Dental
Communication

Biosc.Biotech.Res.Comm. Vol 13 (1) Jan- March 2020 123-128

BBRC
Bioscience Biotechnology Research Communications

Adhesive Bond Strength of Bioactive Cement to Er Cr YSGG Laser Treated Lithium Disilicate Ceramics

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ABSTRACT

The aim of the study was to evaluate the adhesive bond strength of bioactive cements to lithium disilicate ceramics in comparison to the resin based luting cements with two surface treatments, hydrofluoric acid (HF) and Er Cr YSGG laser (ECL). Ninety ceramic disks were fabricated and divided into three groups (n=30) based on the surface treatment employed; HF-S, HF-S (with Silane as control), ECL-2, for 2 minute duration, and ECL-4 for 2 minute duration and ECL4 for 4 minute duration. The laser treated groups were prepared with the use of a gold handpiece using an MZ10 tip size (Er Cr: YSGG, water lase I plus). Surface treated disks in each group were further divided into three sub groups (n= 10) based on the type of cement applied; Bioactive cements, Rely X unicem and Rely X ARC. Subsequent to the application of the cements, Multicore Flow composite was build up with the help of the putty mould; specimens were placed in a thermocycler for 5000 cycles at 5^0C and 55^0C. Each specimen disk was tested under the universal testing machine for shear bond strength (SBS). Data was analysed using analysis of variance and Tukey's – Kramer multiple tests ($p<0.05$). The maximum mean value for shear bond strength achieved was 23.55 (±2.26) for Rely-x unicem¬-HF-S and the minimum mean value was 14.30 (±2.08) for the group bioactive and ECL-2. Therefore, it was concluded that the adhesive bond strength of the bioactive cements on lithum disilicate ceramics was lower than the resin based luting cements. Difference in surface treatments did not influence the adhesive bond strength of bioactive cements to the lithium disilicate ceramic.

KEY WORDS: LITHIUM DISILICATE, ER CR YSGG LASERS, SELF ETCH RESIN LUTING AGENTS, RELY X AND BIOACTIVE CEMENTS.

INTRODUCTION

The most popular technology that has become the centre of choice in dental restoration recently is all ceramic restorations (Zarone et al., 2016). These restorations offer a series of advantage over the metal porcelain used, such as biocompatibility, low thermal conductivity, high optical properties, better chemical stability and comparable coefficients of thermal expansion to the teeth (Niu et al., 2014, Niu et al., 2013, Aboushelib and Sleem, 2014). Lithium disilicate (LD) is the most common type of contemporary adhesive glass ceramics employed in modern dentistry (Elsayed et al., 2017). In Comparison to zirconia, lithium disilicate ceramics are processed through the hot press technique exhibiting greater potential for restoration displaying translucency and aesthetics (Palla et al., 2018). The lithium silicate ceramics comprise of silica glass matrix and lithium oxide (Li_2O) that presents greater flexural strength compared to the leucite-reinforced glass ceramics (Klosa et al., 2013). The crystals in the lithium disilicate ceramic are the unit of foundation that offers better mechanical strength to the

ARTICLE INFORMATION

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Received 5th Jan 2020 Accepted after revision 10th March 2020
Print ISSN: 0974-6455 Online ISSN: 2321-4007 CODEN: BBRCBA

Thomson Reuters ISI Web of Science Clarivate Analytics USA and Crossref Indexed Journal

NAAS Journal Score 2020 (4.31) SJIF: 2019 (4.196)
A Society of Science and Nature Publication,

Online Contents Available at: http//www.bbrc.in/
DOI: 10.21786/bbrc/13.1/21

ceramics such as fracture toughness, chemical durability and abrasion resistance (Lee et al., 2017).

Efforts have been made in formulating a resin based luting agent with improved mechanical strength, toughness and fracture resistance (Koizumi et al., 2012). These bioactive cements have demonstrated improved and better functional properties critical for clinical function and survival. The volumetric shrinkage of these materials is <1.7% compared to the resin based composites (Al-Sowygh, 2017). These materials have successfully presented with properties equivalent to glass ionomer including intimate contact with the dentin walls and display hydrophilicity (Pekkan and Oezcan, 2012). Studies have pointed out that the adhesive bond between the ceramic restoration and the cement is the main factor for fracture resistance and marginal adaptation rather than the strength of the ceramic (Özcan and Mese, 2012, Koizumi et al., 2012).

