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# Bioactive Cement versus Resin Cement Regarding the Perceived Shade of E-Max Restorations (An In Vitro Study)

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## ABSTRACT

***Background:*** Perceived shade change of ceramic restorations can be disappointing to both operator and patient; therefore evaluation of how the underlying cement can impact the final perceived shade is required. ***Aim****:* Compare effect of bioactive cement versus resin cement on the perceived shade of final ceramic restoration with and without presence of substrate**.** ***Methodology:*** Forty plate shaped samples of lithium disilicate ceramic were prepared (E-max CAD (HT A1/C14 – 1.3 mm thick). Composite substructure of shade A3.5 and 2mm thickness were prepared (n=20). E-max Samples were divided to four groups named (B, R, BC, and RC) (n=10). Groups (B, R) received cement only; (B) for bioactive cement (ACTIVA), (R) for resin cement (Rely X unicem) respectively, both cements were selected of the same shade (A2) as a universal shade. Groups (BC, and RC) were cemented to composite substructure. Shade of E-max Ceramic Specimens was evaluated using spectrophotometer to obtain L*, a*, b* values after etching only, after etching and cement application only, then after cementing to substrate respectively. The generated data was statistically analyzed. The significance level was set at $P \leq 0.05$. ***Results:*** there was no significant difference between Activa and Rely X without substrate, while with substrate; Activa showed statistically significantly higher mean ΔE than Rely X. ***Conclusion****:* The type of luting agent had a great influence on the perceived shade of the lithium disilicate ceramic.

***Keywords:*** bioactive cement – shade – resin cement – Activa

## INTRODUCTION

A natural appearing smile reflects youth and acceptance. To create a dental restoration that gives a natural look, it requires accurate replication of size, shape, surface texture, contouring, translucency and color of natural dentition.

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Dental ceramics are classified into three main classes glass matrix ceramic, polycrystalline ceramics and resin matrix ceramics. Lithium disilicate is one of the most famous glass- ceramics. It has adequate mechanical properties and offers attractive esthetic quality.[1]

The basic function of dental cements is to seal the space between tooth structure and the restorative material, and to improve the resistance of restoration to dislodgement during function. Cements are divided into two categories; luting cements and bonding cements. Bonding is a term that implies chemical or physical interaction occurs to both surfaces to be joined. Luting refers to micromechanical interlocking between materials.[2]

Resin cements are widely used for cementation of lithium disilicate as it bonds chemically to underlying glass ceramic increasing strength and longevity of dental restoration. Furthermore, it has a clinically accepted color effect and stability. Resin cements can be classified according to adhesive scheme to three classifications. First; total etch resin cements that have high bond strength and reduced microleakage. Second; Self-etch system, this type of resin cements shows weaker bond strength to enamel than that of the total- etch type.[3]

Self-adhesive cements, also called universal adhesive cements, were a new category of resin cement introduced in 2002. They do not require any pretreatment of the tooth surface as these cements contain phosphoric acid in their components that reacts with filler particles and dentine forming the bond. Once the cement is mixed, it is applied on tooth structure Authors assume that bonding of self-adhesive cements with dentin is better than with enamel so, selective etching of enamel is suggested to improve bond strength of those cements with enamel.[3,4]

Another category of newly introduced cements is bioactive dental cement, it is claimed to contain ionic resin matrix, mimic the physical and chemical properties of natural teeth. Additionally, it releases and recharges calcium, phosphate and fluoride. In 2013, Pulpdent Corporation acquired approval from the US FDA for two materials Activa Bioactive-Restorative and Activa Bioactive-Base Liner. They combine the advantages of glass-ionomer systems and resin-based composites.[5-7] According to the manufacturer, Activa-Restorative and Activa -B/L contain an ionic resin matrix that act as a shock-absorbing resin component. They also contain bioactive fillers that imitate the physical and chemical properties of natural teeth. They are durable, wear- and fracture-resistant, chemically bond to tooth, seal against bacterial microleakage, release and recharge with calcium, phosphate, and fluoride ions more than glass ionomer. Activa contains no bisphenol A (BPA), bisphenol Aglycidyl methacrylate (bis-GMA), or BPA derivatives; so it penetrates and fills micro-gaps, reduces sensitivity, guards against secondary caries, and seals margins against microleakage. In addition; Activa is a self-adhesive triple cure bioactive cement; indicated for indirect restorations as zirconia, glass ceramics, resin, PFM and stainless steel crown.[8, 9]

