Effect of Different Surface Treatments on the Tensile Bond Strength to Lithium Disilicate Glass Ceramics.

J Adhes Dent. 2018 Jun 14;:1-8

Authors: Lyann SK, Takagaki T, Nikaido T, Uo M, Ikeda M, Sadr A, Tagami J

Abstract

PURPOSE: To evaluate the influence of different surface treatments of lithium disilicate glass ceramics on the bonding efficacy of three luting composites.

MATERIALS AND METHODS: A total of 450 blocks of e.max CAD (Ivoclar Vivadent) ground with 600-grit silicon carbide paper were prepared and divided into three groups (n = 150) according to the composite cements used: Variolink Esthetic DC (VE), Multilink Automix (MA), and SpeedCEM (SC). Each group was further divided into five subgroups (n = 10) according to the surface treatment performed: no treatment (control), Monobond Plus (MP), 37% phosphoric acid and Monobond Plus (PA), < 5% hydrofluoric acid and Monobond Plus (HF), and Monobond Etch & Prime (ME). All treated ceramic specimens were bonded with three composite cements and light cured. After 24-h water storage and 5000 or 10,000 thermocycles, tensile bond strength (TBS) was measured. The specimens underwent failure mode analysis. The results were statistically analyzed using two-way ANOVA and t-tests with Bonferroni correction.

RESULTS: The TBSs were significantly influenced by surface treatments (p < 0.05). There were no significant differences in HF and ME among most of the groups. Furthermore, ME showed the highest bond strength with MA after 10,000 thermocycling. Most specimens of the ME groups exhibited cohesive failures, whereas a combination of adhesive failures and mixed failures were observed in control, MP, PA, and HF groups.
CONCLUSION: In the surface treatment of lithium disilicate glass ceramics, Monobond Etch & Prime was found to be a possible substitution for the combination of hydrofluoric acid and Monobond Plus.

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Effectiveness of Titanium Tetrafluoride on the Bond Strength of Resin Cement to Titanium.

Effectiveness of Titanium Tetrafluoride on the Bond Strength of Resin Cement to Titanium.

J Adhes Dent. 2018 Apr 24;:1-7

Authors: Elsaka SE

Abstract
PURPOSE: To assess the influence of titanium tetrafluoride (TiF4) solution on the adhesion of composite cement to commercially pure titanium (cp Ti).
MATERIALS AND METHODS: cp Ti plates with dimensions of 30 mm × 8 mm × 1.5 mm were machined and polished. The specimens were divided into seven groups according to the surface treatment as follows: group 1: control (machined); group 2: sandblasted with 110 μm Al2O3; group 3: hydrofluoric acid (HF); group 4: TiF4 (5%-5 min); group 5: TiF4 (5%-10 min); group 6: TiF4 (10%-5 min); and group 7: TiF4 (10%-10 min). One type of composite cement (Multilink Speed) was applied to each group for assessing the bond strength using strain energy release rate (Gvalue, J/m2) test. SEM analysis and surface roughness evaluation of cp Ti were carried out after treatment. The debonded specimens
were examined with a stereomicroscope and SEM. Data were analyzed by the Kruskal-Wallis and Dunn’s multiple comparison tests. Statistical significance was set at the 0.05 probability level.

RESULTS: All the tested groups showed significantly higher bond strengths compared with the control group (p < 0.05). Surface treatment of cp Ti with TiF4 (10%-10 min) showed higher bond strength compared with sandblasting and HF groups (p < 0.05). Surface topography of treated cp Ti showed alterations in surface roughness and morphology.

CONCLUSION: Adhesion between composite cement and cp Ti could be improved by using TiF4 (10%-10 min) solution prior to composite cement application as an alternative technique to sandblasting and HF.

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Durability of Resin Bonding to Lithium Disilicate and Zirconia Ceramic using a Self-etching Primer.

J Adhes Dent. 2017 Dec 12;:1-6

Authors: Wille S, Lehmann F, Kern M

Abstract

PURPOSE: The purpose of this in vitro study was twofold: 1. To evaluate the surface conditioning effect of a self-etching ceramic primer on lithium disilicate
and zirconia ceramics; (2) to study the bond durability provided by the self-etching ceramic primer after artificial aging compared with conventional ceramic conditioning methods.

