

Influence of Shear Bonding Strength of Lithium Disilicate to Enamel under Different Surface Treatments

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ABSTRACT

BACKGROUND

Accurate diagnosis and proper treatment planning should be established before making any prosthetic rehabilitation to restore the good appearance of the tooth, a better smile, phonetics, and to achieve perfect occlusion/mastication. We wanted to evaluate the changes of surface treatment protocols on the tensile bonding strength between enamel and ceramic restorations (lithium disilicate).

METHODS

A total of 20 freshly extracted, non-carious teeth were stored in normal saline solution at 25°C until used. Clean enamel surfaces 2 mm X 2 mm were obtained from the extracted teeth by using wheel diamond bur. Each enamel side received one E-max sprue, so four sprues of different surface treatment protocols per tooth (G1 TS: Etching, bonding, and curing. CS: Etching, monobond, curing, overall light cure) (G2 TS: Bonding, curing, self-etch / self-adhesive resin cement, overall light cure. CS: Monobond etch and prime) (G3 TS: Etching, bonding without curing. CS: Etching, monobond, curing, overall light cure) (G4 TS: Etching, bonding, curing. CS: Etching, monobond, bonding, curing, light-cure resin cement, overall light cure). Tensile bonding strength was measured using the Instron testing machine. One-way ANOVA test was used to analyse the data.

RESULTS

The highest mean was observed in Group 3 (124.34±43.47) followed by Group 1 (104.29±50.09), which is control group. In contrast, the lowest mean was observed in Group 2 (83.64 ± 53.56) and Group 4 (94.14±57.91). The results of ANOVA test have shown a significant difference between the surface treatment groups at 5% significance level.

CONCLUSIONS

Hydrofluoric acid and primer/silane coupling agent create a porous surface on the ceramic that allow a good interaction with silane coupling agent.

KEY WORDS

Enamel, Hydrofluoric Acid, Lithium Disilicate, Primer, Silane, Tensile Bonding Strength

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BACKGROUND

The success and reliability of ceramics bonding to dental substrate result in the long-term survival of aesthetic restorations. Several surface treatments regarding chemical bonding and micromechanical retention were suggested to enhance resin cement bonding to ceramics.^{1,2} The best surface treatment to be utilized is determined through the composition of the ceramic. Similarly, the effect of etching protocols used on glass matrix ceramics has been examined by several studies.³⁻⁶ In this regard, acid etching is considered as the most effective treatment method for the bonding surface of glass ceramic restorations. Selective removal of the glassy matrix of silicate ceramics allows micromechanical interlocking of the luting composite, resulting in a micromorphological 3D porous surface.⁴ The acid type and its concentration, the treated ceramic type, and the etching time are dependent components for achieving successful effects of acid etching.⁵

One of the most often used acids is the hydrofluoric acid while the ammonium hydrogen difluoride and acidulated phosphate fluoride is also frequently observed.⁶ Some silicon ammonium fluoride and tetrafluoride are created due to the mixture of silica matrix and the ammonium hydrogen difluoride. The use of this acid is examined as an intermediate or a glass etching for the hydrofluoric acid production.⁷ Higher bond strengths are generated through hydrofluoric acid etching followed by silanization as compared to the treatment alone. The application of silanization is understood in creating covalent bonding and hydrogen bonding between the ceramic and the resin as well as escalated wettability of the ceramic surface whereas the mechanical interlocking is provided by etching.⁸ Silane promotes the chemical adhesion to promote higher mean bond strength values as compared to the micromechanical retention created by any etchant.

Recently, the instigation of a simplified acid ceramic primer has claimed to perform a mild acid silanization and etching using a single solution.⁹ This monobond Etch & Prime (MEP) and one bottle system combine silane and ammonium polyfluoride on the basis of trimethoxy propyl methacrylate to show a thin chemically bonded layer.¹⁰ The introduction of this primer was to simplify the bonding procedure by priming glass and etching ceramics using a one-step process. In particular, a milder acidity is observed in ammonium polyfluoride as compared to hydrofluoric acid, resulting in weaker etching pattern.¹¹ Several prior studies have been conducted to compare the efficiency of the protocol using 2-step surface treatments and silane followed by concentration and application times.¹²⁻¹⁴ Comparative outcomes are provided by these studies on field-emission scanning electron microscopic analysis, micromorphological analysis, tensile bond strength, shear bond strength, and contact angle.

