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Bond Strength of Machinable Ceramic Using Conventional Silane and Multipurpose Primer.

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Abstract:

Purpose: To evaluate the bond strength to machinable glass ceramic using conventional silane and multipurpose primer after different surface treatments to composite build up.

Materials and methods: A total of 64 lithium disilicateIPS e.max CADceramicspecimens with dimension(12X10X3) were fabricated using milling technology, thespecimens were equally divided into 4 main groups (n=16) according to the method of surface treatment as follows. In Group I: polished, Group II: Acidulated phosphate fluoride (APF) application, Group III: Hydrofluoric acid (HF) application, Group IV:Monobond Etch & Prime (MEP) application. Each main group was further equally subdivided into 2 subgroups (n=8) according to methods of chemical bonding. Subgroup (A):Conventional silane.Subgroup (B):Multipurpose primer.Composite resin cylinder (Master fill) were fabricated and cemented to the treated specimens by multistep adhesive resin cement. All bonded specimens were stored in water bath for 5 months and subjected to 10000 thermal cycles. Shear bond strength (SBS) test was performed afterwards. Scanning Electron Microscope (SEM) was used for specimens examination

.**Results:** The statistical analysis were done using two-way ANOVA and serial one-way ANOVAs followed by Post Hoc Tukey-HSD test at α =(0.05). Application of multipurpose primersignificantly increased SBS (P=0.001). The highest SBS mean value (14.8 ± 2.2 MPa) was obtained for APF followed by Monobond Etch & Prime with conventional silane (14.7 ± 2.8 MPa)On the other hand,APF with conventional silane showed the lowest SBS mean value(4.6 ± 2.0MPa). There was statistically significant difference between polished and APF considering conventional silane and multipurpose primer, and no statistically significant difference was reported between HF and Etch & Prim for conventional silane and multipurpose primer.

Conclusions: Multipurpose primershowed superior bond strength in comparison to conventional silanewhen used for conditioning of lithium disilicateIPS e.max CADceramics.

Keywords: Hydrofluoric acid, Monobond Etch & Prime, Acidulated phosphate fluoride, conventional silane, lithium disilicate IPS e.max CAD.
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Introduction

Metallic restorations have a long-standing history of clinical use in dentistry. However, increase in patients' esthetic expectations and demands caused the metallic restorations to be sidelined and led to the development and emergence of porcelain fused to metal (PFM) restorations.[(1)] Despite the brittle nature of porcelain, PFM crowns are widely used because the metallic frameworks afford superior mechanical durability.[(2)]

Recently, all-ceramic restorations, including a large variety of glass-ceramics and polycrystalline ceramics, have been used in dentistry owing to their superior mechanical properties, high flexural strength, excellent esthetic and biocompatibility.[(3)] Over the last two decades, with the development of computer aided design & computer aided manufacturing (CAD/CAM) technology, digital dentistry has gained popularity and (CAD/ CAM) fabricated all-ceramic restorations have been used instead of exhausting and time-consuming traditional techniques.[(4)]

The current gold standard for cementation of silica based ceramics is the pretreatment of the ceramic with hydrofluoric acid (HF) etching, followed by application of a silane coupling agent and subsequently a resin cement.[(5)] The HF acid etches ceramic surfaces containing at least some glassy component, leading to increased surface area for micromechanical interlocking to enhance bond strength.[(6)]

1.23% acidulated phosphate fluoride (APF) gel, which is weaker than hazardes HF acid, is used as an alternative etchant as it was more beneficial to the patient, it serves as a safe and effective substitute for etching porcelain surfaces.[(7, 8)]

Recently, another option for ceramic surface treatment has been tested and is considered a potential alternative to or even a substitute for hydrofluoric acid etching, as it is less toxic.[(9)]Single-bottle, multi-purpose primers which contain varying contents of a silane coupling agent, acidic adhesive monomers and dithiooctanoate monomers were investigated for their effectiveness of bonding a light-curing resin composite to the ceramic adherends. Study showed that a ternary combination of silane coupling agent, acidic adhesive monomers, and dithiooctanoate monomers was effective in delivering high bond strengths to the all adherends [(16)]

The purpose of this in-vitro study was to evaluate the shear bond strength (SBS) of lithium disilicate glass ceramic (IPS e.max CAD) to resin composite material using conventional silane and multipurpose primer with different surface treatment methods.