MATERIALS AND METHODS: Lithium disilicate blocks (10 × 10 mm, 3.4 mm thick) and zirconia disks (8 mm diameter, 3.4 mm thick) were each divided into two groups. In group 1, the lithium disilicate disks (Li) were etched with hydrofluoric acid (HF), while zirconia (Zr) disks were treated with airborne-particle abrasion, both followed by application of a universal primer for restorative materials (MP; Monobond Plus, Ivoclar Vivadent). In group 2, Li disks were not etched with HF, while Zr disks were treated with airborne-particle abrasion, both followed by a self-etching primer (ME; Monobond Etch & Prime, Ivoclar Vivadent). Surface conditioning effects were evaluated using SEM. The specimens in both groups were bonded to a composite with a luting resin and divided into two subgroups. Subgroup 1 was stored in water (37°C) for 3 days, and subgroup 2 was stored in water for 30 days before undergoing 7500 thermal cycles (5°C to 55°C).

RESULTS: The self-etching ceramic primer had a significant effect only on the lithium disilicate surface topography. The mean initial bond strength of ME-Zr was relatively low (24.4 MPa) in comparison with all other material combinations (MP-Li: 34.3 MPa; ME-Li: 33.5 MPa; MP-Zr: 31.1 MPa). After 30 days of water storage and thermocycling, the bond strength decreased significantly in all groups.

CONCLUSION: The self-etching primer provided bond strengths to lithium disilicate ceramic comparable with those of the well-established bonding method using hydrofluoric acid etching and a primer containing silane. To zirconia ceramic, however, it provided statistically significantly lower bond strength than did the established bonding method.

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Abstract

PURPOSE: To investigate the effects of room-temperature etching with hydrofluoric acid (HF) in the presence and absence of ultrasonic irradiation on the bonding of yttria-stabilized tetragonal zirconia polycrystals (Y-TZP) to resin.

MATERIALS AND METHODS: Y-TZP specimens were etched with 40% HF at room temperature for different time periods (2, 5, 10, 15, 30, 60, and 90 min) with and without ultrasonic exposure. The surface roughness, micromorphology, dimensions, and phases of the treated Y-TZP specimens were evaluated by atomic force microscopy (AFM), scanning electron microscopy (SEM), digital caliper measurement, and x-ray diffraction, respectively. The HF etching conditions that resulted in the most drastic Y-TZP surface morphology and highest roughness values were used to prepare specimens for shear bond strength (SBS) testing; the effect of thermocycling on SBS was also examined. Alumina-sandblasted Y-TZP specimens were used as the control.

RESULTS: The Y-TZP surfaces etched with HF without ultrasonic exposure for 30, 60, and 90 min and those surfaces ultrasonically etched with HF for 10 and 15 min were severely etched, although their dimensions were not changed by etching. Monoclinic-phase zirconia was observed only in the alumina-sandblasted Y-TZP specimens. Surface roughening from HF etching for 30 min and ultrasonic etching for 10 min resulted in higher mean SBS compared to roughening with alumina sandblasting.

CONCLUSION: Ultrasonic etching with 40% HF at room temperature for 10 min may be used as an alternative roughening method for improving the bonding of Y-
Abstract

PURPOSE: To evaluate the influence of different surface treatments of six novel CAD/CAM materials on the bonding effectiveness of two luting composites.

