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Original Article

Effects of adhesive system, thermal aging and ceramic shade on the final color and bond strength of monolithic lithium disilicate ceramics

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Abstract

Aim: The present study aims to observe the effects of adhesive systems and thermocycling on the final color of monolithic lithium disilicate ceramics, also impacts of ceramic shade on the bond strength of resin cement.

Material and Methods: Monolithic lithium disilicate ceramic discs in VITA shades A1 and A3 and also self-adhesive, self-etch and total-etch adhesive resin cement systems that were used were investigated in this study. The same color of resin cement (A2 shade) was used for cementation. Ceramic samples of 0,5 mm in thickness were obtained from the IPS e.max CAD blocs. All specimens were distributed into two main groups and six subgroups (n=10) according to the ceramic shade and adhesive system. All specimens were exposed to thermocycling (10,000 cycles). A contact spectrophotometer measured the color differences according to the CIEL*a*b* system. The color differences between before resin cementation (1), after resin cementation procedure (2) and after thermocycling (3), were evaluated. Shear bond strength test was performed after thermocycling.

Results: All L values decreased after the resin cementation. The A3-VL (total etch-A3) group had statistically significantly lowest values of L* ($p<0.05$). A3-RX (self-adhesive-A3) group had the statistically significant highest a* and b* values ($p<0.05$). A1-VL (total etch-A1) group had the highest ΔE values, and there were no statistically significant differences between the A1-RX (self-adhesive-A1) and A3-VL (total etch-A3) groups ($p>0.05$). A3-RX group had the highest bond strength results, and there were no statistically significant differences between the A1-VL group ($p>0.05$).

Conclusions: All of the adhesive systems affected the final color of ceramics. Otherwise, the thermal aging procedure did not lead to the changes in the color of the ceramic-resin specimen. Furthermore, ceramic shade affected the bond strength of resin cement after thermocycling.

Keywords: CAD/CAM; esthetic; spectrophotometer; strength test

INTRODUCTION

Aesthetically successful treatments of the anterior teeth are possible recently because of the improvement in the ceramic materials. Cementation of ceramic systems has been provided from different adhesive techniques of resin cement. Advances in resin cement and ceramic systems make it possible to provide the same optical and mechanical properties as natural teeth. Porcelain laminate veneers, which were 0.5-1 mm in thickness let the higher translucency, have been frequently preferred as an aesthetic and conservative treatment (1). Thus, the shade and color durability of the resin cement under the restoration can be crucial in the longevity of the ceramic restorations (2).

Resin cement has several benefits, such as high bond strength (BS) to tooth and ceramic, low solubility in the oral circumstances, satisfactory esthetic results, increased mechanical features and support for ceramic superstructure (3). Internal and external discoloring due to microleakage is the disadvantage of resin cement (2-4).

Internal discoloration may occur from the changes in the resin's structure, such as the formation of oxygen by-products (2). In dual-cure and auto-cure resin cement, inhibitors and amine accelerators which have oxidized reactive groups may cause the color changes (5,6). Previous studies reported that higher yellow hue values arise from the decomposition of the inhibitors (5,7,8). Several studies in the literature have been reviewed to decide the color durability of the adhesive cement (5,6,9). On the other hand, the published studies have mostly

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focused on the adhesive cement itself rather than the influences of the adhesive cement on the ultimate color of ceramic (10-12).

The longevity of ceramic restoration mostly depends on the underlying cement (13). The durability of all ceramic systems arises from an increased BS between dental hard tissue-resin cement-ceramic composition (14).