Materials and methods:

Specimens' preparation:

Sixty-four ceramic specimens (n=64) were fabricated from Lithium disilicate glass ceramic block (IPS e.max CAD, Lot No. P27379, Ivoclar Vivadent, Schaan, Liechtenstein) by using milling technology. Ceramic specimens were designed by fabrication of a wax pattern with the feting dimension (length=12 mm, width=10 mm and thickness=3mm).

Specimens grouping:

According to methods of surface treatment all 64 ceramic specimens were divided into 4 maingroups (n=16)Group I: specimens were subjected to polishing process (RA 105 Diamond,Lot No. 431282, Eve, Ernst Vetter, Gmbh, Germany polisher points W16dg, W16 Dmf.W16D). Group II: Specimens were etched by 1.23% Acidulated phosphate fluoride (APF, Ionite,Lot No. D190918, USA) that was applied with small brush for 4 min. Group III: Specimens were etched by 4.5% Hydrofluoric acid (HF, Lot No. W44153, Ivoclar Vivadent, Schaan/Liechtenstein) that was applied with small brush for 1 min. Group IV:Specimenswere cleaned inultrasonic cleanerand then etched by Etch & Prime (MEP, Lot No. V09353, Ivoclar Vivadent, Schaan/Liechtenstein) that applied onto bonding surface of the ceramicspecimens using small brush for 1min. All Etched specimens were steam cleaned, ultrasonically cleaned in 95% alcohol for 5 min and dried with oil free air., Each main group were divided in to 2 subgroupsaccording to chemical conditioning:Subgroup (A): Conventional silane (SCA, Lot No. BHFMZ, Ultradent Jordan USA)Subgroup (B): Multipurpose primer (Monobond N,Lot No. Y29210, Ivoclar Vivadent, Schaan/ Liechtenstein).

Composite resin discs fabrication:

The total number of composite resindiscsproduced was 64 using multi holes teflone mold (4 mm internal diameter and 3 mm thickness). The teflone holes were filled with composite resin incrementally (Biodinâmica, Ibiporã,Paraná,Brazil)

Bonding of resin composite discs to IPS e.max CAD specimens:

Composite resindiscs were cemented to previously treated surfaces of IPS e.max CAD specimens using resin cement (Multilink®N, Lot No. Y26001, ivoclar vivadent, Liechtenstein) Ceramic specimens were secured to a specially designed device with lever system.The composite discswere then placed onto the ceramic specimen and the constant load 1 Kg was applied on the composite disc, excess resin cement was removed with a brush then curing was done using light cure (liteQ LD-107, MONITEX, Taiwan) from four directions for 20 seconds. from each surface, and the constant load was left for 5 min. After cementation, Specimens were stored in a water bath at 37°C one hour after cementation for a continuous five months. Specimens were thermalcycled for 10000 cycles by thermalcycling simulation machine (THE-1100, SDMechatronik, Germany) between 5° and 55°C in water.

Shear bond strength test (SBS):

The universal testing machine (Model 3345: Instron Industrial Product, Norwood, MA, USA) was used for shear bond strength measurement with a cell of load 5 KN at across head speed of 0.5 mm/min.

Mode of failure evaluation:

The mode of failure was determined by examination of bonding surface of debonded specimensusing optical reflection microscope (S300II; Inoue Attachment Corp) at × 8magnification and was divided into three types:

1) Adhesive mode of failure: failure between the ceramic and resin cement interface.
2) Cohesive mode of failure: failure within composite resin disc or resin cement.
3) Mixed mode of failure: including cohesive and adhesive failure.

Scanning electron microscope (SEM):

In order to investigate surface characterization of debonded specimens, one specimen from each subgroup was examined using SEM (JEOL.JSM.6510LV) at different magnifications (50x, 500x, 1000x, 2000x). (Figure.1)

Statistical analyses:

Data were analyzed with statistical package for social science (SPSS) version 25(SPSS Inc. Chicago,Ⅲ, USA). The normality of data was first tested with Shapiro-Wilk test, variables were presented as mean ± SD (Standard Deviation) In several steps, statistical analysis of data was performed. Initially, descriptive statistics for each group results. Two-Way ANOVA test was used to detect the effect of each variable (Chemical bonding and surface conditioning methods) on shear bond strength. Tukey (HSD) honest significant difference was used for multiple comparison between different groups