Color is an essential parameter that attracts attention of patients. Dental restorations, with acceptable shade, translucency and fluorescence, should match that of natural dentition to mimic the vitality. For the shade matching, there are two main ways; visual technique and instrumental technique. Visual measurement is done with use of classical or 3D master shade guides for shade matching depending on Munsell color system. Instrumental technique mainly depends on CIE Lab system; L* refers to lightness and darkness, a*, b* refers to; red-green axis and yellow-blue axis respectively. $\Delta E^*ab = (\Delta L^{*2} + \Delta a^{*2} + \Delta b^{*2})^{1/2}$ represents the difference in lightness and chromaticity.[10-12]

In 2001 CIE developed a more sophisticated formula that is the CIEDE2000, to calculate color differences.[13] Instrumental shade matching devices classified into three categories: spectrophotometer, colorimeters and imaging systems; spectrophotometer are considered the most accurate method of color measuring.[14] The null hypothesis of this study was there is no difference between perceived shades of tested ceramic samples cemented by bioactive cement or resin cement either with or without composite substrate.

## MATERIAL AND METHOD

Sample size was calculated according to the power analysis for a 2 x 3 fixed effects analysis of variance; the first factor (Substrate) includes 2 levels and the second factor (Cement type) includes 2 levels. Since no relevant literature was found, the clinically acceptable limit of color change ($\Delta E = 3.5$) was used for the calculations. Using alpha ($\alpha$) level of 0.05 (5%) and Beta ($\beta$) level of 0.20 (20%) i.e. power = 80%; the study will include 10 samples per cell for a total of 40 samples. Sample size calculation was performed using IBM® SPSS® Sample Power® Release 3.0.1. A total of forty (n=40) plate shaped samples of CAD Lithium Disilicate (E.max-CAD; Ivoclar Vivadent) shade (A1 HT) were fabricated in dimensions of 12×14 mm and 1.3mm thick. The samples were divided into two main groups (n=20) in accordance to type of cement used. Each main group was subdivided into two subgroups, with and without substrate (n=10). Sample grouping is listed in table (1).

**Table (1):** sample grouping

| | **Presence of substrate** | **Activa** | **Rely X** |
|---|---|---|---|
| **Lithium disilicate plate specimens (n=40)** | No substrate (n=20) | N= 10 | N=10 |
| | With substrate (n=20) | N= 10 | N=10 |

To fabricate and standardize the samples to the desired thickness; three customized split molds were machined from Teflon material. First mold was (14×12×1.4) mm used to fabricate the cement with thickness of o.1mm = 100µ.[16-18] (Figure 1).

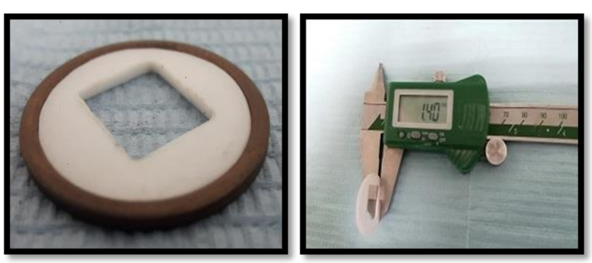

**Figure (1):** 1.4mm Teflon mold

The Second mold was (14×12×2 mm) to fabricate the composite substrate to a thickness of 2mm (Figure 2), and the third one was (14×12×3.4) to accomodate the sample ceramic/cement/substrate in one mold, so the ceramic sample (1.3mm thick) was placed then the cement was spread and finally the substrate (2mm thick) was inserted with pressure to simulate the clinical condition (Figure 3).

