is commonly used, allowing easy comparison of the obtained data to other studies.²⁸ In our study, perceptible color change threshold ($\Delta E = 3.7$) was used.

After cementation: The color change in Group CH was significantly higher, indicating that dual-cured resin-cement lacking amine initiator was more color stable upon cementation.^{13,29} Our results came in agreement with Abdalhalim et al.,³⁰ who found that light-cured resin-cement (RelyX veneer) showed higher color change than amine-free MDP-containing dual-cured cement (RelyX ultimate) after cementation. However, our results did not coincide with Ural et al.,¹³ who found that amine-free MDP-containing dual-cured resin-cement (RelyX

ultimate) showed higher color change than light-cured cement (NX3) after cementation.

The color change in Group ZC was within the perceptibility level, thus was considered clinically acceptable. The color change in Group CH ($\Delta E = 3.8$) was higher but very close to the perceptibility level. These results emphasized the effect of the cement type on the color of thin esthetic restorations.

The color change seen in Group CH might be attributed to the presence of unreacted camphorquinone molecules, which tend to turn yellow after 24 hours of light polymerization.³¹ This justified our results, which showed increase in (b*) positive value (yellowness) in CH Group. It might also be due to the type of restorative material, where translucent zirconia can limit the amount of curing light transmission compared to lithium disilicate, subsequently significantly affecting the degree of resin-cement conversion leaving unreacted camphorquinone molecules.^{25,32} Furthermore, the shade of the ceramic material used (A2) might have affected the curing light transmission as reported by Passos et al.,³³ who stated that zirconia with lighter shades have better light transmission than darker one.

After thermocycling: The significantly higher color change in Group ZC might be attributed to the difference in the tested cements chemical composition, where MDP-containing dual-cured resin-cements were found to undergo oxidation of inherent unreacted carbon-carbon double bonds upon aging, which might cause cement discoloration.¹³ Our results were consistent with Saati S et al.,⁷ who reported that light-cured resin-cement (Choice2) had significantly less color change than amine-free dual-cured resin-cement (Panavia V5). Our results did not coincide with Marchionattiet al.,³⁴ who reported that light-cured resin-cement (Variolink esthetic) showed insignificantly higher color change than dual-cured cement (variolink esthetic). The difference might be due to different chemical composition of their cements.

The color changes seen in both groups were below the perceptibility level, indicating that both cements showed adequate color stability after aging. According to our findings, (L^*) values decreased, whereas the a^* and b^* values increased after aging for both groups. Such findings might be attributed to the presence of TEGDMA (in ZR-Cem) and Bis-GMA (in Choice2), which are susceptible to high degree of water sorption upon thermocycling and subsequently hydrolytic degradation causing hydrolysis of polymer bonds, leading to color change.^{25,30,35} Bis-GMA in the cement and the adhesive used also has an inherent tendency to turn yellow, especially when exposed to heat.³⁴ Additionally, in Choice2, these changes might be attributed to presence of camphorquinone, which although useful in initiating the polymerization reaction, it is a yellowish compound that degrades and causes cement discoloration over time.³⁵

Furthermore, these color changes might be due to the effect of aging on the ceramic material itself. Translucent zirconia tends to turn darker, yellowish, and reddish after thermocycling,³⁶ due to the transformation of tetragonal phase into monoclinic phase after aging.³⁷ Also, the coexistence of tetragonal and monoclinic phases after aging increases refractive indices difference, hence, decreased the translucency and induced color instability.³⁸ Additionally, thermocycling can affect

the coloring pigments added to Y-TZP, causing pigment breakdown resulting in color instability and affect translucency, which is related to the changes in lightness and yellow–blue coordinates.³⁹ Another reason might be the effect of aged PMMA substrate showing water sorption, which might have played a role in color change in both groups.⁴⁰ Although, the effect of ceramic and substrate are evident, their effect was neglected because they were similar in both groups.

Regarding the effect of thermocycling: In Group CH, color change after cementation was significantly higher than after thermocycling, indicating high color stability; however, it drew the attention to the immediate color mismatch that might occur clinically if the cement shade was not meticulously selected. In Group ZC, color change after cementation was significantly lower than after thermocycling, indicating satisfactory immediate color match and stability.

Limitations of our study were using one thickness of ceramic material, using a single shade of monolithic zirconia and single shade of resin-cements and thermocycling using water rather than saliva or different beverages.

V. CONCLUSION

Based on our findings, it is concluded that resin-cement type and aging affect the final color of thin high-translucent zirconia restorations. Thermocycling produced higher color change in amine-free MDP-containing dual-cured resin-cement compared to light-cured cement, but the change was within the clinically accepted threshold. Light cure resin-cement has more resistance to artificial aging compared to amine-free MDP-containing dual-cured resin-cement.

The study's clinical implication showed that when constructing laminate veneers restorations made of high-translucent zirconia, amine-free MDP-containing dual-cured resin-cement is considered a promising alternative to conventional light-cured resin-cement in terms of color change.

- **Conflict of interests:**

The authors have no conflicts of interest relevant to this article.

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- **Ethics:**

This study protocol was approved by the ethical committee of the faculty of dentistry- Cairo university on: 27/10/2020, approval number [111020].

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