

Shear Bond Strength of Composite Resin Cements to Ceramics

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Abstract

The study aimed to evaluate the comparison of the shear bond strength of IPS Empress II and recent IPS e.max ceramics luted with eight different luting resins tested with three adhesion types: total etch, self-etch or self-adhesion. Two cylindrical shaped (7.2 mm×4.1 mm) ceramic specimens (IPS Empress II, IPS e.max) were used for each test group yielding a total number of 160 specimens. The specimens in each group which were randomly divided into 8 groups (n:10) were luted with eighth different resin composite luting cements (Variolink with Heliobond adhesive system, Bifix QM with Solobond Plus adhesive system, Choice with One Step Plus adhesive system, Multilink with Primer A+B adhesive system, Bifix QM with Futurabond DC adhesive system, experimental self adhesive luting resin, G-cem self adhesive luting resin, BisCem self adhesive luting resin). In all specimens, HF (5%) and silane were applied. All specimens were stored in water for 24 h and then subjected to 10000 cycles of thermocycling (5 C° and 55 C°). Bond strength was measured by means of a shear test, using Zwick Z010¹ universal testing machine with 0.5 mm/min speed until failure. To determine the statistical significance of the differences between the mean shear bond strength values, Kruskal-Wallis, Dunn's multiple comparisons test and Mann-Whitney U tests were used. Shear bond strength of luting resins using total-etch system showed better mean values than the resin cements using self etch and self adhesive systems (total etch 22.40 ±9.95; self-etch 16.76±7.78; self-adhesive 8.05±3.04 for IPS Empress II) (total etch 20.44±5.48; self-etch 17.59±5.18; self-adhesive 8.41±3.27 for IPS e.max). The shear bond strength values of self adhesive

system were significantly lower (P<0.05) than the other systems. No significant differences were observed between IPS Empress and IPS e.max ceramics according to shear bond strength. Adhesive failure was the most prevalent type of failure for both IPS Empress¹ and IPS e.max¹.

IPS Empress II¹ ceramics gave promising results, using with total-etch adhesive systems under the conditions of this *in vitro* study.

Keywords: IPS Empress II¹, IPS e.max¹, shear bond strength, luting resin, total etch, self etch, self adhesive.

Introduction

Conservation of tooth structure associated with aesthetic treatment techniques for posterior teeth led to increase in the placement of indirect restorations in recent years. Resin composites and ceramic materials have broadened the choices for aesthetic restorations (1). Because of their excellent biocompatibility, resistance to abrasive wear, color stability, high resistance to compress, ceramic materials are widely used for indirect restorations (2). But ceramics have some disadvantages like brittle fracture and low resistance to tensile (3). The long term success of ceramic restorations not only depends on the structure of material, but also depends mainly on the strength and durability of the bond of the luting composite to the tooth and the ceramic substrates (4).

Several dental ceramic systems were developed for inlay and onlay restorations during the last two decades. For this reason, more clinical investigations were published (5-7). One of these systems, IPS Empress II¹ multiphase glass-ceramic with a high degree of crystallinity is a heat-pressed, lithium disilicate-reinforced material (8). IPS e.max¹ is also heat-pressed, lithium disilicate-reinforced material but its flexion resistance is higher than IPS Empress II¹. In comparison with IPS Empress II¹, IPS e.max¹ is more economical and a fast produced restoration.

Resin-based adhesive luting materials are extensively used for cementation of indirect esthetic restorations. At the tooth surface, an adhesive system is also used to bond the luting agent to the tooth substrate. Currently, all adhesives are categorized as either etch-and-rinse or self-etch adhesives (9). A multi-step application technique is time consuming and technique sensitive, and

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Table 1. Ceramic materials and resin cement groups in the study.

| CERAMICS (n=160) | | Groups and Materials | | | | | | |
|--------------------------|-------------------|-----------------------------|--------------------|--------------------|------------------------------|-------------------------------|--------------------|--------------------|
| IPS Empress II (n=80) | Group 1 (n=10) | Group 2 (n=10) | Group 3 (n=10) | Group 4 (n=10) | Group 5 (n=10) | Group 6 (n=10) | Group 7 (n=10) | Group 8 (n=10) |
| | Variolink2 | Bifix QM (Solobond Plus) | Choice | Multilink | Bifix QM (Futura Bond DC) | Experimental Self Adhesive | G-Cem | BisCem |
| IPS e.max (n=80) | Group 9 (n=10) | Group 10 (n=10) | Group 11 (n=10) | Group 12 (n=10) | Group 13 (n=10) | Group 14 (n=10) | Group 15 (n=10) | Group 16 (n=10) |
| | Variolink2 | Bifix QM (Solobond Plus) | Choice | Multilink | Bifix QM (Futura Bond DC) | Experimental Self Adhesive | G-Cem | BisCem |

