



## Shear bond strength between lithium disilicate ceramic and different luting cements

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### Abstract

This study aimed to evaluate and compare the shear bond strength and durability of three different luting cements to lithium disilicate ceramic. A total of 96 ceramic discs were fabricated from IPS e.max press, half of them large (8.5mm X 3mm) and the other half was small (5.5mm X 3mm). The small discs bonded to the large ones using three different cements (glass ionomer cement, Variolink II and RelyX Ultimate). The samples were stored in distilled water at 37°C. Half of them stored for 24hrs, while the other stored for one month and then thermocycled between 5°C and 55°C for 500 cycles before testing. The shear test was applied using universal test machine at a crosshead speed of 1 mm/min. One way ANOVA, LSD and student's t-test were used for analysis of the data ( $P \leq 0.05$ ). In the non-aged condition, the highest mean shear bond strength was recorded for group RelyX Ultimate (23.549 MPa) and the lowest was of group glass ionomer (4.745MPa). After aging the shear bond strength values of the glass ionomer and RelyX Ultimate were significantly lowered to (0.308MPa) and (15.435MPa) respectively, however the bond strength of Variolink II was not significantly changed. In conclusion the adhesive resin cement Variolink II is favorable to be used for luting lithium disilicate ceramic restorations.

Keywords: Shear bond strength, Ceramic restorations, Lithium disilicate, Luting cements, Adhesive resin cements.

### Introduction

In the last years there have been increased demands for esthetic restorations due to the popularity of all-ceramic materials as an alternative to metal-ceramic restorations. Dental ceramics have the optimal esthetic properties that enable the production of highly esthetic restoration that better reproduce the appearance of the natural teeth. Lithium disilicate glass ceramic is one of the most commonly used ceramic materials for its esthetic and fracture-resistance; it is commercially marketed by Ivoclar Vivadent in 1998 as IPS Empress II, now called IPS e.max Press and IPS e.max CAD. The crystal structure of IPS e.max lithium disilicate gives the material excellent strength (360–400MPa) and durability as well as outstanding optical properties. It is a versatile material that can be used in many indications ranging from inlay and only to 3-unit fixed partial denture in the anterior and premolar region. Due to its high strength, IPS e.max Press restorations can be cemented by adhesive, self-adhesive or conventional cementation depending on the indication (Fabianelli *et al.*, 2006; Tysowsky, 2009; Marocho *et al.*, 2012). In an all-ceramic

restoration, the cement has the task of supporting this brittle material during loading due to the lack of a strong metal substructure. Quantitative fractography and finite element analysis of failed ceramic restorations demonstrated that fractures occur in All-ceramic crowns as a result of the extension of pre-existing surface defects that occupy the inner "fit" surface of the restoration under tensile loading. Clinically, the dental cement used to retain the restoration on the prepared tooth structure influence the environment of the inner surface defects (Fleming and Addison, 2009). Significant strengthening of dental ceramics when bonded to resin cement had been demonstrated by many in vitro studies (Pagniano *et al.*, 2005; Fleming *et al.*, 2006; Alakhras, 2011). Thus, the cementation process and bonding effectiveness play a vital role in the clinical success of all-ceramic restorations (Zareen *et al.*, 2013). However, more information are required about which composite resin cement and ceramic surface conditioning method produce the most durable bond strength. The aims of this study were to evaluate and compare the shear bond strength of three different luting cements

with lithium disilicate ceramic, also to evaluate the effect of aging on the bond.

### Materials and Methods

A total of ninety six ceramic discs were fabricated from lithium disilicate ceramic ingots (IPS e-max press, Ivoclar Vivadent) using the hot pressing fabrication technique. Forty eight large discs were with dimensions of (8.5mm diameter and 3mm thickness), the other 48 discs were small with dimensions of (5.5mm diameter and 3mm thickness). The ceramic discs were ground flat by abrading with wet 600 grit aluminum oxide abrasive papers using a custom made grinding machine (Gokhan *et al.*, 2005). Every abrasive paper was used for grinding of 8 discs and changed. A custom made holder were fabricated to facilitate the handling of the ceramic discs during the grinding process which take about 1 minute for each disc under water cooling. The ceramic discs were then cleaned in ultrasonic bath containing distilled water for 5min and air dried (Patel *et al.*, 2011). The large ceramic discs were embedded in acrylic blocks leaving 1mm of the disc out of the acrylic. The ceramic discs were then randomly divided into three groups depending on the luting cement that would be used for bonding. Each group contained sixteen large discs embedded in acrylic and sixteen small free discs. The small discs were bonded to the larger ones using three different cements, thus we get 16 specimens from each group.