Few authors have employed different surface treatments for stronger adhesive bond strength between LD ceramics and bioactive cements through increasing surface roughness (Bagis et al., 2011, Al-Sowygh, 2017). According to Koizumi et al. (2012), sandblasting reported serious damage to the ceramic surface resulting in low flexural strength; however, hydrofluoric surface treatment chemically modified the surface creating irregular interlocking surface. This technique improved interfacial bonding through an increase in the bonding area. In many instances, clinicians have applied silane coupling agent to decrease the wettability contact angle and increase the free energy of the ceramic surface (Koizumi et al., 2012, Al-Sowygh, 2017). Recently, the use of lasers in modern dentistry has led to increased beneficial effects in terms of painless removal of infected dentine and long term retention of the restoration. Er Cr YSGG (ECL) laser working is based on micro explosion creating surface irregularities for micro retention, which is considered favourable surface treatment compared to hydrofluoric acid that has the tendency to ablate the tissue in process of etching surface. Hence, using hydrofluoric acid on the chairside is risky for the patient, (Kursoglu et al., 2013, Vohra et al., 2019).

In the current view, the self-etched resin had replaced the conventional bonding technique of the glass ceramics using the resin based luting agents (Yilmaz-Savas et al., 2016). Self etch agents such as Rely X unicem are commonly employed as bonding cement in ceramic restoration. The recent introduction of Bioactive cements with inherent beneficial properties such as an efficient dentinal bond, resin tag supported adhesion, caries resistance, continuous fluoride release, dentin formation, and pulp protection compels to opt as a luting agent in the ceramic restoration (Al-Sowygh, 2017, Chaharom et al., 2018). Therefore, it is hypothesized Bioactive cements would show higher adhesive bond strength in Er Cr YSGG laser treated disilicate glass ceramics. Literature provides series of studies comparing the self etch and conventional luting agents bond strength in ceramics; however, there is no plausible data for displaying the adhesive bond strength in regards to bioactive cements in ceramic under different surface treatment (Yaman et al., 2014, Aguiar et al., 2014). Thus, the present study intends to evaluate the adhesive bond strength of bioactive cements in Lithium disilicate ceramics under two surface treatment; hydrofluoric acid and Er Cr YSGG treatment in comparison to the resin based luting agents.

MATERIALS AND METHODS

The present in vitro study evaluated the adhesive bond strength of Bioactive cement to Er-Cr-YSGG laser treated Lithium disilicate ceramics. Under the heat pressed technique, a total of 90 lithium disilicate ceramic disks were fabricated (EMax Press, Ivoclar Vivadent, AG, Schaan/Liechtenstein MO1 lot no.10721) with height (Ø) and diameter of 3 mm x 3 mm, respectively. The materials details are provided in table 1. To achieve a flat base, the ceramic disc was embedded into the acrylic resins with PVC (polyvinyl chloride) particles. The disks were finished through grinding under running water using silicon carbide paper on a polishing machine (Buehler Polishing Machine type: 49-5100-230, No 620-PXB-22061, Germany). The 90 specimens were initially divided into three main groups (n= 30) followed by distribution into 3 groups in each category (n= 10) depending upon the surface treatment and the type of luting agent used, correspondingly.The group distribution according to the surface treatment is described as follows:

Group 1 HF -S (silane) (Control): The specimen's surface was treated with the 9.6% concentration hydrofluoric acid (Pulpdent Corporation, USA). The etchant is applied for 1 min and washed along with air dried for 2 minutes. Subsequently, after cleaning, silane adhesive (Silane bond enhancer, Pulpdent- Watertown, MA, USA) was smeared over the etched surface using a micro brush and allowed to dry for 5 mins.

Group 2 ECL-2: The specimen's surface was treated with Er Cr:YSGG laser (water lase I plus, Biolase, USA) at a power of 3.75W and frequency of 15 Hz (L1), air-water 90-70% for 2 mins. Followed by the application of silane adhesive with micro brush and allowing it to dry for 5 mins.

Group 3 ECL-4: A similar procedure as group 2 proceeded; however, the laser surface treatment prolonged for 4 Minutes. Moreover, the specimens in both laser treated groups were prepared with the use of a gold handpiece using an MZ10 tip size (Er Cr:YSGG , water lase I plus, Biolase, USA). The focus of the handpiece was in the centre for 30 seconds followed by a standard clockwise rotational movement for the remaining time at 2 mm distance. Subsequently, each of the three groups was further divided according to the type of luting agent used.