Today, three different adhesive systems (self-adhesive, self-etch and total-etch) have been found for the cementation. Self-etch and total-etch adhesive techniques are utilized to create interaction among the tooth surface and resin cement. Thus, self-etch and total-etch adhesive techniques object to compose a hybrid layer on the surface of dentin. The self-adhesive technique does not include a phosphoric acid application. However, many studies have demonstrated decreased BS on dentin and enamel (15). The advantages of the self-adhesive system decreased the postoperative sensitivity and complex multi-steps. On the other hand, superficial interaction with the dentin surface and having limited etching capability cause lower BS. It is, thus, significant to evaluate how the shade of the ceramic may affect the BS of various resin cement, besides that observe the interaction among the luting material and ceramic. Also, clinical usage may affect the longevity of shade of ceramic and BS of resin cement. Thus, in many in vitro studies, the mechanical properties have been evaluated after thermal aging to simulate the oral condition (16,17).

This study aimed to examine the influences of different adhesive systems and ceramic shade on the final color and BS of ceramics after thermocycling.

The tested null hypotheses were that: (i) type of adhesive system and (ii) thermal aging would not influence the final color of monolithic lithium disilicate ceramics and (iii) ceramic shade would not influence the BS of resin luting material.

MATERIAL and METHODS

Two shades (VITA shades A1 and A3) of 60 monolithic lithium disilicate ceramic specimens (IPS e.max CAD MT blocs; Ivoclar Vivadent AG, Schaan, Liechtenstein) were employed for this study. Ceramic samples of 0,5 mm in thickness (ceramic slice dimensions:10x8x0,5 mm) were prepared from the E.MaxCAD blocs by cutting with a low speed saw (IsoMet 1000; Buehler Ltd., Lake Bluff, IL) then heated for crystallization according to the manufacturer's instructions. Under running water for 15 seconds, samples surfaces were polished using 400–600-800-1200 grit silicon carbide abrasive papers (3 M ESPE, St. Paul, MN, USA). Grinding machine (Minitech 233; Presi, Grenoble, France) was used for polishing at a 170 rev/min. The polished samples were ultrasonically cleaned for three minutes in deionized water and ethanol, and then air-dried. Ceramic thickness was measured and controlled with a digital caliper (Electronic Digital Caliper Co Ltd, Guilin, China). Subsequently, all samples were distributed into two main groups and six subgroups (n=10) according to the ceramic shade and adhesive system. Self-adhesive, self-etch, and total-etch resin cement in the same shade (A2) were selected as luting material. A contact type spectrophotometer (Vita Easyshade®, Vita-Zanhnfabrik®, Bad Säckingen, Germany) measured the color changes. The light probe dimension of the spectrophotometer was 6 mm in diameter. Thus, the diameter of the testing field size was 6 mm for the luting material.

Groups and cementation of ceramics

Each A1 and A3 shade ceramic group was divided into three subgroups according to the adhesive cementation system. Sample surfaces were coated using hydrofluoric acid (IPS Etching Gel; Ivoclar Vivadent) for 60 seconds before cementation, and then dried with air. Metal ring (0.5mm thickness and 6mm diameter) was prepared for use in the cementation area. Three adhesive systems were used in the same color (A2). Groups and cementation procedure were as follows:

Groups of A1-VL and A3-VL: Variolink N (VN group; Ivoclar Vivadent AG, Schaan, Liechtenstein) was utilized as the total-etch resin cement. The etched ceramic surface was covered with a silane material (Monobond-S; Ivoclar Vivadent) for 60 seconds and then dried with air. Variolink N base (white A1 shade) and catalyzer (yellow A3 shade) were mixed (1:1 ratio) and put inside the metal ring on the ceramic surface. The ceramic surface was seated using finger pressure, and light-curing was performed by a LED (light-emitting diode) curing unit (Valo Cordless; Ultradent Products, Inc., South Jordan, UT, USA) for 20 seconds with an intensity of 1200 mW/cm2.

Groups of A1-P and A3-P: Panavia F 2.0 (PF group; Kuraray Medical Inc., Tokyo, Japan) was used as the self-etch resin cement. A and B pastes of Panavia F 2.0 were mixed equal ratio (1:1) and put inside the metal ring on the ceramic surface under figure pressure. LED curing occurred through the surface of the sample. According to the manufacturer's instructions, an oxygen blocking agent (Oxiguard II; Kuraray Co. Ltd.) was kept on the resin surface for three min.