Table 2. Investment and dental ceramic firing procedures used in this study.

| Investment and dental ceramics | Burnout cycle | Starting temperature (°C) | Heating rate (°C/min) | Firing temperature (°C) | Holding time (min) | Vacuum temperature on-off (°C) |
|---|---------------|---------------------------|-----------------------|-------------------------|--------------------|--------------------------------|
| IPS Empress II Special Investing material (burnout) | First | 21 | 5 | 250 | 30 | |
| IPS Empress II (pressing) | Second | 250 | 5 | 850 | 60 | 500-920 |
| | | 700 | 60 | 920 | 20 | |

consequently may compromise bonding effectiveness. It is still not fully understood which adhesive system is most reliable to bond ceramic inlays to enamel and dentine (10). But total-etch adhesive is accepted as gold standart for luting the ceramic onlay/inlay restoration (5).

Although searchers have used a variety of bond strength methods, shear bond strength test has become an accepted evaluation method. Shear stresses are believed to be major stresses involved in *in vivo* bonding failures of restorative materials (11).

In this study, both glass lithium ceramics were investigated to obtain best bonding from three different bonding systems. Therefore, the aim of the present *in vitro* study was to evaluate the shear bond strength of IPS Empress II[®] and IPS e.max[®] ceramics luted with eighth different luting resins onto dentinal surfaces.

Materials and Methods

Experimental design was planned as *in vitro* on human molar teeth, eight luting resins and three different adhesive systems were grouped and shear bond strength among the groups were compared. The approval from the ethical committee of The Marmara University Institute of Health Sciences Clinical Research Preliminary Evaluation Board was obtained (MAR-YÇ-2006-0131). This research was ethically conducted in accordance with the Declaration of Helsinki (World Medical Association).

Tooth Preparation

One hundred- sixty recently extracted human, caries- free, third molar teeth at the clinics of the Oral Surgery Department of Marmara University, Faculty of Dentistry were selected and stored in a solution of distilled water for clean environment. The teeth

were debrided of residual plaque and calculus. Silicone (Zetaplus, Zhermack, Italy) molds in 2 diameter were prepared to provide standart samples sizes on the press. Then, the teeth which cervical region were 2 mm above silicone mold, were embedded in autopolymerizing acrylic resin (Panacryl, Arma, İstanbul, Türkiye).

All teeth occlusal surfaces remaining 2 mm above CEJ (cementoenamel junction), were abraded with no.12 fissure diamond burs (Komet, Lemgo, Germany) that were changed after each five teeth under profuse water cooling. To obtain homogeneous surfaces, cut surfaces were ground with 400 and 600 grit silicon carbide abrasive paper (3M, USA) Prepared teeth were randomly distributed into sixteen groups, ten teeth were (numbered and grouped among them) selected for each group (Table 1).

Two ceramics; IPS Empress II[®] and IPS e.max[®] were divided for the three adhesive system; total etch, self etch, self adhesive followed with eight resin cement systems; Vaiolink II, Bifix QM, Choice, Multilink, Experimental self adhesive cement, G-Cem, Bis Cem.

Ceramic Specimen Preparation

Eighty IPS Empress II[®] and eighty IPS e.max[®] press cylinder-shaped specimens were prepared in laboratory Marmara University Faculty of Dentistry Laboratory by a calibrated dental technician. Specimens made of lithium disilicate ceramic were invested, heated and pressed according to the manufacturer's instructions. The firing procedures for the ceramic and investment were described in Table 2.

Investment cylinders were bench cooled and divested by airborne-particle abrasion with 100 µm aluminum-oxide at 1 bar pressure. All ceramic specimens had a diameter of 4.1 mm and highness of 7.2 mm.

Table 3. Luting resins, its bonding agents and its contents used in this study.