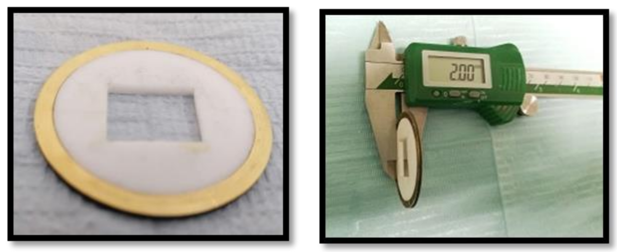

**Figure (2):** 2mm Teflon mold

**Figure (3):** 3.4mm Teflon mold

The Lithium Disilicate samples (HT A1) size C14 were sectioned with low speed saw (Isomet 4000, Linear Precision Saw). The saw was gauged to 1.3mm thick. The sectioning was done at low speed with heavy water coolant. Samples thickness was verified using digital caliber. Then the samples were adjusted with required thickness using ceramic finishing stone. The final size of the specimen was 14×12×1.3

mm Ceramic Specimens were crystallized in ceramic furnace according to manufacturer's recommendations (Figure 4).

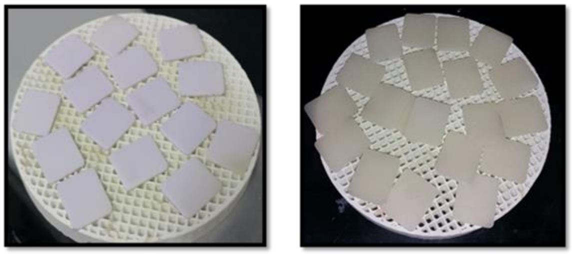

**Figure (4):** Samples before and after crystallization

The composite plate samples were packed and cured inside the mold of the dimension (12×14) and 2mm thick as the residual dentine thickness after crown preparation is suggested to be 2mm to protect the pulp.[19]

For application of cement layer only; the same procedure was carried out for both Activa (group B) and Rely-X (group R). Ceramic samples were treated with 9% hydrofluoric acid etchant for 20 seconds. The specimens were then rinsed thoroughly for 60 seconds and dried with oil free air. The surfaces were then silanized and left to react for 60 seconds. The samples were placed in the 1.4mm customized Teflon mold in order to control the cement thickness at 0.1mm. Cements (Activa and Rely X respectively) were directly applied on the ceramic sample. Polyester film strip was used to cover the sample, then the sample was positioned between two glass slabs under a load of (1kg) and then light cured with woodpecker I LED curing light according to manufacturer instructions; 20 seconds for both Activa and Rely X. (Figure 5).

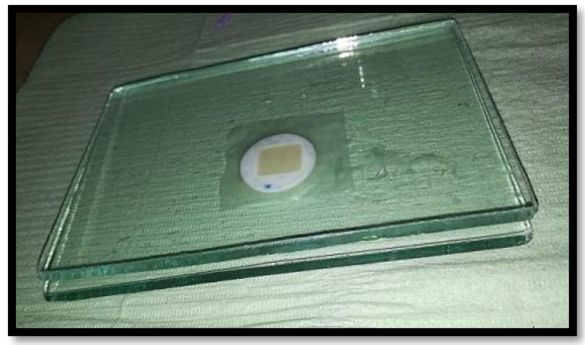

**Figure (5):** Specimen placed between two glass slabs with polyester film

Same cementation procedures were followed for both cement groups. The prepared composite substrate was placed in the 3.4 mm thick mold, ceramic samples were etched, coated with silane and each respective cement was added then placed into the mold. Then the Cements were cured.

Spectrophotometer (Agilent cary 5000) was used for color measurement. Three readings were obtained, after etching, after cement application, finally after cementation to composite substrate.

## RESULTS

The results showed that cement type (regardless of process steps) had a statistically significant effect on mean $\Delta E$; Activa $6\pm1.67$, Rely X $5.56\pm1.16$. Process steps (regardless of cement type) also had a statistically significant effect on mean ΔE; after etching 4.33±0.41, with cement 5.57±0.49, with cement and composite 7.53±0.63.