Groups of A1-RX and A3-RX: Rely X Unicem (RXU; 3M ESPE, Seefeld, Germany) was utilized as the self-adhesive resin cement. The tube was filled with the cement, which was activated using Aplicap™ activator (3M ESPE, Seefeld, Germany) according to the manufacturer's directions. The activated tube was then stirred in a high-frequency mixer (amalgamator) for 15 s. The mixed tubes were placed in the Aplicap (3M ESPE, Seefeld, Germany). The resin cement was put inside the metal ring on the sample surface, and then light-curing was occurred by LED.

All ceramic samples were kept in the dark and 37.8°C distilled water for one day. Following the polymerization was completed, all samples were exposed to the thermocycling procedure (SD Mechatronik Thermocycler, Germany) for 10,000 cycles among 5–55°C with a transfer time of 10 seconds and existing time of 20 seconds in each bath.

Measurement of color changes

A contact type of spectrophotometer (Vita Easyshade®, Vita-Zanhnfabrik®, Bad Säckingen, Germany) was utilized for measuring the color values of ceramic groups onto one side of the ceramics, according to the CIEL*a*b* system on a white floor and under the standard illuminant D65 according to International Organization for Standardization (ISO7491) (18). The calibration of spectrophotometer was made before the in-vitro measurements according to the manufacturer's instructions. The baseline color values measured were L* [brightness; from 0 (black) to 100 (white)], a* [shade measured throughout the red-green axis (redness)], and b* [shade measured along the yellow-blue axis (yellowness)]. The color differences between before resin cementation (1) after resin cementation procedure (2) and after thermocycling (3) were evaluated. Calculation of color differences was performed utilizing the following equitation:

$\Delta E=[(\Delta L*)^2+(\Delta a*)^2+(\Delta b*)^2]^{1/2}$ (19).

Shear bond strength (SBS) test and fracture analysis

Resin cemented ceramic specimens were settled in the autopolymerizing acrylic resin mold for a SBS test after thermocycling. The samples were attached perpendicularly to their bases with a special tip in a universal testing machine (Shimadzu AG-X, Tokyo, Japan). The crosshead speed of the testing machine was settled 1 mm/min till the bonding failure happened. The load values (kilogram) should convert to megapascals (MPa). This convention was provided through the convention of failure load to Newtons (N) and was divided by the bonding area (square millimeter)(20). The eventual stress (MPa) of the resin cement-ceramic was measured as follows (20):

Stress (MPa)=Failure load (N) /Surface area (pi x r^2) (mm^2)

Statistical analysis

Shapiro-Wilk test was used testing the normality of distribution of continuous variables. One-way ANOVA and LSD test (for normal data), Kruskal Wallis and Dunn multiple comparison tests (for non-normal data) were used to compare between six independent groups and Paired t-test and Repeated measures of ANOVA (for normal data) Freidman test (for non-normal data) were applied to investigate within-group differences. Mean ± standard deviations (mean ± SD) were given as descriptive statistics. Statistical analysis was carried out using SPSS for Windows version 22.0 and a p-value < 0.05 was accepted as statistically significant.

RESULTS

This research enrolled sixty lithium disilicate ceramic samples, which were cemented different adhesive systems. The three different periods of color change measurements included the L*, a*, b*, and ΔE values for the sixty ceramic samples. Also, a BS test was applied for cemented samples.

1. L* values

In the current study, brightness changes were symbolized as L*. L1= First the values of ceramic before cementation; L2= immediately after cementation; L3= after thermocycling.

There were statistically significant differences between L1 and L2, L3 within group comparisons ($p < 0.05$). In the contrary, there were no significant differences between L1, L2, L3 for the A3-RX group. All L values decreased after the resin cementation. In addition, the A3-VL group had significantly lowest values of L* for L2* and L3*, but there were no statistically significant differences with the A3-P group (Table 1).