Group HF-S-Bioactive: Bonding with ACTIVA- Bioactive cement (Pulpdent- Watertown, MA, USA).
Group HF-S-Rely-X: Bonding with RelyX Unicem (3M, St. Paul, MN, USA)

Group HF-S-Rely-ARC: Bonding with RelyX-ARC (3M, St. Paul, MN, USA)
Group ECL-2-Bioactive: Bonding with ACTIVA- Bioactive cement
Group ECL-2-Rely-X: Bonding with RelyX Unicem,
Group ECL-2:Rely-ARC: Bonding with RelyX-ARC
Group ECL-4-Bioactive: Bonding with ACTIVA- Bioactive cement
Group ECL-4-Rely-X: Bonding with RelyX Unicem
Group ECL-4:Rely-ARC: Bonding with RelyX-ARC

Following the preparation, each of the specimen disks was coated with either of three different types of the luting agent. The luting agents were polymerised using curing light (Bluephase®, Ivoclar Vivadent, Schaan, Liechtenstein) at a light intensity of 1,000 mW/cm2 for 10 seconds. Consequently, after complete preparation of the specimen, using a putty mould (Polyvinyl siloxane-Express, 3M Center St. Paul, MN, USA) the resin composite (Multicore flow, Ivoclar Vivadent Schaan, Liechtenstein) (Ø 2 mm, depth 2 mm) was build upon the specimen disk and polymerised from each side for 40 seconds in total. Each specimen was placed in thermocycler afterward (Thermocycler SD Mechatronik, GmbH Dental Research Equipment, Germany) for 5000 cycles at 5^0C and 55^0C (dwelling time: Cold bath, 30 sec; Hot bath, 30 sec).

Subsequent to thermocycling, the specimen from each group was placed under load in the universal testing machine (Instron 8500 Plus, 100 Royal St. Canton USA). The chisel of the universal machine is placed on the specimen at a perpendicular direction at a control force rate of 1 mm/min until the build-up materials were detached from the ceramic surface. To examine the failure mode of the detached surface, the digital microscope (Hirox- KH7700) was used. The failures are observed at three levels: adhesive failure at the luting agent and ceramic interface, cohesive: failure within the cement or the ceramic or composite materials, and admixed: Failure at luting/ceramic interface, progressing into luting cement.

All the data collected for the adhesive bond strength was processed and tabulated using a statistical program for social science (SPSS). The analysis of normally distributed data was performed using Kolmogorov-Smirnov test. The shear bond strength was analysed using analysis of variance and Tukey's – Kramer multiple tests ($p<0.05$). Furthermore, all data recorded was subjected to Levene's test of homogeneity of variance ($\alpha= 0.05$) following the assumption of equal variances.

RESULTS AND DISCUSSION

Kolmogorov-Smirnov test presented with normal distribution of data. The analysis of variance (ANOVA) displayed a significant difference in the adhesive bond strength of bioactive cement to Er Cr YSGG laser treated Lithium disilicate ceramics in comparison to the resin based luting cements ($p=0.01$). The outcome of the present study displayed significant difference ($p<0.05$) for the two different surface treatments employed in the bioactive cement group. Resin luting cements also presented significant difference with the two surface treatment; HF and Er Cr YSGG laser treatment employed ($p<0.05$).

The present study demonstrated that the maximum mean value for adhesive shear bond strength was 23.55 (±2.26)

Table 1: Details of the materials used in the study

Material	Composition Details	Filler
RelyX™ Unicem 3M, ESPE, St Paul, MN,USA	Methacrylated phosphoric ester Dimethacrylate (TEGDMA, Bis-GMA), Stabiliser, Peroxy compound, Substituted pyrimidine Pigment, Calcium hydroxide	Barium glass, ytterbium trifluoride, and mixed oxide
RelyX™ ARC 3M, ESPE, St Paul.MN. USA	TEGDMA, Bis-GMA, Benzoic peroxide, amine, photo-initiator, pigment.	Barium-alumino-fluoro-borosilicate glass, Strontium alumino -fluoro-silicate glass , Zirconia powder
ACTIVA TM, Bioactive dental cement Pulpdent, Watertown, MA, USA	Blend of diurethane and other methacrylates with modified polyacrylic acid (44.6%) contain no Bisphenol A, No Bis-GMA, No BPA derivatives	Amorphous silica (6.7%) Sodium fluoride (0.75%)

