# Microtensile Bond Strength of Three Restorative Core Materials with IPS E.max Press Ceramic by Two Resin Cements

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**Abstract**
***Objectives***: The aim of this study was to compare the microtensile bond strengths (µ TBS) of three core materials with one lithium disilicate reinforced ceramic using two resin cements.
***Methods:*** Three core materials (Nulite F® (Biodental Technologies), Filtek Z250® (3M-ESPE), Prettau-Anterior® (Zirkonzhan, Germany)) were prepared as blocks (10×10×4 mm3) according to the manufacturer’s instructions. Lithium disilicate ceramic blocks were also constructed and bonded to core specimens with two dual curing luting resin cements (Duo-Link® (Schaumburg, IL), Bifix QM® (VOCO, Cuxhaven, Germany)). Micro-bar specimens were prepared and loaded in tension to determine the µ TBS Failure modes were classified by scanning electron microscope (SEM). Data were analysed using two-way ANOVA and Tukey HSD test.
***Results:*** The µ TBS varied significantly depending on the core materials and resin cements used ($P< 0.05$). The µ TBS of Bifix QM was significantly higher than of Duo-Link in all core materials. The µ TBS of zirconia core was significantly higher than of both composite cores with both resin cements. There were no statistically significant differences among Nulite F and Filtek Z250 ($P> 0.05$). The highest bond strength was obtained between zirconia core and Bifix QM (45.3 ± 6.7 MPa).
***Conclusion:*** *In vitro* µ TBS of glass ceramic blocks bonded to zirconia core material showed higher bond strength values than resin-based core material, regardless of the resin cement type used.
***Key words***: Materials, Glass ceramics, Zirconia

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## Introduction

The esthetic properties of ceramic crowns are preferable in the anterior region for prosthetic treatment. Glass based all-ceramic crowns are more translucent than their alumina or zirconia counterparts and it has been shown that they need up to 2 mm of porcelain to block out dark underlying colour (1,2). For this reason, glass based, all-ceramic systems should not be used on dark underlying surfaces .The seating surface or core materials are conventionally made from metals. Cast metal post and core foundations have a long history of successful use due to their superior physical properties (3). However, esthetic properties of these materials are limited since the grey-color is apparent when used to support translucent all-ceramic restorations. This has led to the use of tooth-colored materials, including resin composites, ceramics, glass ionomer cements and compomers as esthetic alternatives. An important factor for the clinical success of ceramic restorations is the bond strength of the luting cement to the seating surface (4).

Resin composite cores are tooth-colored, and can be bonded to tooth using dentin adhesives. As they set quickly, core and tooth preparation can be completed without delay (5), however, the bond strength between aged composite cores and resin cements is still a challenge (6, 7). A variety of techniques have been proposed to improve the bond strength of aged composite core to resin cements. It has been shown in previous studies, that surface roughening with air-borne particle abrasion is effective in increasing the bond strength of composite resin material to resin cements (7-9).

Despite the advantages of composite cores, one study showed that pre-fabricated posts with direct cores made of glass ionomer, composite resin, or amalgam are less reliable than a one-piece post & core, primarily because of delamination at the interface between the post and the core (10). In addition, composite materials lack the strength to resist deformation when used to support crowns in severely coronal destructed teeth (11).

Hence, zirconia as a one piece post and core has been introduced to providegre at toughness, biocompatibility, maximal adaptability to canal, stability in shape, as well as adequate esthetic (12, 13). The major limitation regarding the use of zirconia is the difficulty to adhere to this material (14).

It seems that both most convenient and esthetic options as core materials have the same problem with bonding to resin cements.

Bozogullari *et al*. showed that the bond strength of composite resin core materials are higher than ceramic-based core materials, using one resin cements (5). Many studies concluded that the bond strength between ceramic restoration and core material may be one aspect for the selection of a core build up material (5, 15, and16). But to the knowledge of authors to date there was on study conducted to compare zirconia with composite resin, as a core material.