2. a * values (Redness)

In the current study, redness changes were symbolized as a*. a1= First a values of ceramic, before cementation; a2= immediately after cementation; a3= after thermocycling.

There were no statistically significant differences between a2 and a3 measurements within group comparisons. A3-RX group has the highest a* values and statistically significant differences between all groups except the A3-P group (Table 2).

3. b* values (Yellowness)

In this study, yellowness changes were symbolized as b*. b1= First a values of ceramic, before cementation; b2= immediately after cementation; b3= after thermocycling.

There were no significant differences in the b2*, b3* values within-group comparisons. All of the b values increased after the resin cementation. A3-RX group has the highest b* values, and there were statistically significant differences between all groups (Table 3).

4. ΔE values and SBS

In the current study, color changes were symbolized as ΔE. ΔE1= First color of ceramic (FC)-immediately after cementation; ΔE2= FC–after thermocycling.

There were statistically significant differences in the ΔE1 and ΔE2 values within the groups. A1-VL group had the highest ΔE values, and there were no statistically significant differences between the A1-RX and A3-VL groups (Table 4). A3-P group had the lowest ΔE values, and there were no statistically significant differences between the A1-P and A3-RX groups (Table 4).

There were statistically significant differences between the SBS results of the groups. A3-RX group had the highest BS results, and there were no statistically significant differences between the A1-VL group (Table 4). A3-P group had the lowest BS results, and there were no statistically significant differences between the A3-VL group (Table 4).

Table 1. Comparisons of the L* values (translucency) results of the groups

Groups	A1-P (mean±SD)	A1-RX (mean±SD)	A1-VL (mean±SD)	A3-P (mean±SD)	A3-RX (mean±SD)	A3-VL (mean±SD)	P between groups
L1	99.38 ± 0.55^{a}	99.6 ± 0.28^{a}	99.44 ± 0.41^{a}	93.65 ± 2.38^{b}	92.85 ± 1.91^{b}	94.36 ± 1.86^{b}	0.001*
L2	96.28 ± 1.25c.^{e}	98.78 ± 0.67^{c}	89.03 ± 1.14^{d}	88.5 ± 1.86$^{d.f}$	90.5 ± 1.16$^{b.d.e}$	83.71 ± 2.48^{f}	0.001*
L3	96.23 ± 1.73$^{c.b}$	97.84 ± 2.74^{c}	89.03 ± 1.12^{d}	88.4 ± 1.62$^{d.f}$	90.46 ± 1.51$^{b.d}$	83.97 ± 2.94^{f}	0.001*
P within groups	0.002*	0.002*	0.001*	0.001*	0.025*	0.001*	

There is no significant differences between the same lowercase letters.
*Significant at 0.05 level. Kruskal Wallis. Dunn test and Friedman test were used.

Table 2. Comparisons of the a* values (redness) results of the groups

Groups	A1-P (mean±SD)	A1-RX (mean±SD)	A1-VL (mean±SD)	A3-P (mean±SD)	A3-RX (mean±SD)	A3-VL (mean±SD)	P between groups
a1	-0.6 ± 0.2^{a}	-0.51 ± 0.1^{a}	-0.82 ± 0.22^{a}	3.88 ± 0.43^{b}	3.73 ± 0.63^{b}	4.23 ± 0.54^{b}	0.001*
a2	1.55 ± 0.3$^{c.d}$	2.17 ± 0.57$^{c.e}$	-0.11 ± 0.34^{d}	4.75 ± 0.55$^{g.f}$	6 ± 0.34^{f}	3.02 ± 0.64$^{e.g}$	0.001*
a3	1.47 ± 0.28$^{c.h}$	2.03 ± 0.58$^{c.e}$	-0.2 ± 0.28$^{a.d.h}$	4.75 ± 0.46$^{g.f}$	5.77 ± 0.46^{f}	3.04 ± 0.57$^{e.g}$	0.001*
P within groups	0.001*	0.001*	0.001*	0.004*	0.001*	0.001*	

There is no significant differences between the same lowercase letters.
*Significant at 0.05 level. Kruskal Wallis. Dunn test and Friedman test were used.