The bonding strength is the main source of the E-max/lithium disilicate restorations to compare the other types of crowns, which include Zirconia or PFM, which retains the cement itself for producing the E-max unique.¹⁵ Not only the preparation designs, resistance, and retention forms have been reported as highest factors that affect the longevity of the bonding strength. However, there are many other factors considered for the type of restorations on tooth surface or its natural teeth with no restorations on it.^{16,17} Individuals are

looking for tooth coloured and aesthetic crowns specifically in the anterior region, which reduce all ceramic restorations.¹⁸ Furthermore, the reliance of a durable ceramic crown is on the cementation technique and the cement type. Ceramic restorations are recommended for different types of cement, if a lithium disilicate restoration is used based on the silica.¹⁹

The resin cements have many advantages such as it comes with different shades and highly aesthetic materials. Most importantly, the bond strength between the ceramic/lithium disilicate and tooth structure is considered due to limited literatures.¹⁴ Also, the surface treatment of the fitting side of the ceramic restorations should take place to increase the bond strength. The usual technique and materials to increase the bonding strength are divided into tooth part and ceramic part surface treatment. The conventional surface treatment onto the tooth side is to etch by 35% phosphoric acid and then resin bond. Hydrofluoric acid and primer/silane coupling agents were used for the ceramic side as they create a porous surface on the ceramic that allow a good interaction with silane coupling agent.¹¹ Despite of the advantages of the HF acid etching followed by silane application, the protocol of etching ceramics is still not clear.¹⁰ In this regard, the study aims to evaluate the changes in surface treatment protocols on the tensile bonding strength between enamel and ceramic restorations (lithium disilicate). Following hypotheses have been proposed based on the aforementioned objective.

Hypothesis

H₀: There is no difference in tensile bonding strength between surface treatment protocols.

H₁: There will be a difference in tensile bonding strength between surface treatment protocols

METHODS

Experimental Design and Specimen Preparation

A total of 20 freshly extracted, non-carious molars teeth were stored in normal saline solution at 25°C until used. Every tooth was mounted in orthoresins block [Protechno Famadent, S. L. U. 17469 Vilamalla (Girona) Spain]. The enamel of the four tooth surfaces (buccal, lingual, mesial, and distal) were roughened by using Wheel Diamond Bur which represented the tooth bonding surface. 80 heat pressed E-Max sprues (Ivoclar Vivadent, Schaan, Liechtenstein) of clean ceramic were used in this study. Each ceramic sprue had 2x2 surface diameter. Then, those E-max sprues were cemented on the different surface sides of the same tooth which carried different surface treatments protocols. All sides of tooth-ceramic surfaces had the same bonding surface area.

Microshear Bond Strength (μ SBS)

The following experimental groups were formed for determining microshear bond strength test.

Group 1 (Control)

On the tooth surface

1. 37% Phosphoric Acid semi Gel Meta Etchant (meta Biomed CO. LTD, United Kingdom).
2. Washing for 10 seconds then drying for 10 seconds.

- Bonding with resin bonding agent (Optibond, Kerr via Passanti, Italia).
- LED Light cure 10 seconds.

On the ceramic surface

- Etch with 9% HF acid (Ultradent Porcelain Etch 9% buffered hydrofluoric acid) for 20 seconds.
- Washing for 10 seconds then drying for 10 seconds.
- Monobond N Refill (Ivoclar Vivadent AG FL-9494, Schaan/Liechtenstein) for 60 second. Then gentle air drying for 10 seconds.
- Light-cure resin cement (Kerr corporation NX3 NEXUS Third Generation, U. S. A).
- Overall LED Light cure for 30 seconds.