Group A: Bonded with conventional glass ionomer cement (Vivaglass CEM PL, Ivoclar Vivadent).

Group B: Bonded with the resin cement Variolink II (Ivoclar Vivadent).

Group C: Bonded with the resin cement RelyX Ultimate (3M ESPE).

The bonding surfaces of the large and small ceramic samples were treated according to the manufacturer's instructions of the IPS e.max ceramic and the luting cement used, as following:

Group A: Luting with GIC

The bonding surfaces of the discs were etched with 5% hydrofluoric acid gel (IPS ceramic etching gel, Ivoclar Vivadent) for 20 seconds and then thoroughly washed by water and air dried prior to cementation.

Group B: Luting with Variolink II

The bonding surfaces of the discs were etched with 5% hydrofluoric acid gel (IPS ceramic etching gel, Ivoclar Vivadent) for 20sec and thoroughly washed by water and air dried. A saline containing primer (Monobond N, Ivoclar Vivadent) was applied and allowed to react for 1minute and then sprayed by a gentle steam of air.

Group C: Luting with RelyX Ultimate

The bonding surfaces of the discs were etched with 5% hydrofluoric acid gel (IPS ceramic etching gel, Ivoclar Vivadent) for 20sec and thoroughly washed by water and air dried. A saline containing adhesive (Single Bond Universal Adhesive, 3M ESPE) was applied and allowed to react for 20sec and sprayed by a gentle steam of air.

In order to standardize the cementation procedure, an adhesive tape with a hole of 5.5mm in diameter was applied onto the exposed surface of the large discs prior to cementation. This adhesive tape was used to restrict the area of bonding and help in removing the excess of cement (Dawood, 2014). The cements were dispensed according to the manufacturer's directions on paper pads and by using plastic mixing spatula they were mixed and applied to the exposed surface of the embedded ceramic disc (Patel *et al.*, 2011). The bonding side of the small ceramic disc from the same group was seated onto its respective area on the exposed surface of the large embedded disc, and a load of 750g was applied vertically on the small disc for 5 minutes with the aid of dental surveyor (Hummel and Kern, 2004). Excess cement was removed with an explorer tip for the glass ionomer cement and with a micro brush for the two resin cements before the light-curing. The resin cements were light cured from three different directions. According to the manufacturer instructions of each cement, the Variolink II (Ivoclar Vivadent) was cured for 40sec per side and the RelyX Ultimate (3M ESPE) was cured for 20sec per side. After cementation each group was divided into two subgroups depending on the aging and storage period. Eight specimens from each group were stored for 24 hours in a dark water bath containing distilled water at 37°C and then tested (Marocho *et al.*, 2012). The remaining eight specimens in each group were stored in the same water bath for 30 days and thermocycled for 500 cycles between 5°C and 55°C before testing (Mohammed-Salih, 2013). Thus the final sample grouping was as follow:

G A<sub>1</sub>: Luting with GIC + Storage for 24hrs.

G A<sub>2</sub>: Luting with GIC + Storage for 1mon and thermocycling.

G B<sub>1</sub>: Luting with Variolink II + Storage for 24hrs.

G B<sub>2</sub>: Luting with Variolink II + Storage for 1 month and thermocycling.

G C<sub>1</sub>: Luting with RelyX Ultimate + Storage for 24hrs.

G C<sub>2</sub>: Luting with RelyX Ultimate + Storage for 1mon. and thermocycling.

During shear test, the bonded samples were attached to a universal testing machine and subjected to a shear load using a stainless steel

notched chisel at a crosshead speed of 1mm/min until failure occurred (Hara *et al.*, 2001). The load that caused failure was recorded for each specimen and shear bond strength was calculated by dividing the force at which the bond failure occurred by the specimen bonding area and expressed in MPa according to the following equation (Usman and Nisha, 2014): Shear bond strength (MPa) = Maximum force (N) / bonding area (mm<sup>2</sup>).

After shear testing, the debonded surfaces were examined under a stereomicroscope at 20 X magnification to determine the mode of bond failure. The failure modes were classified as follow (Nagayassu *et al.*, 2006; Dawood, 2014):

1- Adhesive failure: when all or most cement dislocate from the ceramic; more than 75% of the ceramic surface was visible.

2-Cohesive failure of the cement: When there was fracture in the cement layer with more than 75% of the ceramic surface covered with cement.

3- Cohesive failure of the ceramic: fractured ceramic adhered to the cement.

4- Mixed Failure: When there was combination of adhesive and cohesive fractures.

One way analysis of variance (ANOVA), least significant difference (LSD) and student's t-test were used for analysis of the data and (P ≤ 0.05) was considered the level of statistical significance.