As stated, success with resin bonded all ceramic restorations are highly dependent on obtaining a reliable bond, which has to integrate all parts of the system, including core material, luting cement and ceramic, into one coherent structure (17). Thus the aim of this study was to evaluate the µTBS of three different core materials to a lithium disilicate reinforced ceramic, using two luting agent. The hypotheses tested were: (1) The bond strengths of resin-based core materials are higher than zirconia core material, and (2) The µTBS values do not depend on the luting agent used.

## Methods

*2-1.Preparation of ceramic blocks*

Information of the manufacturers, compositions, and setting reactions of materials used in this study are provided in Table 1.

Twenty four $10\times10\times4mm^3$ Lithia disilicate-based hot-pressed ceramic (IPS e.max Press®, Ivoclar Vivadent, Schaan, Liechtenstein) blocks were invested, heated and pressed according to the manufacturers' instructions.

Bonding surfaces of the ceramic specimens were treated using air-borne particle abrasion with 100µm grain-sized aluminum oxide particles. Subsequently, the ceramic surfaces were rinsed, air-dried, followed by etching with 10% hydrofluoric acid (Porcelain Etchant®; Bisco, Schaumburg, IL) for 2 minutes. Finally the specimens were cleaned in an ultrasonic bath containing distilled water for 3 minutes, to remove residual acid. Coating of the surfaces was performed using a silane coupling agent (Bis-Silane®; Bisco, Schaumburg, IL) for 60 seconds before drying with air again.

*2-2.Preparation of core material blocks*

Core materials used in this study included resin-based composite (Nulite F, and Filtek Z250), and a zirconium core material (Zirkonzhan). Information of the manufacturers and compositions of these materials are provided in Table 1.

**Table 1- Materials used in this study**

| | Brand | Definition | Manufacturer | Composition |
|---|---|---|---|---|
| 1 | IPS e.max Press® | Lithium disilicate(pressable) all ceramic system | Ivoclar/Vivadent, Schaan, Liechtenstein | SiO2, LiO2, K2O, P2O5, ZrO2, ZnO Coloring oxides, other oxides |
| 2 | Filtek Z250® | Microhybrid light cure composite | 3M-ESPE, USA | UDMA, Bis-GMA, Bis-EMA, TEGDMA, Silicate glass, Zirconia glass |
| 3 | Nulite F® | Hybrid composite | Biodental Technologies, Australia | Bis-GMA, Micro-rod glass |
| 4 | Prettau-Anterior® | Yttrium stabilized tetragonal Zirconia polycrystalline ceramic | Zirkonzhan, Germany | Yttrium, Zirconia oxide |
| 5 | Duo-Link® | Dual-cure composite luting cement | BISCO, Schaumburg, IL | Bis-GMA,UDMA, TEGDMA, fine glass particles, Hydrofluoric acid |
| 6 | Bifix QM® | Dual-cure composite luting cement | VOCO, Cuxhaven, Germany | Bis-GMA, HEMA, Benzoyl peroxide, high Fluoride amin |
| 7 | Futurabond DC® | Dual cure self-etch adhesive | VOCO, Cuxhaven, Germany | Bis-GMA, Acidic monomer, 2 hydroxymethacrylate, H2O2, Ethanol |
| 8 | All-Bond 3® | Dual cure adhesive resin system | BISCO, Schaumburg, IL | Bottle 1: Ethanol, NTG-GMA, Bottle 2: Bis-GMA, HEMA, BPDM |
| 9 | Z-Prime plus® | Zirconia-Alumina-Metal primer | BISCO, Schaumburg, IL | Bis phenyl dimethacrylate, HEMA, Et hanol |
| 10 | Porcelain Bonding Resin® | HEMA free, light cure resin | BISCO, Schaumburg, IL | Bis-GMA, UDMA, Tri-EDMA |
| 11 | Etch-37® | Enamel and dentin etchant | BISCO, Schaumburg, IL | 37%H3PO4,Benzalkonium Chloride |
| 12 | BIS-SILANE® | Two component silane coupling agent | BISCO, Schaumburg, IL | Ethanol, Silane |
| 13 | Porcelain Etch® | Porcelain etchant | BISCO, Schaumburg, IL | 9.5% Hydrofluoric acid |