Group 2

On the tooth surface

- Bonding with resin bonding agent (Optibond, Kerr via Passanti, Italia).
- LED Light cure 10 seconds.
- Self-Etch / Self-Adhesive Resin Cement (Maxcem Elite Clear, Kerr via Passanti, Italia).
- Overall LED Light cure for 30 seconds.

On the ceramic surface

- Monobond Etch and prime (Ivoclar Vivadent AG 9494, Schaan/Liechtenstein) for 60 seconds.
- Washing for 10 seconds then drying for 10 seconds.

Group 3

On the tooth surface

- 37% Phosphoric Acid semi Gel Meta Etchant (Meta Biomed CO.LTD, United Kingdom)
- Washing for 10 seconds then drying for 10 seconds
- Bonding with resin bonding agent (Optibond, Kerr via Passanti, Italia) without curing

On the ceramic surface

- Etch with 9% HF acid (Ultradent Porcelain Etch 9% buffered hydrofluoric acid) for 20 seconds.
- Washing for 10 seconds then drying for 10 seconds.
- Monobond N Refill (Ivoclar Vivadent AG FL-9494, Schaan/Liechtenstein) for 60 second. Then gentle air drying for 10 seconds.
- Light-cure resin cement (Kerr corporation NX3 NEXUS Third Generation, U.S.A).
- Overall LED Light cure for 30 seconds.

Group 4

On the tooth surface

- 37% Phosphoric Acid semi Gel Meta Etchant (Meta Biomed Co. Ltd., United Kingdom).
- Washing for 10 seconds then drying for 10 seconds
- Bonding with resin bonding agent (Optibond, Kerr via Passanti, Italia).
- LED Light cure 10 seconds.

On the ceramic surface

- Etch with 9% HF acid (Ultradent Porcelain Etch 9% buffered hydrofluoric acid) for 20 seconds.
- Washing for 10 seconds then drying for 10 seconds.

- Monobond N Refill (Ivoclar Vivadent AG FL-9494, Schaan/Liechtenstein) for 60 seconds. Then gentle air drying for 10 seconds.
- Bonding with resin bonding agent (Optibond, Kerr via Passanti, Italia).
- LED Light cure 10 seconds.
- Light-cure resin cement (Kerr corporation NX3 NEXUS Third Generation, U. S. A).
- Overall LED Light cure for 30 seconds.

The specimens were mounted on the bottom fixture of a universal testing machine (the Instron testing machine) and the top apparatus grips the bonding resin cement.

Formula for the Calculation of Tensile Bond Strengths

$$\sigma = P/A$$

where,
 σ is the tensile bond strength,
 A is the interfacial area (mm²), and;
 P is the force (N).

Statistical Analysis

One-way ANOVA and Tukey test were used to analyse the data using a statistical program (SPSS version 22, IBM, Chicago, IL) ($p < 0.05$). Furthermore, mean \pm SD for surface treatment groups were presented through descriptive statistics.

RESULTS

Descriptive Statistics					
	N	Minimum	Maximum	Mean	S.D.
Group 1	19	25.3700	216.2400	104.294211	50.0917033
Group 2	14	11.98	209.81	83.6457	53.56625
Group 3	19	44.0900	188.7700	124.342105	43.3786934
Group 4	18	5.2400	194.7600	94.141111	57.9162065

Table 1. Shear Bond Strength of Lithium Disilicate with Different Surface Treatments (Mean \pm SD)

Note: SD = Standard Deviation

ANOVA					
	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	15342.922	3	5114.307	1.947	.031
Within Groups	173360.356	66	2626.672		
Total	188703.279	69			

The one-way ANOVA: statistically significant at $p < 0.05$

Tukey HSD				
	Mean Difference	Std. Error	Sig	
Group 1	Group 2	6.68	1.891	0.003
	Group 3	6.10	1.891	0.008
	Group 4	12.12	1.891	0.000
Group 2	Group 1	6.68	1.891	0.001
	Group 3	6.65	1.891	0.000
	Group 4	6.20	1.891	0.000
Group 3	Group 1	8.14	1.891	0.001
	Group 2	11.51	1.891	0.003
	Group 4	8.64	1.891	0.000