Table 3. Comparisons of the b* values (yellowness) results of the groups

Groups	A1-P (mean±SD)	A1-RX (mean±SD)	A1-VL (mean±SD)	A3-P (mean±SD)	A3-RX (mean±SD)	A3-VL (mean±SD)	P between groups
b1	14.62 ± 1.05^{a}	13.73 ± 0.8^{a}	14.39 ± 1.09^{a}	35 ± 1.47^{b}	34.51 ± 0.74^{b}	35.8 ± 1.64^{b}	0.001*
b2	21.94 ± 0.74^{c}	24.74 ± 0.91^{d}	19.56 ± 1.51^{e}	38.43 ± 0.52^{f}	41.59 ± 0.89^{g}	34.88 ± 0.87$^{h.b}$	0.001*
b3	21.82 ± 0.81^{c}	24.73 ± 1.19^{d}	19.62 ± 1.59^{e}	38.39 ± 1.08^{f}	41.73 ± 0.95^{g}	34.69 ± 0.8^{h}	0.001*
P within groups	0.001*	0.001*	0.001*	0.001*	0.001*	0.031	

There is no significant differences between the same lowercase letters.
*Significant at 0.05 level. One way ANOVA. repeated measures of ANOVA and LSD test were used.

Table 4. Comparisons of the ΔE values (color change) and shear bond strength results of the groups

Groups	A1-P (mean±SD)	A1-RX (mean±SD)	A1-VL (mean±SD)	A3-P (mean±SD)	A3-RX (mean±SD)	A3-VL (mean±SD)	P between groups
Deltae1	8.33 ± 0.98^{a}	11.39 ± 1.07^{c}	11.75 ± 0.93^{c}	6.67 ± 1.88^{b}	7.94 ± 0.8$^{a.b}$	10.86 ± 3.3^{c}	0.001*
Deltae2	8.3 ± 1.15$^{a.b}$	11.75 ± 1.38^{c}	11.78 ± 0.92^{c}	6.77 ± 1.83^{b}	8.12 ± 1.21^{b}	10.63 ± 3.6^{c}	0.001*
P within groups	0.993	0.377	0.891	0.659	0.501	0.402	
Bond Strength	5.56 ± 1.08^{A}	4.07 ± 0.74^{D}	5.72 ± 1.01$^{A.B}$	3.22 ± 0.41^{C}	6.42 ± 0.86^{B}	3.25 ± 0.36^{C}	0.001*

There is no significant differences between the same lowercase and uppercase letters.
*Significant at 0.05 level. One way ANOVA and LSD were used..

