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Comparative evaluation of the effect of different resin luting agents on the color of lithium disilicate crowns after aging (An In Vitro Study)

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ABSTRACT

BACKGROUND & OBJECTIVE: Ceramics made of lithium disilicate are popular because of their superior optical properties and biocompatibility. The aim of this study was to evaluate the cement types that yield the best results and to assess the effects of various resin cements on the shade of lithium disilicate restorations, both before and after thermocycling.

METHODOLOGY: Lithium disilicate blocks were used to restore thirty prepared central incisors from typodont teeth. The restorations were cemented with 3 types of resin cement (G-Cem One, Breeze, and Itena). A spectrophotometer was used to measure the color parameters of the restorations in three stages: 1) prior to cementing, 2) following cementing, and 3) following 5000 cycles of thermocycling. The CIEDE2000 formula was used to calculate color differences (ΔE_{00}).

RESULTS: There was no noticeable difference found between the three research groups in the means of ΔE_{00} 1 and ΔE_{00} 2. However, the highest mean was observed in the GC Resin Cement. ΔE_{00} 3 assesses the color change after thermocycling compared with readings after cementing. The means of ΔE_{00} 3 did not differ significantly across the three groups, but Itena Resin Cement has the highest mean.

CONCLUSION: There was some color change in resin cements of the same color made by different manufacturers, although these changes were usually within clinically normal limits.

KEYWORDS: Resin Cements, Luting Agents.

INTRODUCTION

Aesthetics is now a concern for many dentists and for most patients. Due to increased demand, ceramic is one of the best materials dentists use. The main problem is color matching between restored teeth and adjacent teeth, and many factors affect color matching and clinical success, including cements, material types, material thickness, and adhesive systems^[1].

In recent years, ceramics have become the most widely used restorative materials because of their superior aesthetic properties and excellent biocompatibility. One of the ceramic restorations is Lithium disilicate, which is considered the preferred option because of its high translucency, similar to that of enamel, and mechanical properties, including a flexural strength of about 350 MPa^[2]. There are two types: machinable blocks that use CAD/CAM milling technology (E-max CAD) and press ingots that use the lost-wax technique (E-max Press). E-max blocks are a preferred restorative material because their hardness is the same as the hardness of enamel^[3].

In recent years, digital dentistry has enabled the manufacture of CAD/CAM ceramic restorations with excellent specifications, including high translucency and aesthetics. The other advantages include reduced chairside time, improved shade selection, enhanced communication between the lab technician and the dentist, and, most importantly, reduced fabrication flaws such as voids, fractures, and fire issues^[4].

Also, the underlying dental substrate's translucency and ability to hide color are significantly impacted by increases in ceramic thickness. The potential for aesthetic restoration to minimize defects is enhanced when the translucency parameter decreases with increasing ceramic thickness^[5]. However, the cement color became more noticeable when the ceramic restoration's thickness decreased to less than 1.5 mm^[6].

Glass ionomer, resin-modified glass ionomer, zinc phosphate, polycarboxylate, and resin cement are several types of dental cements^[7]. Glass ceramic resin cements can be self-etching or self-adhesive, and they vary in

How to cite this: Khidher LB, Rasheed RS. Comparative evaluation of the effect of different resin luting agents on the color of lithium disilicate crowns after aging (An In Vitro Study). *Journal of University Medical & Dental College*. 2025;16(4):1156-1162.



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viscosity, number of application steps, and requirements for light polymerization. So crowns, bridges, inlays, and onlays can now be cemented more quickly and easily with self-adhesive resin cement, which eliminates the need for separate etching, priming, bonding, and mixing. However, the resin content of the cement may influence the degree of microleakage, potentially affecting the long-term marginal integrity and color stability of restorations [8,9].

One of the most widely used aging techniques recently for examining restorative materials is thermocycling, with temperatures between 5 and 55 °C considered the upper limit for oral-cavity conditions, and 6 months in the oral cavity equivalent to 5000 temperature cycles applied to the test samples [10].