Table 2. One-Way ANOVA with Post-Hoc Tukey Test for Surface Treatments

The highest mean was observed in Group 3 (124.34 \pm 43.47) followed by Group 1 (104.29 \pm 50.09), which is control group. In contrast, the lowest mean was observed in Group 2 (83.64 \pm 53.56) and Group 4 (94.14 \pm 57.91). Mean \pm standard deviation for shear bond strength of lithium disilicate with different surface treatments are presented in Table 1. Table 2 shows one-way ANOVA with post-hoc Tukey test for surface treatments. The results of ANOVA test have shown a

significant difference between the surface treatment groups. The statistical power of the performed test is 0.031 at 0.05 significance level.

DISCUSSION

This study has evaluated and compared the tensile bonding strength after the ceramic surface was conditioned with different surface treatments. Traditionally, HF acid etching and primer is carried out separately as a two-step technique. Nevertheless, recently, both procedures are completed in a one-step conditioning system. The results of this study showed and compared between one-step conditioning system and the two-step conditioning system as part of this study. One-step conditioning system had lower adhesion strength values than the two-step conditioning system.

A study by Prochnow et al.²⁰ also reported lower adhesion strength values with the one-step conditioning system. This is because of the weak acidic salt of hydrofluoric acid in the ammonium poly-fluoride (poly-NH₄HF₂) that the one-step conditioning system Monobond TM Etch and Prime.

The 5% HF acid etch had been used in the two-step conditioning system, which creates low surface roughness. Siqueira et al.²¹ have also reported that partial dissolution of the glassy phase will be created, when ceramic surfaces were etched by using one step conditioning system. Poor resin cements flow into the micropores could be explained by surface roughness and mechanical interlocking. The low surface roughness adversely affects the mechanical interlocking with resin tags.²² Chemical bonding with a silane coupling agent requires adequate acid etching to exposed hydroxyl ions to facilitate good bonding strength.²³

The specimens used in Group 3 showed a greater bond strength without curing of bonding agent that applied on tooth surface as compared to cure bonding agent of tooth surface in Group 1 (control group). This could be related to the intimate contact of enamel to light cure resin cement, consequently, to improve the overall bond strength. Tribst et al.¹¹ have presented both ceramics for HF-treated surfaces based on contact angles and wettability. The study has indicated that the exposure of chemical ligands and removal of the glass matrix is likewise in both tested ceramics. However, the surface wettability should be influenced by any overtreatment for increasing the number of pores. Tribst et al.¹¹ have further assessed and compared the ceramic-resin cement adhesion strength after undertaking one-step and two-step conditioning systems. Acid etching and primer application was independently carried out in the conventional two-step conditioning system. The study has found some differences between the resin composite and adhesive resin for some fundamental cement attributes, which include viscosity, mode of curing, tension, and wettability. Ramakrishnaiah et al.⁸ have studied the influences of disinfectant, saliva, hydrogen peroxide contamination, and desensitizer to investigate adhesive-dentin bonding using a clinical try-in process. The findings obtained did not support the findings of the current study and indicated that the tensile bond strengths of both adhesive-ceramic interfaces and adhesive dentin are similar for all dentin surface situations.

Thereby, this study has rejected that there is no difference in tensile bonding strength between surface treatment protocols. The results have indicated that the silane aspect was not effective to optimize the ceramic resin bond before applying the universal adhesive. It is argued that pre-treated lithium disilicate along with silane should be used by clinicians before applying the universal adhesive. This claim has been corroborated by the findings of previous study that the implementation of silane improves microshear bond strength between composite resin and lithium disilicate from 4.10 MPa to 14.58 MPa.^{4,8} Furthermore, when silane was applied with etched HF to lithium disilicate, the microshear bond strength was improved from 14.04 MPa to 24.70 MPa.⁸

The importance of topographic change is to maintain adhesive strength as better micromechanical bonding area is allowed through an increased number of widths and pores of various sizes. The wettability of the ceramic surface for the application of resin composites and silane-coupling agents is affected by this increase in surface roughness.^{20,21} The importance of adequately porous surface is observed for the durable cementation of both Fd and Ld ceramic indirect restorations.²⁴ Clean surfaces, the use of low-viscosity adhesives, cements, and suitable wetting are the other factors that contribute to the effectiveness of bonding between the materials.²⁵