For resin-based core materials, specimens were prepared in a brass mold ($10\times10\times4mm^3$) composite resin was incrementally inserted (2mm), condensed into the mold, and light activated for 40 seconds (Deml®, Kerr,USA), until each mold was completely filled. All specimen surfaces were ground with 150-grit silicon carbide paper. Bonding surface of the composite resin specimens were sandblasted with 50µm aluminum oxide particles for 15 seconds using an intraoral air-abrasion device (Dento-prep®; Roving Dental Mfg. Dougard, Denmark) with the nozzle 10mm distant from the specimen surface. All specimens were rinsed and dried for 5 seconds using an air/water spray.

Eight zirconia blocks ($10\times10\times4mm^3$) were prepared using copy milling technique. Bonding surface of the specimens were sandblasted like composite resin blocks. All specimens were rinsed and dried for 5 seconds using an air/water spray.

Three different core materials were further divided into two subgroups with respect to the applied resin cement

(Duo-Link and Bifix QM). According to Bozorgullari *et al*. (5) and Coneppele *et al*. (6) the sample size was determined as 60; six groups and ten specimens in each group:

*Group 1*

Etched ceramic surfaces were coated with an adhesive layer (Porcelain Etchant) and kept under a black shield.

Duo-Link dual-cure composite luting cement system was used. Sandblasted zirconia specimens were cleaned using 32% phosphoric acid for 30 seconds. After water rinse and air drying, two layers of zirconia primer (Z-prime®, Bisco, Schaumburg, IL) was applied on the substrate surface and left for 5 seconds before drying with mild air-flow. Subsequently one layer of dual-cure adhesive resin (All-bond 3®, Bisco, Schaumburg, IL), was applied and air dried. Then Duo-Link paste was applied to the zirconia surface. The e.max Press and zirconia blocks were then joined and placed under a 750-gr static load, applied for 5.5 minutes according to manufacturers' recommendation time for chemical setting of resin cement. The excess cement was removed with a brush before light polymerizing with an LED light curing unit for five 40-second periods at right angles to each other.

*Group 2*

Etched ceramic surfaces were treated like first group. Bifix QM dual-cure composite luting cement system was used. Two layers of zirconia primer (Z-prime) were applied on the sandblasted zirconia specimens' surface and left for 5 seconds before drying with mild air-flow. Subsequently one layer of self-etch dual-cure adhesive resin (Futura bond DC®, Voco, Cuxhaven, Germany) was applied and air dried for 20 seconds.

Then Bifix QM pastes were applied to the zirconia surface. The e.max Press and zirconia blocks were joined and placed under a 750-gr static load, applied for 3 minutes according to manufacturers' recommendation time for chemical setting of resin cement. The excess cement was removed with a brush before light polymerizing.

*Groups 3 & 4*

Etched ceramic surfaces were treated like the first group. Duo-Link dual-cure composite luting cement system was used.

Sandblasted composite resin specimens (Nulite F and Filtek Z250) were cleaned using 32% phosphoric acid for 10 seconds. After water rinsing and air drying, one layer of dual-cure adhesive resin (All-bond 3), was applied and air dried.

Duo-Link pastes were applied to the composite surface. The e.max Press and composite resin blocks were then joined and treated like the first group.

*Groups 5&6*

Etched ceramic surfaces were treated like the first group. Bifix QM dual-cure composite luting cement system was used. One layer of self-etch dual cure adhesive resin (Futura bond DC) was applied on the sandblasted composite resin (Nulite F and Filtek Z250) specimens' surface and air dried for 20 seconds.

Bifix QM paste was applied to the composite surface. The e.max Press and composite resin blocks were then joined and were treated like the second group.

*2-3.Microtensile bond strength test*

Using a low-speed diamond cutting saw (Isomet®; Buehler, Lake Bluff, IL), the ceramic-cement-core material sets were cut into beam specimens with ≈1.00mm$^2$ cross section. The inner specimens from each experimental group were selected. The tensile bond strength test was evaluated using a microtensile tester machine (Bisco, Schaumburg, IL): specimens were fixed to the machine by cyanoacrylate adhesive (Mitreapel®, Beta Chemical Ind., Turkey), and stressed to failure in tension at a cross-head speed of 1 mm/min. After testing, specimens were removed from the testing device and the cross-sectioned area of the fracture sites were measured with a digital caliper (Mitutoyo corp., Tokyo, Japan), to calculate the ultimate tensile bond strength expressed in MPa.