Various approaches exist for assessing a restorative material's masking ability. They can be determined using either visual or instrumental methods. The spectrophotometer was among the most accurate, versatile, and effective color-matching tools. On the other hand, the visual method remains the most commonly used procedure for shade identification because shade guidelines are available, inexpensive, and relatively simple to apply [11]. A critical difficulty in dental color treatment is determining a reference ΔE value to interpret spectrophotometer results, even though this device can accurately detect color differences. The range of 1 to 3.3 is considered clinically acceptable. In contrast, ΔE levels beyond 3.3 are considered inappropriate [12].

In 2011, Chaiyabutr and colleagues in Washington found that the cement color became more noticeable as the thickness of the ceramic restoration decreased to less than 1.5 mm [6]. In 2021, Tabatabaian F and colleagues in Iran found that using digital devices for color measurements increased accuracy by 94% [11]. In 2023, Yigit Yamali and colleagues in Turkey found that a resin cement thickness of 40 μm showed less color difference than 80 μm or 120 μm [13].

This study aims to compare the final shade of high-translucency lithium disilicate crowns before and after thermocycling aging using three of the most commonly used resin luting agents in Iraq: GC Self-Adhesive resin cement, Breeze Self-Adhesive resin cement, and Itena Dual Cure resin cement.

They represent different chemistries and have documented clinical performance. GC resin cement products include resin-modified glass ionomer and universal self-adhesive resin formulations that combine chemical bonding potential to tooth structure, fluoride release, established biocompatibility and bond strength in laboratory and regulatory testing.

Itena's TotalCem/Total C-Ram are self-adhesive, self-etching resin cements (including 4-META or similar chemistries) with a dual-cure mechanism; they offer competitive bond strengths, low film thickness, and physical properties appropriate for ceramic and metal restorations.

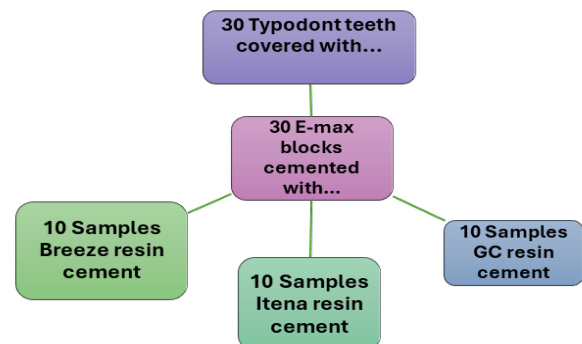
So, all three brands are readily available and have prior laboratory/clinical data that make them suitable for studies of luting performance (adhesion, microleakage, water sorption/solubility, and clinical handling).

METHODOLOGY

The study was conducted in vitro using typodont teeth. As such, ethical approval was not required. However, all procedures were performed in accordance with national and international guidelines for research ethics and laboratory practice.

Data collection was conducted from November 2024 to March 2025 in 2 cities in Iraq (Baghdad & Erbil). A total of 30 typodont teeth [14] of the maxillary left central incisor were prepared digitally using exocad software, with one digital cast, and then classified into three groups based on the type of resin cement used. Then, they are covered with crowns milled from lithium disilicate glass ceramic blocks (Cameo, A1, High Translucency, C14; Aidite) (n=10) using a chairside CAD-CAM system [3] (Table -I-). The restorations were bonded with different types of resin cement to 3D-printed dies of the tooth preparation and divided into 3 groups (n=10 per group): E-max blocks were cemented with translucent GC self-adhesive (G-Cem One Universal Tr, Japan), translucent Breeze self-adhesive (Pentron Clinical West Collins Orange, USA), and translucent Itena dual-cure (Totalcem dual cure, Itena, France) (Figure -I-).

Figure -I- Study Design.



Dental Lithium Disilicate Glass-Ceramic blocks (Cameo, A1, High Translucency, C14; Aidite) shade (A1) were designed to the dimensions of the original typodont tooth [3]. Then, all restorations were milled using the same design parameters.

A maxillary left central incisor typodont tooth shade A2 was prepared digitally using exo-cad software, with a 1mm reduction from all surfaces, according to preparation guidelines for E-max crowns downloaded from the manufacturer's web pages (Figure II-).

Figure -II- Digital cast.