| Adhesion | Company | Cure | Acid | Bonding systems and contents | | Luting Resin and contents | |
|----------------|--|-----------|---------------------------------|------------------------------|--|--------------------------------|---|
| Total-etch | Variolink II (Ivoclar Vivadent AG, Schaan, Liechtenstein) | Dual-cure | %37 phosphoric acid (Unietch) | Syntac primer | Maleic acid Polyethilen glycol dimetacrylate Diluted ketone | Variolink II base and catalyst | Üretandimetacrylate Triethilenglycol dimetacrylate Silanized barium glass filler Ytterbiumtrifloride (YbF3) Oxides Ba-Al-Flor-Silicate glass Catalyst Stabilizers Colouring agent |
| | | | | Syntac adhesiand | Polyethilen glycol dimetacrylate Diluted glutaraldehyde | | |
| | | | | Heliobond | %60 Bis-GMA %40 Trietihilen glycol dimetacrylate | | |
| Total-etch | Bifix QM (VOCO GmbH, Cuxhaven, Germany) | Dual-cure | %34.5 phosphoric acid (Vococid) | Solobond Plus primer | Maleic acid HEMA Polyfunctional monomers Sodium florid water Acetone | Bifix QM base and catalyst | Bis-GMA Benzoilperoxide Amines Baryum-aluminyum-borosilicate glass |
| | | | | Solobond Plus adhesiand | Polyfunctional monomers Initiators Hydrophilic metacrylates (HEMA) Acetone | | |
| | | | | One Step PLUS | Bisphenil dimetacrylate (%15-40) HEMA (%15-40) Acetone (%40-70) Glass (%1-10) | | |
| Total-etch | Choice (BISCO, Inc. Schaumburg, USA) | Dual-cure | %32 phosphoric acid (Uni-etch) | Porcelen Primer | Ethanol (%30-70) Acetone (%30-70) Silan (%1-20) | Choice liquid catalyst | Glass particules (%40-70) Amorf silica particules (%10-40) Bis-GMA (%5-30) Bis-GMA (%30-60) Benzoil peroxide (%1-5) |
| | | | | | | | |
| | | | | | | | |
| Self-etch | Multilink (Ivoclar Vivadent AG, Schaan, Liechtenstein) | Self-cure | None | Multilink Primer A | Reaction initiator | Multilink base and catalyst | Ethoxilated Bis-EMA UDMA Bis-GMA HEMA Barium glass Ytterbium trifloride Oxides |
| | | | | Multilink Primer B | HEMA Phosphoric acid monomer Acrylic acid monomer | | |
| Self-etch | Bifix QM+Futura Bond (VOCO GmbH, Cuxhaven, Germany) | Dual-cure | None | Liquid A | Metacryl Phosphoric acid ester Carbonic acid modified metacrylic ester | Bifix QM base and catalyst | Bis-GMA Benzoilperoxide Amines Barium-aluminyum-boro-silicate glass |
| | | | | Liquid B | Water Ethanol Silica | | |
| Self-adhesiand | Experimental Self Adhesiv (VOCO GmbH, Cuxhaven, Germany) | Self-cure | None | | None | | Unknown |
| | G-Cem (GC Corporation, Tokyo, Japan) | Self-cure | None | | None | G-Cem base and catalyst | UDMA Phosphoric acid ester monomer 4-META Water Dimetacrylates Silica powder Iniator Stabilizer Floro-alumina silicate glass |
| | Bis-Cem (BISCO, Inc. Schaumburg, USA) | Self-cure | None | | None | Bis-Cem base and catalyst | Bis-HEMA phosphate (%10-30) Tetraethilene glycol dimetacrylate (%10-30) Glass particules (%40-70) |

Cementation Procedure

IPS Empress II[®] and IPS press e.max[®] ceramic cylinders (4.1 mm in diameter, 7.2 mm in high) were bonded with eight different resin cements on the exposed dentin surfaces. All ceramic cylinders were etched with 5% HF acid (IPS Ceramic Etching Gel, Ivoclar, Schaan, Liechtenstein) for 20 seconds, then rinsed thoroughly for 20 seconds and dried for 20 seconds. Ceramic specimens were luted on dentin surfaces with the different three luting systems (total-etch, self-etch, self-adhesive), in accordance with the manufacturers' instructions. During cementation period, static load (5 kg) is applied to the specimens. Light polymerization was applied for 40 second (Bleuphase C5, Ivoclar Vivadent AG, Schaan, Liechtenstein). Luting resins, its bonding agents and its contents used in this study listed in Table 3.

Thermocycling

After bonding procedure, all specimens were thermocycled in water for 10000 cycles between 5 and 55 °C, with dwell times of 30 seconds in each bath and a transfer time of 3 seconds between baths. 10000 cycles were preferred since it corresponds to one year clinical evaluation (12). The thermocycling was built up by Engineer Yalçın Hocaoglu and dentist Gürol Ozyoney.

Shear Bond Test

Shear loading was 0.5 mm/min. (Zwick Z010, Zwick GmbH, Ulm, Germany). The samples were positioned in the sample holder with the dentin surface parallel to loading piston of testing machine. Maximum shear load at the point of failure was recorded and shear bond strengths were calculated by the computer.