DISCUSSION

Resin cement is utilized widely as luting material for full ceramic restorations. Resin cement is classified into three groups according to the adhesive system: self-adhesive, self-etch and total-etch. In the current study, A1 and A3 shade of ceramic groups showed final color changes cemented with self-etch and total-etch systems and there were no statistically significant differences within groups. However, the self-adhesive system may cause more color change ($p< 0.05$) in the A1 group of ceramic. Therefore, the first null hypothesis, type of resin cement would not affect the final color of ceramic was rejected. All of the resin cement influenced the ultimate color of ceramics same as the study of Atay et al. (21), also Dede et al. (22) reported that resin cements in same shade from different manufacturers had different effects on the color of lithium disilicate ceramic . Resin cement is important both used for cementation and decided the final color of restoration (23). In this study, the L values of all groups decreased after cementation the same as previous studies (24, 25). Changes in L* (translucency) values are significant for the color durability of ceramics. The a* (redness) and b* (yellowness) values increased after cementation in this study consistent with the literature (21). The findings obtained in this study showed that there were significant differences in ΔE values between the resin cement. This can be based on the variation in the chemical structure of the resin cement, their tendency to water absorption and their degree of polymerization (21). In addition, much resin cement had the ester and hydroxyl groups of the Bis-GMA molecules. These molecules absorb water and are responsible for the hydrolysis. These hygroscopic and hydrolytic effects have been crucial for the color changes of resin cement (26). On the other hand, characteristics of the light transmittance affect the color of resin materials (27, 28). The quantity of transmitted, scattered and absorbed light of composite resins are essentially decided by the opaque content, pigment and filler of the material (29). A ΔE value < 3.3 is a clinically acceptable color change for ceramic restorations; it is very hard to recognize color changes in restorations with the human eyes (30). Demirkol et al.(30) reported that cause of high ΔE values may be originated from the material's optic structures, which is defined as translucent ceramic due to the optical composition of a glass matrix that decreases internal scattering of the light as it passes through the ceramic. Therefore, medium translucency lithium disilicate (E.maxCAD MT block) ceramic, which was used in this study with a glass matrix structure that decreased internal light scattering as it translated through the ceramic may cause high ΔE values in this study.

Thermal aging is one of the methods used for in-vitro studies to simulate clinical conditions. Thermocycling was preferred by dental researchers because of reflecting intraoral circumstances by exposing the samples to moisture via heat exchange (24, 31). Atay et al.(21) demonstrated, all resin cements used in their study were showed clinically acceptable color change after thermocycling. Also, drawing on the previous studies (24), the 10.000 cycle of thermocycling was made in this study and did not cause any acceptable color changes of lithium disilicate ceramics. Thus, the second null hypothesis, thermal aging, would not influence the ultimate color of ceramic, was accepted.

The BS of resin cement depends on several factors, such as adhesion type (total-etc, self-etch, self-adhesive), polymerization process, characterization of resin cement, ceramic thickness, ceramic shade, material features, the translucency of resin and ceramic. The literature showed that the darker ceramics absorbs more light compared to the lighter ceramics (32). Ceramic shade influence the transmittance of the light and lighter shades demonstrated fewer absorption coefficients, so lighter shades ceramics were more translucent (32), which supports the findings of the current study according to L* values results.

A certain percentage of the curing of the luting resin underlying the material is important for the survival of a ceramic (33, 34). Previous studies showed that the degree of polymerization was affected negatively with the absorption of the light from the pigments of the ceramic (35, 36). Thus, more energy transmit the photo-cured resin cement when there is a lighter ceramic specimen(37). In the current study, the BS values of all of the adhesive systems were much higher for the more lighter ceramic shade (A1) same as the literature (32, 38). Thus, the third null hypothesis, the ceramic shade, would not affect the BS of the resin cement was rejected. In addition, A3-RX group (self-adhesive resin cement) was showed higher bond strength in this study same with Piwowarczyk et al.(16) This result may be based on continued polimerization after initial light polymerizing during thermocycling process (39) or silane coupling agent of self-adhesive system which provides chemical bonding on slica-based ceramics (40).

This research is limited by composing only one shade of resin cement and one kind of ceramic. The second limitation of this study was the thermal aging procedure performed before the BS so that a control group comparison for the BS of resin cement could not be made. Thus, future studies are required to research other factors affecting the optical and mechanical attitude of ceramics and resin cement.

CONCLUSION

The relation between ceramic and resin cement is constantly improving. With the development of CAD-CAM systems, this issue has become even more important for clinicians. In our study, the effects of resin cements and ceramic shade on the final color and bond strength were investigated. According to the current study all of the adhesive systems affected the final color of ceramics. However, when the color change after thermocycling was examined, there were no significant differences between the cementation stages within-group comparisons. Furthermore, the shade of the ceramics affected the BS of the resin cement. Consequently, in order for the restoration to be successful, clinicians should be attentive in choosing ceramic color and resin cement.

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