The interaction between the two variables had a statistically significant effect on mean ΔE. Since the interaction between the variables is statistically significant, so the variables are dependent upon each other (Figure 6, Table 2-4)

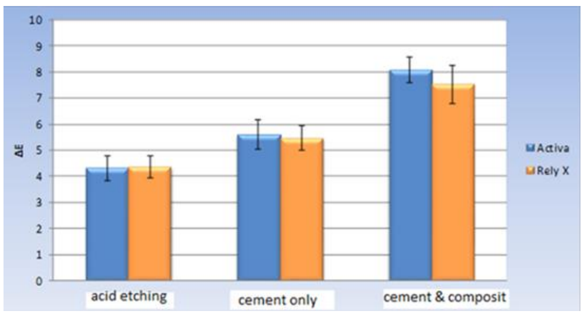


**Figure (6):** Bar chart representing mean and standard deviation values for ΔE with different interactions of variable

**Table (2):** Descriptive statistics of ΔE, ΔL, Δa, Δb values

| | Process Steps | Activa Mean ± SD | Rely X Mean± SD |
|---|---|---|---|
| ΔE | After etching | 4.31±0.48 | 4.36±0.41 |
| | With cement only | 5.6±0.57 | 5.47±0.49 |
| | With cement & composite | 8.08±0.5 | 7.53±0.5 |
| ΔL | After etching | 3.91±0.49 | 3.95±0.36 |
| | With cement only | 1.53±0.13 | 1.23±0.11 |
| | With cement & composite | 2.41±0.11 | 1.66±0.12 |
| Δa | After etching | 0.7±0.09 | 0.68±0.08 |
| | With cement only | 0.46±0.05 | 0.27±0.03 |
| | With cement & composite | 0.56±0.05 | 0.37±0.07 |
| Δb | After etching | -1.64±0.2 | -1.7±0.16 |
| | With cement only | 1.06±0.1 | 0.83±0.1 |
| | With cement & composite | 1.34±0.09 | 1.14±0.05 |

**Table (3):** The mean, SD values and results of two-way ANOVA test for comparison between ΔE, ΔL, Δa, Δb of the cement types regardless of process steps

| | Activa Mean ± SD | Rely X Mean±SD | P-Value | Effect size |
|---|---|---|---|---|
| **ΔE** | 6±1.67 | 5.56±1.16 | 0.001* | 0.197 |
| **ΔL** | 2.62±1.04 | 2.28±1.24 | <0.001* | 0.306 |
| **Δa** | 0.57±0.12 | 0.44±0.18 | <0.001* | 0.540 |
| **Δb** | 0.25±1.38 | 0.09±1.29 | <0.001* | 0.319 |

**Table (4):** The mean, SD values and results of two-way ANOVA test for comparison between ΔE, ΔL, Δa, Δb of the process steps regardless of cement type

| | After etching | | With cement only | | With cement & composite | | p-value | Effect size |
|---|---|---|---|---|---|---|---|---|
| | Mean | SD | Mean | SD | Mean | SD | | |
| **ΔE** | 4.33 | 0.41 | 5.47 | 0.49 | 7.53 | 0.73 | <0.001* | 0.900 |
| **ΔL** | 3.93 | 0.42 | 1.38 | 0.19 | 2.03 | 0.4 | <0.001* | 0.948 |
| **Δa** | 0.69 | 0.08 | 0.37 | 0.11 | 0.46 | 0.11 | <0.001* | 0.828 |
| **Δb** | -1.67 | 0.18 | 0.94 | 0.15 | 1.24 | 0.13 | <0.001* | 0.992 |

**DISCUSSION**

This study was designed to evaluate the effect of the Activa cement on the shade of lithium disilicate ceramic and compare it

with Rely X unicem in addition to compare the masking ability of the two cements to an underlying dark substrate. Shade perception after cementation of all ceramic restoration may suffer change affected by the shade of the underlying cement as well as the substrate. The null hypothesis in this study was rejected.