### Results and Discussion

The results of this study showed that for the non-aged groups, the highest mean of shear bond strength was 23.549MPa and seen in group C<sub>1</sub>, followed by group B<sub>1</sub> with mean bond strength of 19.396MPa, while the mean shear bond strength recorded for group A<sub>1</sub> was much lower than in the other two groups and it was 4.745MPa. After aging, the highest mean shear bond strength value was recorded for group B<sub>2</sub> (20.936MPa) followed by group C<sub>2</sub> with mean bond strength of 15.435MPa and the lowest mean of shear bond strength was recorded in group A<sub>2</sub> (0.308 MPa), (Table 1). One way ANOVA and LSD tests showed that there was statistically significant difference in the shear bond strength values recorded for different types of luting cements before and after aging, (Table 2). To examine the effect of aging on the shear bond strength values in each luting a material, student's t-test was applied (Table 3). The results of this test showed that the aging significantly decreased the bond strength of GIC and RelyX Ultimate, however the bond strength values of variolink II was not significantly changed before and after aging. The mode of bond failure after shear bond test was observed under stereomicroscope and the results are summarized in (Table 4). This table showed that

the adhesive mode of failure was predominant in groups (A<sub>1</sub> and A<sub>2</sub>) which were luted with glass ionomer cement, however in the other groups which were luted with resin based cements, the cohesive failure in the cement was the predominant failure mode. The other types of bond failure occur less frequently.

Table (1): Descriptive statistics

Sub-groups	N	Mean	S.D.	Min.	Max.
A <sub>1</sub>	8	4.745	0.873	3.71	5.8
B <sub>1</sub>	8	19.396	2.125	16.84	23.16
C <sub>1</sub>	8	23.549	2.613	18.61	27.37
A <sub>2</sub>	8	0.308	0.095	0.2	0.43
B <sub>2</sub>	8	20.936	2.521	17.85	24.34
C <sub>2</sub>	8	15.435	2.104	12.45	18.53

Table (2): LSD after ANOVA

Subgroups	Mean Difference	S.E.	p-value
A1 B1	-14.651	1.004	0.000 (HS)
A1 C1	-18.804	1.004	0.000 (HS)
B1 C1	-4.153	1.004	0.000 (HS)
A2 B2	-20.629	0.948	0.000 (HS)
A2 C2	-15.128	0.948	0.000 (HS)
B2 C2	5.501	0.948	0.000 (HS)

Table (3): Student's t-test for the effect of aging on the bond strength

Subgroups	Subgroups' difference (d.f.= 14)		
	Mean difference	t-test	p-value
A1	4.438	14.292	0.000 (HS)
A2	-1.541	-1.322	0.207 (NS)
B1	8.114	6.840	0.000 (HS)
B2			
C1			
C2			

Table (4): Modes of bond failure

Subgroups	Adhesive failure (no.)	Cohesive cement (no.)	Cohesive ceramic (no.)	Mixed failure (no.)
A1	5	----	----	3
A2	8	----	----	----
B1	----	7	1	----
B2	----	5	3	----
C1	----	7	----	1
C2	1	6	----	1

In the luting process of an indirect glass-ceramic restoration two major interfaces are involved:

tooth/luting cement and luting cement/ceramic material. So it seems clinically relevant to achieve an optimal bonding performance in both interfaces. The present investigation aimed to study the ceramic/cement interface for evaluating the single effect of this interface on the bond strength, and so ceramic to ceramic bonded specimens were used. This specimen's assembly is able to eliminate other factors such as cement to dentin bond strength, which may affect the recorded ceramic to cement bond strength (Patel *et al.*, 2011; Murrillo and De Goes, 2014). According to several previous studies, a standard load of 750 g was applied onto ceramic specimens during the cementation procedure to simulate the finger pressure applied clinically during cementation of an all-ceramic restoration (Patel *et al.*, 2011; Marocho *et al.*, 2013; Passos *et al.*, 2013). Storing the bonded specimens in water at 37°C, 60°C, and 100°C for different time periods and thermocycling are methods used to investigate the durability of the bond strength by several previous studies (Wegner *et al.*, 2002; Hooshmand *et al.*, 2004; Salvio *et al.*, 2007; Marocho *et al.*, 2012). These methods are used to simulate the aging process that affect the resin bond to ceramic. Differences in the coefficient of thermal expansion of resin cements are promoted by thermocycling based on their components (matrix and fillers); by these differences the internal stresses increased and the bonded interface subjected to hydro-thermal degradation (Marocho *et al.*, 2012). There is a large variation in the number of cycles and in the temperature extremes between studies. This large variation led the International Organization for Standardization (ISO) to make standard protocol for thermocycling tests to enable investigators and industry to interpret and compare results. According to this protocol a thermocycling regimen comprising 500 cycles in water between 5 and 55°C is an appropriate artificial ageing test and thus our study was carried out following the ISO standard (ISO/TR 11405: 1994) (Mohammed-Salih, 2013). The results of this study showed that the values of shear bond strength vary with different resin cements. This result is in accordance with (Braga *et al.*, 1999; Kumbuloglu *et al.*, 2005; Altintas *et al.*, 2008; Marocho *et al.*, 2012), who concluded that the properties and bond strengths of resin cements may be influenced by their composition. The bond strength of GIC was much lower than the mean bond strength recorded for the other two resin cements; Variolink II and RelyX Ultimate. This can be explained by the lower mechanical properties of the GIC as compared to resin based cements and the absence of chemical bond to ceramic (Attar *et al.*, 2003). After aging the mean bond strength of