The CAD-CAM software was used to design crowns with a uniform thickness of 1 mm because the cement color may become influential when the thickness of the ceramic restoration decreases to less than 1.5mm [6] (Table I-).

Table-I: Devices used in the study.

Devices	Manufacturer
Scanner	Scanner Medit T710, America
Printer	ASIGA, Australia
Washing Machine	Anycubic, China
CAD/CAM milling machine	AMW-400- SKYLINE MED, China
Sintering device	Vita Vacumat, Germany
Thermocycler	Custom - made
LED light	LY-C240, Dentaone second cure, China
Radiometer	LM-1, DTE-Woodpecker, China
Spectrophotometer	Rayplicker, France

After that, all crowns were placed on the firing tray and inserted into a warmed sintering device designed for lithium disilicate ceramics. Table-I- The color of the crowns was changed from purple to white. They were then glazed with a glaze paste and fired again, following the manufacturer's instructions.

Color differences occur when restorations are cemented with (40µm, 80µm, 120µm) resin cements, with the exception of the group bonded with 40µm cement thickness [13]. Therefore, resin cement thickness was set as 40µm using CAD-CAM software, and three types of resin cement were used in translucent shades.

The samples were maintained at room temperature in distilled water throughout the study to prevent dehydration. Additionally, all tooth samples were wrapped with polytetrafluoroethylene (PTFE) tape to prevent excess resin cement from entering the resin cast.

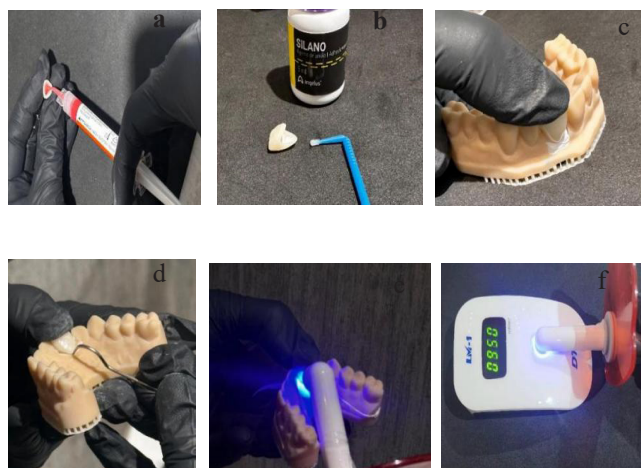
The crowns were divided into 10 specimens per group: Group 1: Breeze Self-Adhesive Resin Cement (Pentron Clinical, West Orange, USA). Group 2: Itena Dual Cure Resin Cement (Totalcem dual cure, Itena, France). Group 3: GC Self-Adhesive Resin Cement (G-Cem one Universal Tr, GC Japan).

The inner surface of the crowns for all groups has been treated similarly before cementation with 10% hydrofluoric acid (Condac porcelana, FGM, Brazil) for 20 seconds, rinsed with air-water spray for 1 minute, and dried. After that, the silane coupling agent (Silano, Angelus, Brazil) was applied for one minute and was left to dry.

Each cement was mixed according to the manufacturer's recommendations using its mixing tips to standardize cementation. The cement was then applied to the crowns' intaglio surface and placed over the typodont tooth with maximum finger pressure [15]. Then, the excess cement was removed from the margin by a dental probe.

An LED light was applied on each surface for 20 seconds at (800-950) mW/cm² according to the cement manufacturer's instructions. After finishing every ten specimens, the curing light output was checked with a radiometer to ensure uniformity. (Figure -III-). Table-I.

Figure -III: A- Etching with hydrofluoric acid, B- Coating with silane coupling, C- Applying cement with finger pressure, D- Removal excess cement, E- Light curing, F -Testing light output.



A thermocycling apparatus was then used to expose the specimens to 5000 temperature cycles, corresponding to 6 months in the oral cavity, with water between 5°C and 55°C for 30 seconds in each bath [10] Table-I.

Three procedures were used to measure the color parameters: before cementing, during cementing, and after thermocycling [13]. Prior to the measurements, the specimens were stored in distilled water for ten minutes. A dental spectrophotometer was used to assess the specimens' colors using the CIELab color scale at a standard illuminant of D65 Table-I.