Composite resin substrate of a dark shade (A3.5) was selected for use in this study in order to mimic a dark discolored preparation.[19] The e-max Samples were cut to 1.3mm thickness according to manufacturer instruction and thickness was increased 0.1mm to mask the underlying dark substrate.[20,21] The split molds were fabricated to be opened on both faces to facilitate light curing by direct contact to the sample.[22] in this study cement thickness was controlled to 0.1mm, ***Magalhães et al 2013***[17] and ***Mesbah AM et al 2016***[18] used this thickness of resin cement in their studies.

Cary 5000 Spectrophotometer provided from Agilent Technologies (USA). Agilent Cary 5000UV-Vis-NIR spectrophotometer is manufactured according to a quality management system certified to ISO 9001. It is a double beam direct ratio recording system. It consists of the photometer unit and a PC computer.[23]

In our study the results showed that perceived color of the lithium disilicate ceramic was affected by the type of cement used. ΔE for Activa after etching, with cement only, with cement and composite substrate were (4.31± 0.48, 5.6±0.57, 8.08± 0.5) respectively. ΔE for Rely X after etching, with cement only, with cement and composite substrate were (4.36± 0.41, 5.47±0.49, 7.53±0.73) respectively. From these results activa showed the highest mean values; so, the proposed null hypothesis is rejected. At the same time, the mean color differences of the two cements in the process steps were above 3.7 ΔE units which mean that the color difference exceeded the perceptibility threshold.[24] Acid etching causes decrease in lightness (ΔL) which made sense, since acid etching of glass ceramic it removes the glassy matrix, exposes the lithium disilicate crystals and produce superficial irregularities of the etched ceramic surfaces.[25] After applying cements only; results revealed that Activa showed statistically higher mean of ΔL, Δa, Δb than Rely X. Although ***Mesbah AM et al 2016***[17] results showed highest ΔE value of

Rely X Unicem; in this study Activa scored higher ΔE than Rely X unicem in the previous study. This higher ΔE of Activa may be due to its chemical composition as it contains glass ionomer component. According to ***Ural Ç etal 2016*** [26], the discoloration of dual cured resin cement may be due to their chemical composition and degradation of residual amines or oxidation of unreacted carbon- carbon double bonds. These findings are in accordance with the results of ***Morsy ZM etal 2020*** [27] ***and Pissaia JF etal 2015*** [28], regarding the color change of dual cure resin cements. Although Activa showed statistically higher mean of ΔL, Δa, Δb than Rely X, there was no statistically significant difference between mean ΔE of the two cement types in the process of applying cements only.

In the groups (BC, RC); Activa (group BC) showed statistically higher mean of ΔL, Δa, Δb than Rely X (group RC). Results showed that there was significant difference between ΔE of the two cement types. Activa cement showed statistically higher mean ΔE than Rely X, which means that Rely X has better masking ability to the underlying substrate than Activa. The result of lower mean ΔE of rely X unicem cement is in agreement with a study done by ***Dede DO et al 2017.***[29] Acid etching causes the highest ΔL and Δa while cementation to the composite substrate has the highest Δb. Increase in mean (b*) indicates the specimens became more yellow.[30]

**CONCLUSION**

Within the parameters used and the limitation of this study, the followings could be concluded:

- The type of luting agent had a great influence on the shade of the lithium disilicate ceramic ( E-max )
- Activa changed the color of the lithium disilicate out of the clinical acceptance range and the results were statistically significant effect but were comparable with Rely X unicem as ΔE in group B = 5.6 while ΔE in group R = 5.46
- In the group with composite substrate the Activa had higher ΔE than Rely X unicem as ΔE in group BC = 8.8 while in group RC = 7.53
- Activa showed higher ΔL, Δa, Δb than Rely X. Acid etching process displayed the highest ΔL and Δa which means samples became darker and more reddish.
- Cementation to composite substrate had the highest Δb regardless of cement type indicates the samples became more yellow.

**CONFLICT OF INTEREST**

The authors of this work have no competing interest that may bias this work submitted to the JFCR

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