*2-4.Fracture Analysis*

The fractured surfaces of all specimens were observed using an SEM (CamScan MV2300®; EOS, Ontario, Canada) at 80 X magnification to identify the mode (type) of failure and classified based on crack initiation and principles of fractography (18).

Mode 1: adhesive separation at the ceramic-adhesive resin interface.

Mode 2: failure starts at the ceramic-adhesive interface, progresses into the adhesive resin and returns to the interface.

Mode 3: failure originates from an internal flaw (penny-shape internal crack).

Mode 4: failure starts at the ceramic-adhesive interface and propagates through the adhesive resin.

Mode 5: failure starts at the ceramic–adhesive interface, propagates through the adhesive resin to reach the core material–adhesive interface.

Mode 6: cohesive failure through the core material.

*2-5.Statistical Analysis*

Statistical analyses were performed using SPSS version 18. The variables were statistically analyzed using two-way ANOVA. Post-hoc multiple comparisons were performed using the Tukey test, with the significance level set at $\alpha$=0.05.

## Results

*3-1.Micro tensile bond strength test*

μ TBS test results are shown in Table 2. μ TBS was significantly affected by the core material ($P$=0.000) and luting cement ($P$= 0.000). Zirconia based core material showed the highest bond strength while one of the composite based core materials (Nulite F) showed the lowest ($P$< 0.01). There were no statistically significant differences among Filtek Z250 and Nulite F ($P$ = 0.58), regardless of luting cement type.

Regarding the cement type, the results showed that all types of core specimens luted with self-etch bonding resin cement (Bifix QM) had significantly higher bond strength than etch and rinse resin cement (Duo-link) (Figure.1).

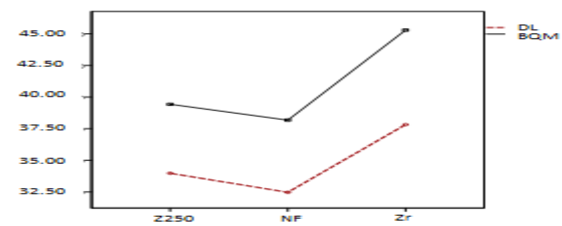



**Figure 1- Representing the µ TBS (Mpa) of the investigated substrates and luting cements. Regarding the cement type, BQM had significantly higher bond strength than Duo-Link. NF: Nulite F, ZR: Zirconia, DL: Duo-link, BQM: Bifix QM**

**Table 2- Mean Values and Standard Deviations ofmicro tensile bond strength in experimental Groups (MPa)**

| Group | Mean | Std. Deviation | Tukey grouping |
|---|---|---|---|
| Zr-DL | 37.8 | 5.9 | B |
| Zr-BQM | 45.3 | 6.7 | C |
| NF-DL | 32.4 | 5.4 | A |
| Z250-DL | 33.9 | 6.8 | A |
| NF-BQM | 38.1 | 7.5 | B |
| Z250-BQM | 39.4 | 4.7 | B |

NF: Nulite F, Zr: Zirconia, DL: Duo-link, BQM: Bifix QM

*3-2.Failure mode (type) analysis*

Results of the failure modes evaluations are given in Table 3.