The Rayplicker Handy device performs an auto-calibration tip that integrates a grey reference card inside. The same investigator conducted each measurement under the same circumstances. After thermal aging, the CIEDE2000 formula was used to calculate color differences. The color variations of the lithium disilicate ceramics were evaluated using the acceptability (1.8) and perceptibility (0.80) standards (ISO/ TR 28642:2016) [16].

After the typodont tooth was placed in the die cast, the calibration tip was stabilized with the base of the cast to allow precise positioning of the tip, and it should be in contact with and perpendicular to the surface of all samples.

The Lab color for each specimen was measured at the cervical, middle, and incisal thirds. It was marked with the same-sized circle using Vision Borea software, and an average was calculated for the color measurements prior to, following cementation, and after thermocycling of all samples. Then, the measured color differences were determined, and the differences between 1 and 3.3 were clinically acceptable, even if expert dentists could detect them. In contrast, ΔE levels beyond 3.3 were regarded as inappropriate because they were visible even to unskilled observers [12].

The Statistical Package for Social Sciences (SPSS, version 26) was used to analyze the data. The Shapiro-Wilk test was performed to assess data normality; as a result, nonparametric tests were used as necessary. One-way Analysis of Variance (ANOVA) was used to compare the means of three groups, and a post hoc test (LSD) was used to compare each pair of

means. Related-Samples Friedman's Two-Way Analysis of Variance by Ranks was used to compare the medians and mean ranks of the LAB color space at different times of the study, and a post-hoc test (Bonferroni) was used to compare the mean ranks of each pair of groups. A p-value of ≤ 0.05 was considered statistically significant.

RESULTS

Table- II: Means of color difference by type of material.

Variables	Material	n	Mean±SD	P-value*	Groups	P-value**
ΔE_{001}	A)Breeze Resin Cement	10	1.296±0.698	0.107	A X B	0.731
	B)Itena Resin Cement	10	1.403±0.649		A X C	0.049
	C)GC Resin Cement	10	1.929±0.713		B X C	0.098
	Total	30	1.543±0.720			
ΔE_{002}	A)Breeze Resin Cement	10	1.290±0.621	0.092	A X B	0.964
	B)Itena Resin Cement	10	1.276±0.697		A X C	0.061
	C)GC Resin Cement	10	1.882±0.711		B X C	0.056
	Total	30	1.482±0.714			
ΔE_{003}	A)Breeze Resin Cement	10	0.781±0.280	0.267	A X B	0.129
	B)Itena Resin Cement	10	1.101±0.641		A X C	0.776
	C)GC Resin Cement	10	0.840±0.370		B X C	0.212
	Total	30	0.908±0.463			

*Calculated by ANOVA. **Calculated by LSD post-hoc test.

SD: standard deviation.

The color change (after cementing, compared with readings before cementing) was assessed by ΔE_{001} . The means of ΔE_{001} didn't vary significantly between the three study groups ($p = 0.107$). However, Table-I showed that the highest mean (1.929) was for GC Resin Cement.

ΔE_{002} measures the color difference between readings after thermocycling compared with readings before cementing. The means of ΔE_{002} did not differ significantly among the three study groups ($p = 0.092$). It was worth noting that the highest mean was observed in GC Resin Cement, although the difference was not significant.

ΔE_{003} assesses the color change after thermocycling compared with readings after cementing. There was no significant variation in the means of ΔE_{003} across the three groups ($p = 0.267$). The highest mean (1.101) was observed in Itena Resin Cement (Table- II).

Considering the Breeze Resin Cement, no significant ($P=0.122$) difference was found between the 'L0' value (median=74.62), 'L1' value (median=74.00), and 'L2' value (median=74.30). There was a significant ($p= 0.001$) increase in 'B' values, starting from a median of 7.72 before cementation (B0), a median of 8.68 after cementation (B1), and then a median of 9 after thermocycling (B2). The difference in 'A' values at different study times was insignificant ($p = 0.509$).