MATERIALS AND METHODS: Six different CAD/CAM materials were tested: four ceramics – Vita Mark II; IPS Empress CAD and IPS e.max CAD; Celtra Duo – one hybrid ceramic, Vita Enamic, and one composite CAD/CAM block, Lava Ultimate. A total of 60 blocks (10 per material) received various mechanical surface treatments: 1. 600-grit SiC paper; 2. sandblasting with 30-μm Al2O3; 3. tribochemical silica coating (CoJet). Subsequent chemical surface treatments involved either no further treatment (control), HF acid etching (HF), silanization (S, or HF acid etching followed by silanization (HF+S). Two specimens with the same surface treatment were bonded together using two dual-curing luting composites: Clearfil Esthetic Cement (self-etching) or Panavia SA Cement (self-adhesive). After 1 week of water storage, the microtensile bond strength of the sectioned microspecimens was measured and the failure mode was evaluated.
RESULTS: The bonding performance of the six CAD/CAM materials was significantly influenced by surface treatment (linear mixed models, p < 0.05). The luting cement had a significant influence on bond strength for Celtra Duo and Lava Ultimate (linear mixed models, p < 0.05). Mechanical surface treatment significantly influenced the bond strength for Celtra Duo (p = 0.0117), IPS e.max CAD (p = 0.0115), and Lava Ultimate (p < 0.0001). Different chemical surface treatments resulted in the highest bond strengths for the six CAD/CAM materials: Vita Mark II and IPS Empress CAD: S, HF+S; Celtra Duo: HF, HF+S; IPS e.max CAD: HF+S; Vita Enamic: HF+S, S. For Lava Ultimate, the highest bond strengths were obtained with HF, S, HF+S. Failure analysis showed a relation between bond strength and failure type: more mixed failures were observed with higher bond strengths. Mainly adhesive failures were noticed if no further surface treatment was done. The percentage of adhesive failures was higher for CAD/CAM materials with higher flexural strength (Celtra Duo, IPS e.max CAD, and Lava Ultimate).

CONCLUSION: The bond strength of luting composites to novel CAD/CAM materials is influenced by surface treatment. For each luting composite, an adhesive cementation protocol can be specified in order to obtain the highest bond to the individual CAD/CAM materials.

Zirconia-reinforced Lithium Silicate Ceramic.

J Adhes Dent. 2016 Apr 1;

Authors: Al-Thagafi R, Al-Zordk W, Saker S

Abstract

PURPOSE: To test the effect of surface conditioning protocols on the reparability of CAD/CAM zirconia-reinforced lithium silicate ceramic compared to lithium-disilicate glass ceramic.

MATERIALS AND METHODS: Zirconia-reinforced lithium silicate ceramic (Vita Suprinity) and lithium disilicate glass-ceramic blocks (IPS e.max CAD) were categorized into four groups based on the surface conditioning protocol used. Group C: no treatment (control); group HF: 5% hydrofluoric acid etching for 60 s, silane (Monobond-S) application for 60 s, air drying; group HF-H: 5% HF acid etching for 60 s, application of silane for 60 s, air drying, application of Heliobond, light curing for 20 s; group CO: sandblasting with CoJet sand followed by silanization. Composite resin (Tetric EvoCeram) was built up into 4 x 6 x 3 mm blocks using teflon molds. All specimens were subjected to thermocycling (5000x, 5°C to 55°C). The microtensile bond strength test was employed at a crosshead speed of 1 mm/min. SEM was employed for evaluation of all the debonded microbars, the failure type was categorized as either adhesive (failure at adhesive layer), cohesive (failure at ceramic or composite resin), or mixed (failure between adhesive layer and substrate). Two-way ANOVA and the Tukey’s HSD post-hoc test were applied to test for significant differences in bond strength values in relation to different materials and surface pretreatment (p < 0.05).

RESULTS: The highest microtensile repair bond strength for Vita Suprinity was reported in group CO (33.1 ± 2.4 MPa) and the lowest in group HF (27.4 ± 4.4 MPa). Regarding IPS e.max CAD, group CO showed the highest (30.5 ± 4.9 MPa) and HF the lowest microtensile bond strength (22.4 ± 5.7 MPa). Groups HF, HF-H, and CO showed statistically significant differences in terms of all ceramic types used (p < 0.05). The control group showed exclusively adhesive failures, while in HF, HF-H, and CO groups, mixed failures were predominant.

CONCLUSIONS: Repair bond strength to zirconia-reinforced lithium silicate ceramics and lithium-disilicate glass ceramic could be improved when ceramic surfaces are sandblasted with CoJet sand followed by silanization.
Zirconia Surface Treatments for Resin Bonding.

Zirconia Surface Treatments for Resin Bonding.

J Adhes Dent. 2015 Dec;17(6):551-8

Authors: Cheung GJ, Botelho MG

Abstract

PURPOSE: To evaluate the bond strength of resin to zirconia treated with different surface conditioning methods.