GIC was significantly lowered to 0.308 MPa. This result is comparable to that of (Kim *et al.*, 2011) who found that the bond strength values of GIC to zirconia was zero or near zero after thermocycling. This can be attributed to the increased acidity of GIC which make it susceptible to early water degradation, resulting in micro cracks which may initiate cracks and facilitate crack propagation in the cement (Conrad *et al.*, 2007). Before aging, the bond strength of RelyX Ultimate was higher than that of Variolink II, this may be attributed to the higher compressive strength (262 MPa) of this cement as compared to that of VariolinkII (240 MPa). Other possible explanations may be that although the filler content of these two cements are comparable, RelyX Ultimate is used with single bond universal adhesive which also contains fillers in its composition and may contribute to the higher bond strength of RelyX Ultimate. Furthermore, the organic matrix of VariolinkII contains the monomer urethane dimethacrylate (UDMA) which is added to provide better degree of conversion. However this monomer, as compared to bisphenol A glycol dimethacrylate (Bis-GMA), has weaker hydrogen bonding which (the hydrogen bonds) favorably affect the mechanical properties. This may contribute to lower bond strength of VariolinkII as compared to RelyX Ultimate which does not contain UDMA (Lemon *et al.*, 2007). After aging the bond strength of Variolink II was not significantly changed however, the bond strength of RelyX Ultimate was significantly lowered to 15.435 MPa. These results may mean that the RelyX Ultimate resin cement undergone more water dissolution and water sorption as compared to the Variolink II. This can be explained with regard to the degree of monomer conversion (DC), since it has been reported that Variolink II has a better DC than RelyX Ultimate (Sulaiman *et al.*, 2015). This difference in DC between the two resin cement may be due to the chemical component of one brand is more efficient in polymerizing than the other, or may be due to the presence of UDMA monomer in variolink II (Lemon *et al.*, 2007; Sulaiman *et al.*, 2015). Materials with lower DC undergo consequent leaching of unreacted monomer and more hydrolysis when exposed to oral fluids or water storage. Furthermore the filler bonding agents can also degrade. All these factors result in material degradation with significant decrease in mechanical properties (Gajewski *et al.*, 2012). Furthermore the presence of HEMA (hydroxyethyl methacrylate), which has a hydrophilic chemical nature, in single bond universal adhesive which is used with RelyX Ultimate may result in high water sorption of RelyX Ultimate and contribute to its lower bond strength

after aging. Water absorbed act as plasticizer within the polymer matrix and lead to degradation of filler/matrix interface and thus resulting in deterioration of mechanical/physical properties of the luting material (Meşe *et al.*, 2008).

In studying the quality of bond one should not depend on the bond strength data alone, the modes of failure should also be considered. The failure mode analysis showed that in the groups luted with Variolink II and RelyX Ultimate the predominant mode of failure was cohesive failure in the cement, however the adhesive failure mode was predominant in GIC luted specimens. These results are in accordance with those of (Zareen *et al.*, 2013; Usman and Nisha, 2014). The results of failure mode analysis support the shear bond strength data and confirm the poor bonding quality of GIC to glass ceramic as compared to the resin based cements.

### Conclusions

Within the limitations of this study, the followings can be concluded:

1. Luting agents of different types and composition yield different bond strength to glass ceramics with highest bond strength obtained by using resin based cements.
2. The aging process differently affects the recorded shear bond strength values for the three different luting cements.
3. The adhesive resin cement (Variolink II, Ivoclar Vivadent) appear to be not affected by aging and yield durable bond to lithium disilicate ceramic, so we can recommend the use of this cement with restorations made from this type of ceramic.

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