Regarding the Itena Resin Cement, the difference in 'L' values was significant ($p = 0.020$), mainly due to the difference in L0-L1 ($p = 0.007$) and L0-L2 ($p = 0.044$). The difference in 'B' values was also significant ($p = 0.002$),

which was attributed mainly to the difference between B0-B2 ($p < 0.001$), where the medians were 7.77 and 8.57, respectively. The median of 'A0' was 0.32, and that of 'A2' was 0.17 ($p = 0.019$).

Considering the GC Resin Cement, a noticeable difference was found between the 'L' values ($p = 0.020$), where there was a decrease of the medians from 74.33 in 'L0' to 73.03 in 'L1' and then 72.95 in 'L2'. The "B" medians at different times during the investigation did not differ significantly ($p = 0.054$), but the lowest median was in 'B0' (7.70). A significant ($p = 0.021$) difference was found between the medians of the 'A' values, where the highest median was of 'A0' (0.32). More details were presented in Table-III.

DISCUSSION

This in vitro study evaluates different types of resin cement for luting lithium disilicate ceramic restorations and compares their effects on the visible features and final color of the ceramic after aging.

Previous clinical reports published in the Journal of University Medical & Dental College (JUMDC) have highlighted the successful clinical application of all-ceramic prostheses in restorative dentistry^[17]. Moreover, studies have shown that when the thickness of glass-ceramic restorations is 2 mm or more, the underlying substrate and cement shade have minimal influence on the final color; however, reducing the ceramic thickness to 1 mm can significantly alter the overall shade of the restoration^[6,18].

Color change in resin-luted crowns

Table- III: The 'LAB'† color space at different times of the study, in each of the studied materials.

Group	Median	Mean rank	P-value*	Groups	P-value**
Breeze Resin Cement					
L0	74.62	2.50		L0-L1	N/A
L1	74.00	1.60	0.122	L0-L2	N/A
L2	74.30	1.90		L1-L2	N/A
B0	7.72	1.1		B0-B1	0.025
B1	8.68	2.1	0.001	B0-B2	< 0.001
B2	9.00	2.8		B1-B2	0.118
A0	0.28	2.00		A0-A1	N/A
A1	0.18	1.75	0.509	A0-A2	N/A
A2	0.27	2.25		A1-A2	N/A
Itena Resin Cement					
L0	74.67	2.70		L0-L1	0.007
L1	73.73	1.50	0.020	L0-L2	0.044
L2	73.63	1.80		L1-L2	0.502
B0	7.77	1.20		B0-B1	0.074
B1	8.22	2.00	0.002	B0-B2	< 0.001
B2	8.57	2.80		B1-B2	0.074
A0	0.32	2.60		A0-A1	0.094
A1	0.17	1.85	0.050	A0-A2	0.019
A2	0.17	1.55		A1-A2	0.502
GC Resin Cement					
L0	74.33	2.70		L0-L1	0.044
L1	73.03	1.80	0.020	L0-L2	0.007
L2	72.95	1.50		L1-L2	0.502
B0	7.70	1.45		B0-B1	N/A
B1	8.83	2.05	0.054	B0-B2	N/A
B2	8.85	2.50		B1-B2	N/A
A0	0.32	2.70		A0-A1	0.034
A1	0.00	1.75	0.021	A0-A2	0.010
A2	0.13	1.55		A1-A2	0.655

*Calculated by Related-Samples Friedman's Two-Way Analysis of Variance by Ranks.

**Calculated by Bonferroni post-hoc test.

†A refers for the position on the red-green axis, B for the position on the yellow-blue axis, and L for lightness, which ranges from 0 (black) to 100 (white).

In this study, three types of resin cement thickness were set as 40µm in translucent shade and lithium disilicate Glass-Ceramic blocks (Cameo, A1, High Translucency, C14; Aidite) were designed to a uniform thickness of 1 mm. As mentioned previously, lithium disilicate ceramic (Cameo E-max) blocks have a hardness almost identical to that of enamel, making them a desirable restorative material. They were above the 300 MPa threshold and also satisfied the ISO 6872:2015 standard for the fabrication of veneers, inlays, onlays, and single anterior or posterior crowns [3].