MATERIALS AND METHODS: Sintered zirconia was surface treated to create 7 groups. Ceramic liner (L) was fired onto three groups of zirconia and subsequently received the following treatments: hydrofluoric acid etching (L/HFE), alumina particle abrasion (L/APA), and alumina particle abrasion with hydrofluoric acid etching (L/APAHFE). All three groups were silane treated immediately prior to bonding. Two other zirconia groups received alumina particle abrasion with and without silane coupling (AP-S and AP). Another group underwent selective infiltration etching, in which the specimens received porcelain powder firing, ultrasonic etching with HF for 15 min, then rinsing under running water for 15 min, followed by silane treatment (SIE). The control group was zirconia as-sintered (ZAS). Twenty composite resin cylinders were luted to each group with a resin cement. Each group was divided into two subgroups (n=10) and subjected to 2 storage conditions: 24 h water storage or 21 days with 6000 thermocycles between 5°C and 55°C. Shear bond strength testing (SBS) was performed, followed by statistical analysis of the results using one-way ANOVA (p < 0.05).

RESULTS: After 21 days of thermocycling, AP and ZAS groups spontaneously debonded prior to testing. The remaining groups showed a decrease in mean
shear bond strength between 11.7% and 58.5% after thermocycling, except the 
L/HFE group, which increased by 11.7%. L/HFE showed the highest bond 
strength at both test intervals, and at 21 days was significantly higher than that of 
the AP-S and L/APA-HFE groups, which in turn were higher than that of the 
L/APA group, which was higher than that of the SIE group (p < 0.05). 
CONCLUSION: The etched, fired ceramic liner with silane treatment provided the 
strongest and most durable bond under the conditions tested. Alumina particle 
abrasion degraded the durability of the ceramic liner. Alumina particle abrasion, 
as-sintered zirconia, and SIE did not provide durable bond strengths.

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**Effect of Hydrofluoric Acid Concentration on Resin Adhesion to a Feldspathic Ceramic.**

J Adhes Dent. 2015 Aug 20;

Authors: Venturini AB, Prochnow C, Rambo D, Gundel A, Valandro LF

Abstract

PURPOSE: To evaluate the effect of different concentrations of hydrofluoric acid 
(HF) on the contact angle and the resin bond strength durability to feldspathic 
ceramic.

MATERIALS AND METHODS: To evaluate the contact angles of distilled water on 
etched feldspathic ceramic, 25 specimens (12 × 10 × 2.4 mm) of VitaBlocks Mark 
II were used, divided into 5 groups (n = 5): one unconditioned control (UC) group
with no ceramic surface treatment, and 4 other groups that were etched for 60 s with different concentrations of HF: 1% (HF1), 3% (HF3), 5% (HF5) and 10% (HF10). The bond testing utilized 40 ceramic blocks (12 × 10 × 4 mm) that were fabricated and subjected to the same surface treatments as previously mentioned (excluding the control). The etched surfaces were silanized and resin cement was applied. After 24 h, the blocks were sectioned to produce bar specimens that were divided into two groups, non-aged (immediate testing) and aged (storage for 230 days + 12,000 thermocycles at 5°C and 55°C), and subjected to microtensile testing (μTBS). Micromorphogical analysis of the treated surfaces was also performed (atomic force and scanning electron microscopy). One-way ANOVA and Tukey’s tests were applied for data analysis.

RESULTS: UC had the highest contact angle (61.4°), whereas HF10 showed the lowest contact angle (17.5°). In non-aged conditions, different acids promoted statistically similar bond strengths (14.2 to 15.7 MPa) (p > 0.05); in terms of bond durability, only the bond strength of the HF1 group presented a statistically significant decrease comparing before and after aging (14.5 to 10.2 MPa).

CONCLUSION: When etched with 3%, 5%, or 10% hydrofluoric acid, the ceramic tested showed stable resin adhesion after long-term aging.