The results of the present study have recommended and indicated lower adhesion strength values when a one-step conditioning is used for ceramic surface as compared to the two-step conditioning system. Lower adhesion strength values with the one-step conditioning system were also reported by Prochnow et al.²⁰ The glassy phase in the ceramic is etched by using prime liquid and Monobond Etch for creating low surface roughness. Siqueira et al.¹ have indicated that the one-step conditioning system is used for the glassy phase when the ceramic surface was etched.

In bonding interfaces, adhesive systems tend to water sorption, which might absorb water from the ceramic structure, resulting in mechanical properties reduction and polymer plasticization, but might further offer water for the cleavage of siloxane bond.¹⁰ A significant reduction of bond strength at the interface is occurred over a time period, when a silanized interface is exposed to water due to the presence of hydrolytic cleavage of siloxane bonds in the siloxane.³ Therefore, all of the aforementioned concerns might contribute to a minimized bond strength investigated in all the groups of this study. Comparing the HF alone, the correlation between self-etching ceramic primer and HF did not indicate any significant modification in the modifying pattern of the lithium disilicate. In contrast, the less pronounced etching pattern is promoted through the self-etching ceramic primer, which leads to a partial rejection of the null hypothesis.

Similar findings have been reported by Siqueira et al.¹ that the association between the ceramic surface and ammonium polyfluoride achieves self-etching ceramic primer to produce the etching pattern. A partial dissolution of the glassy matrix is produced as compared to the production made by hydrofluoric acid where ammonium polyfluoride is comprised of milder acidity and acidic salt as compared to hydrofluoric acid. Furthermore, a partial dissolution is yet sufficient for the promotion of an adhesive interlocking with the ceramic

surface. Therefore, the self-etching ceramic primer cannot be observed as a drawback to produce the less-pronounced etching pattern. It should be noted that any significant benefits cannot be brought due to a more-pronounced etching pattern with respect to its bond strength.

The smear layer and depleted fluoride ions were removed from the acid-etching procedure in the dentin surface, which allow a hybrid layer formation. The tubules are penetrated by resin monomers, which form resin tags with a jagged-like characteristic. In contrast, the etching procedure is neutralized by the deposition of fluoride ions on the dentin surface as well as inhibited a hybrid layer formation.^{26,27} These effects of fluoride deposition on the dentin surface might correspondingly interfere with the bonding process, which indicated that dentin bond strength might be decreased by fluoride-containing desensitized.

Several limitations in this study have been observed. Firstly, only one type of resin cement was used in this study. Secondly, the interpretation of the results was restricted due to the small number of specimens as a higher number of specimens might produce substantial differences with a smaller standard deviation. Thirdly, crystallographic analyses and surface roughness measurements were not examined in this study. Lastly, not curing the bonding agent of the total etch results a higher bond strength. Therefore, future studies are suggested to observe adhesive luting since modifications in wetting ability, mechanical properties and viscosity, and chemical compositions can further impact the adhesive properties of resin cement. Furthermore, both silanization and etching should be performed prior to bonding for lithium disilicate. It is suggested to use different resin cements to evaluate the same present hypothesis. Many available universal adhesives have diverse compositions, which can be used in different interactions with the surface pre-treatment method.

CONCLUSIONS

The use of HF etching and silane to lithium disilicate before applying a universal adhesive, improves resin-lithium disilicate bond strength. It is important to apply a regimen of 5% HF at 20 seconds for reducing surface damage to the ceramic when using the universal adhesive along with silane. Similarly, the use of 9.5% HF can increase bond strength for 60 seconds if an additional step is not applied before adding a universal adhesive. Once self-etching achieves the chemical interaction and long-term strength than the conventional treatment, it can be treated as an alternative to conventional ceramic treatment. The self-etching system is important to maintain adhesive bonds to glass ceramics on the basis of positive outcomes obtained for the surface treatments as well as for the possible risks related with HF procedures.

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