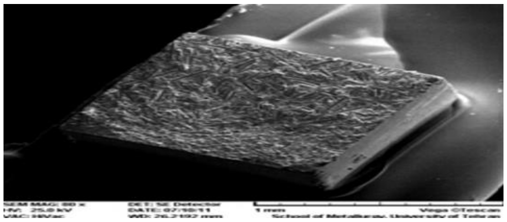
It could be seen that there were more cohesive fracture in NF composite material (Figure. 2A), while in zirconiagroups most specimens showed adhesive failure (Figure. 2B)

Ultradent porcelain repair kit yielded higher shear bond strength than Pulpdent. The LSD test showed that silanization significantly affected the bond strength compared to not applying silane ($p$<0.05, mean difference of 3.09). Also, the LSD test showed that use of Ultradentsilane significantly affected the shear bond strength ($p$<0.05, mean difference of 10.2). However, Pulpdentsilane had no significant effect on shear bond strength ($p$=0.89, mean difference of 0.8). Application of one and two layers of Ultradent (mean difference of 1.06) and Pulpdent (mean difference of 0.14) silanes did not cause a statistically significant difference in results ($p$=0.94 for Pulpdent and $p$=0.60 for Ultradent, Table 2 and Diagram 1).

**Table 3- Failure types in experimental Groups**

| Group | Type 1 | Type 2 | Type 3 | Type 4 | Type 5 | Type 6 | Total |
|---|---|---|---|---|---|---|---|
| Zr-DL | 4 | 4 | 0 | 0 | 12 | 0 | 20 |
| Zr-BQM | 1 | 0 | 4 | 8 | 5 | 0 | 18 |
| NF-DL | 0 | 2 | 0 | 3 | 4 | 11 | 20 |
| Z250-DL | 1 | 1 | 1 | 4 | 13 | 0 | 20 |
| NF-BQM | 0 | 0 | 0 | 3 | 16 | 1 | 20 |
| Z250-BQM | 1 | 3 | 0 | 0 | 16 | 0 | 20 |
| Total | 7 | 10 | 5 | 18 | 66 | 12 | 118 |

NF: Nulite F, Zr: Zirconia, DL: Duo-link, BQM: Bifix QM

A 

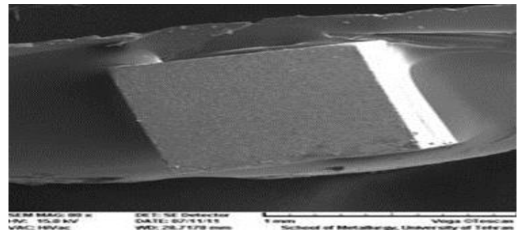
B 

**Figure 2- Examples of typical SEM micrographs depicting different failure modes(types) in test specimens:**
**A: cohesive failure in the NF composite (type 6)**
**B:adhesive failure (type 4)**

## Discussion

In the present study, µ TBS of three different core materials to ceramic blocks, using two different adhesive cement systems, was measured.

The results did not support the first research hypothesis, that bond strength of resin-based core materials is higher than zirconia-based core materials. Zirconia-based core materials showed the highest bond strength, regardless of the adhesive cement used.

Regarding the mechanical bond strength test, the present study used the non-trimming µTBS method. The micro tensile test, a tensile bond test with reduced testing area, was developed as an attempt to eliminate the non-uniform stress distribution at the adhesive interface and to minimize the influence of interfacial defects (19). The reduction in the number of defects in the adhesive zone is thought to decrease bulk cohesive failures and increase the tensile bond strength (20). The non-trimming (bar-shaped specimen) method was used to obtain specimens, as no specimen finishing is necessary and avoids areas of stress concentration (18). In addition, Valandro *et al*. demonstrated that specimens with $1\times1mm^2$ section area gives the best result according to pre-test failure and bond strength results (21). Hence we used bar-shaped specimens with $1\times1mm^2$ section area.

The current study has shown that the bond strength to ceramic is influenced by the core material. Bond strength to zirconia-core material was significantly higher than both composite core materials. This is in contrast with the results of Bozogullari *et al*. Study (5). Structural difference between zirconia ceramic used in this study and use of a new primer agent (Z-Prime plus), may be the reasons for this result.