for the group Rely-x unicem ¬- HF-S and the minimum mean value was 14.30 (±2.08) for the group bioactive cement-ECL-2. The means and standard deviations of bond strength obtained are summarized in table 2. Each luting agent used in the study presented with a different set of bond strength mean value varying according to the employed surface treatments, respectively. Comparing the luting agent outcomes indicated an evident difference in shear bond strength of Activa and Relyx cements (unicem and ARC) ($p < 0.01$). The surface prepared with the silanised process (HF-S) specifically produced better shear bond strength results. Multiple comparisons test demonstrated a significant difference between the bioactive and Relyx cements (unicem and ARC) only under the hydrofluoric – silane surface treatment. In contrast, the laser treated surface (ECL-2 and ECL-4) presented with an evident difference between bioactive and Rely – ARC cement whereas exhibiting comparable results between the Activa and RelyX Unicem. Despite the fact there was a significant difference in the result; nevertheless, Bioactive cements presented with lower bond strength compared to the resin luting agents.

Analysing the effect of the surface treatment on each material, the result of bioactive cements presented a significant difference between the two laser groups [ECL-2 (14.30 (2.08), ECL-4 (17.37 (2.23)] and HF-S group (17. 45 (2.40)) with ECL-2 (14.30 (2.08)) respectively. Whereas comparable outcome was observed between the HF-S and ECL -4 groups ($p > 0.05$). Rely-X cements (unicem and ARC) showed an evident difference between the HF-S and laser treated group (ECL-2); however, Rely X unicem presented similar results between the two types of laser treatment. In addition, a significant difference was observed between the HF-S and laser treated group (ECL-4) in Rely X unicem while no significant difference in Rely X ARC. Thus, bioactive cements showed an insignificant difference in adhesive bond strength in Er-Cr-YSGG treated lithium disilicate compared to the HF-S group. Failure mode outcomes exhibited adhesive failure in the majority of the specimens compared to cohesive and admixed failures. The result indicated that only specimens in ECL-2 group presented in Bioactive material showed 100% adhesive failure. Overall, only 20 – 30% of observed failures were admixed type. The results directed that the ECL-2 had a profound effect on the shear bond strength of bioactive cement compared to Rely X cements. These Rely X cements in between them demonstrated a comparable number of failures in each surface treatment. However, ECL- 4 treatment did not show an apparent difference in the type of failure among either cement.

The present study evaluates the adhesive bond strength of bioactive cement to Er-Cr-YSGG laser treated lithium disilicate ceramics based on the hypothesis that bioactive cements exhibit better adhesive bond strength compared to self etch resin based luting agents. The study compared bonding strength of bioactive cements under hydrofluoric acid and Er Cr YSGG surface treatment for 2 and 4 minute duration. Self etched resin cements (Rely X unicem) demonstrated higher bond strength compared to the bioactive cements. Furthermore, under different surface treatments bioactive cements showed significant change in shear bond strength to LD ceramics. Therefore the hypothesis was rejected. A multitude of explanations can be provided for the outcomes in the present study.

To maintain the homogeneity and standardisation the adhesive shear bond strength was assessed using a universal testing machine. All the specimens were thermocycled to ensure the shear bond strength tested was in a standard environment. Studies have reported that thermocycling determines a positive change in surface bonding, which has beneficial long term effects on the ceramic restoration (Brum et al., 2011, Lee et al., 2017). Thermocycling aids in the water resorption property that causes the ageing of the bond resulting in weak bonds (Brum et al., 2011). The bioactive cements in previous studies have demonstrated an insignificant decrease in adhesive bond strength to ceramics compared to resin based luting cement despite the fact some detrimental effects are associated with water absorptiondue to the methacrylate in resin cements (Brum et al., 2011, Ahrari et al., 2017). In the present study, Rely X cements showed higher bond strength compared to the bioactive cements. Rely X unicem (self etched) displayed higher mean value in HF surface treatment while Rely X ARC (resin based) displayed high mean value in laser treated group. Nevertheless, comparable bond strength was observed between the self etch and resin cements. The results of the present study partially supported the outcomes noted by the previous studies regarding higher bond strength in bioactive cements (Al-Sowegh, 2017).