Results:

Shear bond strength results

Results showed that mean shear stress at max load for conventional silane polished was (6.2 MPa), Acidulated Phosphate Fluoride was (4.6 MPa), Hydrofluoric Acid was (13 MPa) and Etch & prime (14.7 MPa). For Multipurpose primer mean shear stress at max load was 13.3 MPa polished, 14.8 MPa for Acidulated Phosphate Fluoride, 12.6 MPa for Hydrofluoric Acid and 13.5 MPa for Etch & Prime.(Table.1) Two Way ANOVA test was used to estimate the combined effect of changing priming agent and surface treatment on shear stress on max load and revealed that the combined effect of changing priming agent and surface treatment had statistically significant effect ($p<0.001$) on shear stress with 70.1% of shear stress can be affected by their combined effect. One-way ANOVA for the effect of different surface treatments with conventional silane priming Showed thatthere was statistically significant difference between studied groups in surface treatment for conventional silane priming for shear stress at maximum load ($F=29.83$, $P=0.001$). One-way ANOVA for the effect of Multipurpose primer & Etch and prime on shear stress at maximum loadShould thatthere is no statistically significant difference between studied groups in Etch & prime surface treatment for Multipurpose priming for shear stress at maximum load (F=0.885,P=0.461).One Way ANOVA for comparison of shear stress at maximum load between studied groups Should thatthere is statistically significant difference between studied groups with different surface treatments (polishing, APF, HF and Etch and prime) for conventional silane& multipurpose priming for shear stress at maximum load (F=17.35, P=0.001). Post Hoc Tukey test was used to detect pairwise comparison between different surface treatments for conventional priming and illustrates that the following pairs have statistically significant difference; Polished & HF (6.2 & 13, p=0.001), Polished & Etch & Prime (6.2 &14.7, p=0.001), APF & HF (4.6 &13, p=0.001) and APF & Etch & Prime (4.6 & 14.7, P=0.001).

Student t test was used to compare between multipurpose primer & conventional silane priming illustrates a statistically significant higher mean value among multipurpose prime than conventional for polished surface treatment (13.3 versus 6.2) and APF (14.8 versus 4.6) without statistically significant difference for HF and Etch & Prime surface treatments Table (1)

Mode of failure

Failure pattern of all the debonded specimens showed mainly mixed failure pattern (32 specimens) followed by cohesive failure pattern (19 specimens) and the least was adhesive failure pattern (13 specimens).

Table: (1)comparison between multipurpose primer andconventional silane for different surface treatment agents.

Groups	multipurpose primer	conventional silane	p-value
Polished	13.3 ±1.8	6.2 ± 3.4	0.001*
APF	14.8 ± 2.2	4.6 ± 2.0	0.001*
HF	12.6 ± 3.3	13.0 ± 1.7	1.00
Etch &Prime	13.5 ± 3.3	14.7 ± 2.8	0.986

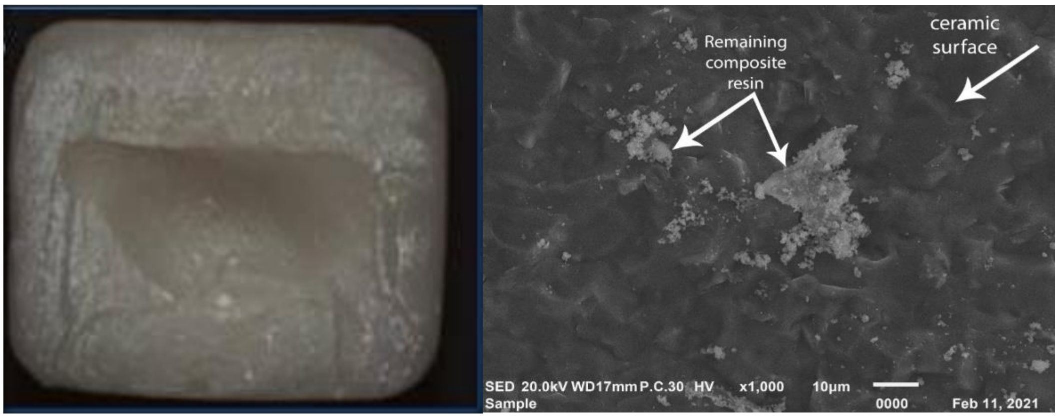

Figure (1): Mode of failure: Cohesive failure. SEM micrograph of group (polish-MPP) cohesive failure mode. At X 1000 magnification.