Zirconium-oxide ceramic resists fracture loads and has optimal strength, but its use requires a reliable bond with the luting agent, which is the major limitation regarding the use of zirconia. The absence of a silica and glassy phase impairs the effectiveness of conventional adhesive cementation techniques including etching with hydrofluoric acid and silanization on zirconia-based ceramics (22). In the present study, air abrasion was used since it has been proven to increase the surface energy and wettability (23). Additionally, it has been shown that association of air-abrasion and primer/luting agents containing acidic monomers could be beneficial and give the best results (24, 25). The Z-Prime plus used in this study includes a mixture of organophosphate and carboxylic acid monomers. Organophosphate monomers are bi-functional molecules like silane which have an organo-functional part, most often a methacrylate group that can be co-polymerized with the monomers of composite resin system (14). The phosphate monomers also contain phosphoric acid groups that can develop bond with the metal oxides, such as zirconium oxide, in the substrate. The other monomers in Z-prime plus such as carboxylic acid monomer are cooperating in development of the bond. Magne *et al*. (14) showed that the use of this new zirconia primer (Z-Prime plus) increased the bond strength of different adhesive luting agents to zirconia which is in accordance with our study. There was no statistically difference between two types of resin-based core materials (Nulite F and Z250), regardless of luting cement type. These results are in accordance with those obtained by Bitter *et al.* (8) and Bozogullari *et al*. (5), who reported no difference between bond strength in different resin-based core materials.

Several dental materials have been proposed for core build-up procedures. The ease of use of direct materials is certainly dominating their selection. Improvement in composite technology made resin composites the material of choice in the restoration of non-vital and vital teeth. Beside the advantages of composite resins as a core material, it is known that the bonding between aged composite cores and new resin cement is difficult and need special management (6,9,26). This is in accordance with our findings which the bond strength to resin-based core materials are significantly lower than zirconia-based core material. Since bond strength was significantly affected by the luting cement the second hypothesis was also rejected.

Resin-based adhesive luting materials are extensively used for the cementation of inlays, onlays, crowns, veneers and posts. Currently, these cements are based on the use of etch and rinse or self-etching adhesives in conjunction with a low viscosity composite resin. The results showed that the resin composite core specimens luted with self-etch bonding resin cement (Bifix QM) had higher bond strength that etch and rinse resin cement (Duo-link). These results are in accordance with the data from literature (7, 9). The matrix structure of an intermediate agent between old and new composite may be important for bonding to aged composite and may affect bond strength (7). The polar nature of the phosphate group in self-etch adhesives may contribute to bonding with the inorganic filler component of ground composites (9).

Bonding to zirconia was also higher with the use of Bifix QM (BQM) resin cement. BQM is self-etch adhesive resin cement which has acidic monomers. These acidic monomers can react with the oxide group on zirconia ceramic surface similar to the surface reaction between silane coupling agents and silica based ceramics (14).

### *4-1.Mode (type) of failure*

Bond quality, however, should not be assessed on strength data alone, because the failure mode is also important. A careful interpretation of failure mode is required to prevent inappropriate conclusions about the utility of the micro-tensile test and the adhesion zone phenomena.

The microtensile test produce variable fracture surface morphology and fracture origins for the same adhesive interfaces within the adhesion zone, hence we used Della Bona classification for failure mode (18). Therefore optical microscopy observation was not enough to determine the mode of failure of bonding interface. Hence a thorough SEM examination of all fracture surfaces was done (Table 3). Analysis of the failure modes showed more mode 4, failure that start at the ceramic-adhesive interface and propagate the adhesive, and mode 5 which then reach the adhesive-composite interface in all groups. These findings are in accordance with achieved high bond strengths. The least mode of failure seen was mode 3 (cohesive fracture in resin cement). This type of failure is a result of cement weakness due to flaws and defects in the cement layer (25). Since we followed ISO 4049 instruction for cementation procedure, the flaws should be negligible.

Until now, the choice of core foundation materials and luting cements has largely left to practitioner`s preference. In view of the results, the bond strength between a core material and cement should be considered in the selection of material for restoring broken down and endodontically treated teeth.

## Conclusion

1-The highest μTBS was observed between e.max Press glass ceramic and zirconia core material with both types of resin cements ($p$<0.05).
2-The μTBS of two types of composite core materials with e.max Press glass ceramic had no significant difference.
3-Bifix QM resin cement had higher bond strength with all core materials than Duo-Link.

**Conflict of Interest:** "None Declared"

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