Microtensile Bond Strength of Lithium Disilicate Ceramics to Resin Adhesives.
Abstract
Purpose: To evaluate the influence of the internal structure of lithium disilicate glass ceramics (LDC) on the microtensile bond strength to a resin adhesive using two surface treatments. Materials and Methods: Milling blocks of three types of LDC were sectioned (4 mm thick) using a precision cutting machine: IPS Empress 2 (conventional LDC), IPSe.max CAD (a refined crystal high strength LDC), and Celtra (zirconia reinforced LDC). Cut specimens received crystallization heat treatment as suggested by the manufacturers. Two surface treatments were performed on each group: hydrofluoric acid etching (HF) and airborne particle abrasion using 50-μm glass beads, while the as-cut surface served as control. Treated surfaces were examined using scanning electron microscopy (SEM). The disks were coated with a silane primer and bonded to pre-aged resin composite disks (Tetric EvoCeram) using a resin adhesive (Variolink II) and then stored in water for 3 months. Bonded specimens were sectioned into micro-bars (1 x 1 x 6 mm) and microtensile bond strength test (MTBS) was performed. Data were analyzed using two-way ANOVA and Tukey’s post-hoc test ($\alpha = 0.05$). Results: Statistical analysis revealed significant differences in microtensile bond strength values between different LDCs ($F = 67, p < 0.001$), different surface treatments ($F = 232, p < 0.001$), and interaction between LDC and surface treatments ($F = 10.6, p < 0.001$). Microtensile bond strength of Celtra ceramic ($30.4 \pm 4.6$ MPa) was significantly higher than both IPS Empress 2 ($21.5 \pm 5.9$ MPa) and IPSe.max ceramics ($25.8 \pm 4.8$ MPa), which had almost comparable MTBS values. SEM images demonstrated homogenous glassy matrix and reinforcing zirconia fillers characteristic of Celtra ceramic. Heat treatment resulted in growth and maturation of lithium disilicate crystals. Particle abrasion resulted in abrasion of the glass matrix and exposure of lithium disilicate crystals, while HF etching produced a microrough surface, which resulted in higher MTBS values and reduction in the percentage of adhesive failure for all groups. Conclusions: Within the limitations of this study, bond strength to lithium disilicate ceramics depends on proper surface treatment and on the chemical composition of the glass ceramic.

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Can Heat Treatment Procedures of Pre-hydrolyzed Silane Replace Hydrofluoric Acid in the Adhesion of Resin Cement to Feldspathic Ceramic?

J Adhes Dent. 2013 Nov 18;

Authors: Cotes C, Carvalho RF, Kimpara ET, Leite FP, Ozcan M

Abstract
Purpose: To evaluate the influence of heat treatment (HT) procedures of a pre-hydrolyzed silane on bond strength of resin cement to a feldspathic ceramic.

Materials and Methods: Ceramic and composite blocks (N = 30) were divided into six groups (n = 5) and subjected to the following conditioning procedures: G1: 9.6% hydrofluoric acid (HF) for 20 s + silane (RelyX Ceramic Primer, 3M ESPE) + resin cement (Panavia F2.0, Kuraray) (control); G2: HF (20 s) + silane + heat treatment in furnace (HTF) (100°C, 2 min) + resin cement; G3: silane + HTF + resin cement; G4: HF (20 s) + silane + heat treatment with hot air (HTA) (50 ± 5°C for 1 min) + resin cement; G5: silane + HTA + resin cement; G6: silane + resin cement. The microtensile bond strength (MTBS) test was performed using a universal testing machine (1 mm/min). After debonding, the substrate and adherent surfaces were analyzed using a stereomicroscope and SEM to categorize the failure types. The data were statistically evaluated using one-way ANOVA and Tukey’s test (5%). Results: The control group (G1) showed no pre-test failures and
presented significantly higher mean MTBS (16.01 ± 1.12 MPa) than did other groups (2.63 ± 1.05 to 12.55 ± 1.52 MPa) (p = 0.0001). In the groups where HF was not used, HTF (G3: 12.55 ± 1.52 MPa) showed significantly higher MTBS than did HTA (G5: 2.63 ± 1.05 MPa) (p < 0.05). All failure types were mixed, ie, adhesive between the resin cement and ceramic accompanied by cohesive failure in the cement. Conclusion: Heat treatment procedures for the pre-hydrolyzed silane either in a furnace or with the application of hot air cannot replace the use of HF gel for the adhesion of resin cement to feldspathic ceramic. Yet when mean bond strengths and incidence of pre-test failures are considered, furnace heat treatment delivered the second best results after the control group, being considerably better than hot air application.

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