Discussion:

The objective of the present research was to evaluate the shear bond strength (SBS) of IPS e.max CAD glass ceramic to resin composite material using conventional silane and multi-purpose primer after diverse surface treatments. The null hypotheses tested were that there would be no difference in shear bond strength (SBS) between both materials using either conventional silane or multi-purpose primer and also the ceramic surface treatment approach had no effect on its values. The study results showed significant variations in the SBS values as a result of applying different surface treatment methods (p<0.001), as well as significant differences with varied priming agent (p<0.001). Therefore, both tested hypotheses were rejected.

In the present study, HF acid application showed better results considering higher SBS valueseither with conventional silane (13 mpa) or multi-purpose primer (12.6 mpa) and not observed adhesive failure type. This is in line withthe results of **Kilinc et al, (2020)**[10] who concluded that HF etching was more effective in achieving durable bond strength for glass ceramics.

Surface analyses of HF acid- and APF gel-etched porcelain have revealed the production of markedly different etching patterns. APF gel etching produced minimal surface roughness. It produced only a few shallow pores and undercuts, whereas etching with HF acid showed greater roughness and irregularity.[8, 10] This can be the reason for significantly decreased SBS value measured when APF gel was used only for 4 min with silane treatment (4.6 mpa) in comparison to HF acid with silane group (13 mpa).

Concerning the lithium disilcate ceramic represented in this study by the IPS e.max CAD, the non-significant difference found between HF-etched and Etch and Prime-treated specimens came on the agreement with **Alrahlah et al, (2017)**[11]who recorded comparable shear bond strength results between resin cement and lithium disilicate specimens treated with HF with silane and Monobond Etch and Prime. They attributed these results to that Monobond Etch and Prime contains trimethoxypropyl methacrylate for silanization and polyfluoride for etching. **Siqueira et al, (2016)**[12] and **Roman-Rodriguez et al, (2018)**[13] found no statistically significant difference in micro-shear bond strength of a lithium disilicate ceramic etched with Monobond Etch and Prime or HF. In addition, as the manufacturer claims, Monobond Etch and Prime achieves similar bond strength as the combination of HF etching with silane application.

Our results are not in accordance with those found by **El-Damanhoury and Gaintantzopoulou, (2018)**[14]in which Monobond Etch and Prime resulted in a smoother surface with fewer irregularities (contains ≤10% of tetrabutyl ammonium dihydrogen trifluoride which is less acidic than HF) and lower bond strengths than HF etching with multi-purpose primer application. Also, theyfound that pretreatment with HF followed by Monobond plus primer resulted in higher SBS in comparison to the not etched polished specimens treated only with the Monobond plus primer. Conversely, our study showed decreased SBS values in case of treatment with the HF and multi-purpose primer (12.6 mpa) when compared with the Etch and Prime method (13.5 mpa) or through using the primer with polished specimens (13.3 mpa).

Our results showed that HF etching with conventional silane produced SBS (13 mpa) better than HF with multi-purpose primer (12.6 mpa), but without significant difference. This may come from the fact that universal primers (Monobond N) are primarily composed of functional and hydrophilic monomers which may contain silane. Silane may be unstable when combined with 10-methacryloyloxydecyl dihydrogen phosphate (MDP) in a one-bottle solution. Under acidic conditions, such as in the presence of MDP and water, a self-condensation reaction might occur between the silanol groupsresulting in a polysiloxane oligomer.[15]

This study observed comparable bond strength values between specimens treated with Etch and Prime method (13.5 mpa) and others treated using HF etching with universal primer (12.6 mpa). This finding is compatible with the results of a study by **Wille et al, (2017)**[16], but not in line with **Dimitriadi et al, (2020)**[17]who concluded that the HF etching has induced significantly higher values than Monobond Etch and Prime.

SEM images were used to observe and investigate morphological changes in the interface after different methods of lithium disilicate glass ceramic surface treatment.

Conclusions:

Within the limitations of this in-vitro study, the following conclusions could be drawn: The multipurpose primer has a superior bond strength in comparison with conventional silane when used for lithium disilicate IPS e.max CAD ceramic bonding regardless the used type of surface treatment.

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