The plausible explanation identified was that self etch cements uses dual cure method to polymerise; therefore, presented with better bond strength (Chaharom et al.,

Table 2: Means and SD for shear bond strength among study groups

Luting agent	HF-S	ECL-2	ECL-4	P value
Bioactive (Activa)	17. 45 (2.40)^{aA}	14.30 (2.08)^{bB}	17.37 (2.23)$^{a\ A}$	< 0.01
RelyX- Unicem	23.55 (2.26)^{bA}	16.37(3.11)^{abB}	18.40 (2.61)^{abB}	
Rely-X ARC	21.37 (2.38)^{bA}	18.33 (2.83)^{aB}	20.65 (2.76)^{bAB}	

Dissimilar superscript small alphabets in same column show significant difference ($p < 0.05$)
Dissimilar superscript capital alphabets in same row show significant difference ($p < 0.05$)

2018). The lower bond strength of the bioactive cements suggested that the durability of the bond depends upon the nature of the mechanical bond between the ceramics and cements (Gurney et al., 2016). As ceramics are inert in nature they do not display chemical change that would contribute to the bonding strength (Kursoglu et al., 2013). In addition, theoretically, the silane bond on the ceramic surface binds effectively to the micro retentive etched surface. However, the bioactive cements offer limited methacrylate group for bonding to the silane groups compared to resin based cements; thus, resulting in low bond strength (Ahrari et al., 2017, Al-Sowygh, 2017).

Several studies documented the evident effect of hydrofluoric acid on the interfacial bonding surface (Lee et al., 2017, Kalavacharla et al., 2015). Whereas few previous studies used different intensity laser application

Table 3: Failure type percentage among study groups

Study Groups	Adhesive	Cohesive	Mixed
Group HF-S-Bioactive	70	0	30
Group HF-S-RelyX	80	0	20
Group HF-S-Rely-ARC	80	0	20
Group ECL-2-Bioactive	100	0	0
Group ECL-2-RelyX	70	0	30
Group ECL-2-Rely-ARC	80	0	20
Group ECL-4-Bioactive	70	0	30
Group ECL-4-RelyX	70	0	30
Group ECL-4-Rely-ARC	60	0	40

to identify the effect on the ceramic surface (Passia et al., 2015, Kalavacharla et al., 2015). Therefore, to observe the evident effect of the varying surface treatment on the ceramic surface, two types of surface treatments were employed HF-S and Er Cr YSGG laser treatment. In the present study, the duration of the laser was varied in order to observe the effect on the adhesive bond strength to lithium disilicate ceramics. It is reported that laser treated surface often displays greater surface roughness irregularities and patterns, unlike the HF that dissolves the ceramics glassy matrix to form interlocking surface (Neis et al., 2015). The interlock mechanism implicates better micromechanical retention compared to an irregular surface. Intriguingly, a prolonged period of laser application increase surface roughness; nevertheless, the laser produces excessive heat that results in the weakening and over destruction of the surface (Gurney et al., 2016). Hence, laser treated ceramics with prolonged duration demonstrated higher bond strengths but comparatively less than the HF study groups.

The mode of failure assessment presented a higher number of adhesive failures followed by admixed. No specimen demonstrated cohesive failure in the present study attesting no measurement of material strength. The bioactive cements displayed an increased number of adhesive failure only in ECL 2 group while comparable results were appreciated in other groups. This indicates employing bioactive cements at laser treated surface with 2 minutes duration results in the formation of weak adhesive bonds comparatively to laser treated surface at 4 minutes duration. Nevertheless, failure mode assessment displayed comparable adhesive bond strength of bioactive cements and resin luting cements.

Certain limitations were identified in the present study despite the fact it provided a clear comparison between the bioactive cements and resin based luting agents. The results of the study are applicable only in reference to the particular surface treatments employed, particularly the type of laser (Er Cr YSGG) used. For better understanding of the bonding strength of bioactive cements to ceramics, studies comparing different types of ceramics are recommended. Although the study presented lower bond strength of bioactive cements to the ceramics; however, the status of fracture resistance of bioactive cements is questionable and needs to be evaluated. Lithium disilicate ceramics are well established restoration used in dentistry exhibiting inherent properties necessary for efficient adhesive bonding and long term retentive restoration. The mechanical and biological properties of bioactive cements shows great potential in bonding with the tooth; however, literature provides limited data with respect to its bonding with the ceramics. Therefore it is recommended to evaluate the fracture resistance of these Biocements prior to the application of different laser with different types of ceramic restorations.

CONCLUSION

The adhesive bond strength of the bioactive cements to lithum disilicate ceramics was lower than the resin based luting cements. Use of 4 minutes of Er Cr YSGG laser treatment displayed an increase in the adhesive bond strength of bioactive cement to Lithium disilicate ceramics. Despite the low bond strength, Bioactive-luting agents exhibited durable bond strength to the ceramics.

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