The study's findings suggest that the means across the three stages did not differ significantly across the three groups. According to ISO/TR 28642:2016, the clinically perceptible threshold for color difference (ΔE_{00}) is 0.8, while the

clinically acceptable threshold is 1.8. These values have been widely used in dental color research and provide a standardized reference for evaluating color changes in restorative materials [16]. In the present study, the mean ΔE_{00} values after aging ranged from 1.00 to 3.30. The GC group showed a mean ΔE_{00} of 1.929, which is above the 1.8 acceptability threshold, indicating that the color change was clinically acceptable but perceptible. The higher ΔE_{00} values observed for Breeze and Itena suggest more pronounced and potentially unacceptable color alterations under the same conditions. So, practitioners should still consider material properties, such as polymerization mode, filler content, and shade-matching features, when selecting a luting agent, particularly for thin or highly translucent restorations.

These findings were consistent with those of a previous study^[19], which reported that identical shades of resin cements from three different types had the same effect on the color of lithium disilicate ceramic at a 1.4 mm thickness. However, other studies found that resin cements of the same shade but different types exhibited different color parameters^[20,21].

This study aimed to reconcile the inconsistent results across studies, which may be due to variations in study methods, including specimen preparation, color measurement methods, and different cement thicknesses and shades. Digital color measurement using the Rayplicker Handy device improved accuracy by up to 94%^[11,19], employing auto-calibration and the CIEDE2000 formula. Thermocycling (5,000 cycles between 5°C and 55°C) was performed to simulate oral thermal stresses and assess the color stability of the restorations. The results show no significant differences in color change among the three types of resin cements tested (as measured by ΔE_{00_2} and ΔE_{00_3} ; $p = 0.092$ and 0.267 , respectively). Despite the mechanical stress opposed by thermocycling (consistent with other previous studies)^[22, 23].

The higher mean color change observed in GC Resin Cement, especially in ΔE_{00_2} , might suggest greater sensitivity to the thermal stresses imposed by aging. This could be due to factors such as the resin cement's chemical composition, filler content, or the polymerization shrinkage occurring during setting^[24]. The susceptibility of GC Resin Cement to thermal aging may also be linked to its water-absorption characteristics, as certain resin cements are known to absorb moisture over time, which can contribute to discoloration^[25]. Conversely, the Itena Resin Cement shows the greatest average color change in ΔE_{00_3} , indicating that although it performs similarly to others after cementation, its performance deteriorates more under thermocycling conditions. This could be attributed to the resin-filler interface's potential weakening during thermal cycling, which allows changes in visual features^[26].

Even after aging, the absence of significant differences in color change following thermocycling across groups suggests that all three tested resin cements may exhibit relatively stable color performance.

CONCLUSION

A certain amount of color change occurs over time in resin cement of the same color produced by different manufacturers, and the variations were generally within clinically acceptable limits. Breeze Resin Cement demonstrates stable lightness but increased yellowness. Itena Resin Cement shows slight darkening and increased yellowness along with a minor shift towards green, and GC Resin Cement exhibits gradual darkening and subtle changes in the red-green axis.

These findings suggest that the choice of resin cement can influence the long-term esthetic outcomes of lithium disilicate crowns, and dentists should consider these factors (resin matrix composition, filler loading, etc.) when selecting luting agents for restorations where color stability is considered a paramount element of resin cements. Although

thermocycling simulated oral temperature changes, this study remains an in vitro investigation using typodont teeth, which may not fully replicate the optical and bonding properties of natural enamel and dentin. The small sample size ($n = 10$ per group) limits statistical power, and only thermocycling was applied, without accounting for long-term factors such as staining, abrasion, or pH fluctuations. These factors may affect the color stability of lithium disilicate restorations in clinical conditions. Consequently, the findings provide valuable baseline information; larger sample sizes are recommended for future in vivo research.

ACKNOWLEDGEMENT: None.

CONFLICT OF INTEREST: None.

GRANT SUPPORT AND FINANCIAL DISCLOSURE: None.

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Authors Contributions:

Lana Bahram Khadhr: Substantial contributions to the conception and design of the work.

Randa Saad Rasheed: Analysis, Interpretation, Drafting the work and reviewing it critically for important intellectual content.

Submitted for publication: 30-06-2025

Accepted after revision: 30-10-2025