Stereomicroscopic Analysis

Fractured specimens were examined with a stereomicroscope at ×80 magnification to determine the mode of failure. Failure modes were evaluated according to the classification if adhesive, mixed or cohesive failure occurred.

Statistical Analysis

SPSS statistical programme (Statistical Package for Social Sciences version 10.0, SPSS Inc., Chicago, Illinois, USA) was used for the analysis. Bond strength data were analyzed with Kruskal Wallis, Dunn's multiple comparisons test and Mann-Whitney U test. Mann-Whitney U test was used for the analysis of double groups, Kruskal Wallis was used for multiple comparisons and Dunn's multiple comparisons test was used for the comparison of subgroup.

Results

The mean of groups and standart deviation shear bond strength values of this study whose aim is the evaluation of eight different resin cements three adhesive systems were demonstrated in Table 4. In the event of spontaneous debonding during thermal cycling, specimens were excluded from this study. When determining the mean of groups and standart deviation, debonded specimens were not calculated. When examining the results of shear bond test, IPS Empress II[®] luted with Bifix QM dual-cured luting resin used with Solobond Plus total-etch adhesive system showed the highest shear bond strength value (41.53 MPa) whereas the lowest value (1.30

Table 4. The mean shear strength values of the groups and standart deviation (MPa).

| Material | Adhesive System | Group | Mean | Standart Deviation (SD) |
|----------------|-----------------|----------|-------|-------------------------|
| IPS Empress II | Total-etch | Group 1 | 17.88 | ±3,48 |
| | | Group 2 | 22.40 | ±9,95 |
| | | Group 3 | 13.86 | ±3,28 |
| | | Group 4 | 16.76 | ±7,78 |
| | Self-etch | Group 5 | 15.85 | ±6,58 |
| | | Group 6 | 8.05 | ±3,04 |
| | Self-adhesive | Group 7 | 6.38 | ±3,27 |
| | | Group 8 | 5.30 | ±2,25 |
| IPS e.max | Total-etch | Group 9 | 19.55 | ±8,67 |
| | | Group 10 | 20.44 | ±5,48 |
| | | Group 11 | 11.08 | ±3,64 |
| | | Group 12 | 15.10 | ±4,22 |
| | Self-etch | Group 13 | 17.59 | ±5,18 |
| | | Group 14 | 8.41 | ±3,27 |
| | Self-adhesive | Group 15 | 8.02 | ±2,60 |
| | | Group 16 | 6.13 | ±3,14 |

MPa) was obtained in the group of IPS e.max[®] luted with Bis-Cem self adhesive luting resin.

In total-etch adhesive systems, both ceramics did not show any significant differences among the groups (Variolik II, P= 0.998; Bifix QM plus Solobond, P= 0.821; Choice, P= 0.13). However, the only differences were observed between the groups of Bifix QM plus Solobond and Choice both ceramics (P< 0.05; P< 0.001). Variolik II and Choice were also significantly different only in e.max groups (P< 0.001).

Self-etch adhesive systems were not statistically different among groups for both ceramics (Multilink, P= 0.705; Bifix QM+Futura Bond DC P= 0.496). Bifix QM+Futura Bond DC e.max group was the highest shear strength value obtained in self-etch systems (17,59± 5.18 MPa).

Self-adhesive systems were not statistically significant among the groups for both ceramics (Experimental adhesive resin, P= 0.895; G-Cem, P= 0.082; Bis-cem, P= 0.501). Experimental adhesive resin was found with the highest shear bond strength values both ceramics (8.05±3.04, Empress II; 8.41±3.27, e.max)

Failure Pattern Analysis

Following the shear bond strength test, the failure mode was recorded by examining all teeth with the stereomicroscope (Leica Microsystems GmbH, Germany). Failure modes were classified as;

Type I: Adhesive failure

Type II: Cohesive failure

Type III: Mixed failure

In this study, type I, adhesive failure (65%) was the most prevalent type of failure for both IPS Empress II[®] and IPS e.max[®]. In IPS

Empress II' groups (n=80), type I failure was observed in 50 specimens while in IPS e.max groups (n=80), this type of failure was seen in 54 specimens. Mixed failure (51.67%) was the most prevalent type of failure for specimens luted with total-etch adhesive systems where as adhesive failure (75% and 83.33%) was the most prevalent type of failure for specimens luted with self-etch and self-adhesive systems.

Discussion

In vivo studies are the most suitable methods to evaluate dental materials. However they are considerably time-consuming, high-cost and difficult to apply for both patient and dentist (13, 14). Thus, *in vitro* studies are preferred to evaluate the dental materials. Pressable ceramic is one of the most popular dental materials due to its excellent mechanical properties (14). IPS Empress II is also a pressable ceramic used clinically. IPS e.max put on the market so as to increase the fracture resistance of IPS Empress. In this study, both IPS Empress II and IPS e.max were evaluated at *in vitro* condition. There are a lot of laboratory test methods used for bond strength measurement of dental materials to dentin structure. Preferred bond strength tests are three-point bending test, tensile and micro-tensile test and the shear and micro-shear test. *Oilo* (1993) discussed the accuracy and clinical relevance of the different testing methods and showed that the shear bond strength is the most common testing method (15). In this study, two ceramic materials' bond strength was measured by shear test method. For determination of bond strength values, the manufacturers' recommendations should be followed for cementation period. After cementation period, it is important that static load applied to specimens until resin cement hardened (16). In this study, in light of the foregoing 5 kg was applied during hardening period. A strong resin bond relies on micromechanical and chemical bonding to ceramic surface, which requires roughening and cleaning for adequate surface activation (17, 18).

Dual-cured resin cements were preferred for luting ceramic inlay and onlay restorations due to offering extended working times and controlled polymerisation (19). Autopolymerizing resin cements had fixed setting times and generally indicated for resin-bonding metal-based or opaque, high strength ceramic restorations (19). *Matsumura et al.* (1997) found that bond strength values of autopolymerizing resin cements were lower than dual-cured resin cements' (20). *Toman et al.* (2008) found that the etch-and-rinse dentin bonding system produced higher bond strengths of IPS e. max Press to dentin surfaces than the self-etching bonding systems and self-adhesive luting system (21).

Manso et al. (2011) found that dual-curing etc-and-rinse or self-etching self-adhesive resin luting cements achieved greater bond strength when light curing was applied. The weakest adhesion was obtained with glass-ionomer luting agent (22).

In this study, IPS Empress' and IPS e.max' were luted with both dual-cured and self-cured resin cements in order to make a comparison. It was observed in this study that both ceramics were equal on shear bond strength values among all luting resin groups. Although dual-cured resin cements were more successful in cementation of ceramic restoration, in this study there was no significant difference between Bifix QM+ Futura Bond dual-cured

luting resin group applied with self-etch systems and Multilink self-cured luting resin group. Long-term clinical success of indirect aesthetic restoration depends on good marginal adaptation and adhesion. Recently ceramic inlay and onlay gained popularity because luting resins began to be used with dentin bonding agent so bond strength of luting resins to ceramic was increased (23). Dentin bonding agents provide hermetic seal between resin composite and dentin, prevent post-operative sensitivity, increase adhesion and serve as a stress-breaker liner (24). *Sorensen and Munksgaard* (1996) compared whether or not applying dentin bonding agent before ceramic inlays luted with dual-cured resin cement. When applying dentin bonding agent, gap related to polymerisation shrinkage decreased about 46-93% (25). According to *De Munck et al.* (2005), all-in-one bonding systems which were fast and simple applying technique didn't reach bonding level such as total-etch (26). In total-etch system, smear layer eliminated because of washing up after acid-applying but in self-etch system hydrolytic stability was questionable because of the rest of acidic monomer. *Al-Ehaideb et al.* (2000) studied about two and three-step total etch systems and reported there was no difference between these two systems in terms of shear bond strength. In all groups, there was adhesive failure about 80% (27). *Yin et al.* (2009) demonstrated that the resin cement based on etch-rinsing bonding system has higher bond strength to dentin than those based on self-etch bonding system and self-adhesive resin cements (28). *Zhang and Degrange* (2010) found that multiple-step dual-cured luting resins perform better than single-step auto-adhesive resin cements (29). Our study showed that adhesive systems affected the shear bond strength of luting resins. Luting resins used with total-etch systems had higher bond strength values than self-etch and self adhesive systems. The resin cement 'Bifix QM' used with total-etch adhesive system 'Solobond' demonstrated the highest bond strength values whereas self-adhesive resin cement 'Bis-Cem' demonstrated the lowest one (41,53 MPa). In this study, when luting resins applying with total-etch adhesives exposed to shear test, mixed-type failure were seen about 51.67%. In self-etch and self-adhesive failure type was adhesive-type failure. It depended on deficient demineralisation of smear layer and deficient thickness of hybrid layer.

As a clinical relevance we may say that glass lithium ceramics may be preferred to lute with a total-etch adhesive system in order to obtain the best bond strength from dental tissue.

In conclusion of this *in vitro* study, shear bond strength of resin cements using total etch system had better mean values than the luting resins using self etch and self adhesive systems. The shear bond strength values of self adhesive system were significantly lower ($P<0.05$) than the other systems.

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