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Effect of Etching Time and Technique on Bonding to Machinable Glass Ceramic

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Abstract:

Objectives: the purpose of this in-vitro study was to investigate the effect of different surface treatment and various etching times on the shear bond strength of machinable glass ceramic using 2 resin cements. Materials and Methods: A total of 64 ceramic samples (10x12x3.5 mm) were fabricated from (IPS e.max CAD) using CAD/CAM technology. Ceramic samples were divided into two main groups (n=32) according to surface treatment: Hydrofluoric acid followed by Monobond N application or Monobond Etch and prime. According to the etching time, either 60 or 120 sec each main group was divided into two subgroups (n=16). Composite resin discs were fabricated (4mm diameter x 3mm thickness) dimensions. Each subgroup was further subdivided into two divisions (n=8) according to type of luting agent used, either adhesive resin cement (Multilink N) or self-adhesive resin cement (G-CEM LinkAce). Bonded specimens were stored in water bath at 37°C for 5 months then exposed to 10,000 thermal cycles. Universal testing machine was used to record the shear bond strength values for each specimen. Failure analysis was performed using a stereomicroscope (10x) and a scanning electron microscope (SEM). Results: SBS values ranged between 11.9 and 15.1 MPa in groups treated with HF pluse Monobond N and between 13.9 and 15.8 MPa in groups treated with MEP. ANOVAs test showed statistically significant difference between the two etching agents used for surface treatment as well as luting agent used for the bonding. However, etching time factor had no statistically significant difference. Moreover, the interaction between the factors had no statistically significant difference P ($\leq$0.05). Most of specimens exhibited cohesive and mixed failure mode. Conclusions: MEP showed comparable SBS results to the combination of HF and Monobond N after artificial aging. Etching time had no effect on the SBS results. The adhesive resin cement showed higher SBS values compared to self-adhesive resin cement regardless of the other factors.

Key words: CAD/CAM, lithium disilicate, Hydrofluoric acid, self-etching ceramic primer, resin cement.

Introduction

Over the last few decades the dental ceramics properties and manufacturing techniques has developed rapidly. Among these advancements is the developing of glass-ceramics, which are highly esthetic and possess exceptional mechanical properties. Lithium disilicate glass-ceramics can be used as resin-bonded veneers, inlays, onlays, crowns, and 3-unit bridges up to the second premolar due to their merits as high flexural strength, relatively high fracture toughness, and good adjustable translucency.[i]

Computer Assisted Design/Computer Assisted Machining (CAD/CAM) system technology can be used to produce restorations in one office visit. First the tooth is prepared, then the preparation is scanned optically and the image is computerized then the restoration is designed by a computer. Finally ceramic blocks are milled by a computer-controlled milling machine to form the restoration. The advantages of this technique are that the restoration accuracies and grants are better than that of traditional technique.[ii]

The success of glass ceramic restorations is attributed to their properties of micromechanical and chemical bonding to resin composite. Very important protocols that increase adhesion property of the bonding between cement and tooth are surface treatments. Hydrofluoric acid (HF) etching is one of the most established protocols for glass ceramics which modifies the ceramic surface by removing the exposed silica in the glassy matrix. Holes that produced in the glassy matrix help in the micromechanical retentive features for the resin composite and improve the wettability of the ceramic for the application of silane to facilitate chemical bond.[iii]

Recently hydrofluoric acid etching/silane coupling agent routine treatment has been altered with self-etching ceramic primer that was introduced as a single-component ceramic primer which aims to eliminate the toxic potential of the hydrofluoric acid, reduce the time required and the technique sensitivity of etching ceramic with the conventional methods.[iv]

Resin cements are the key factors for successful bonding of the glass ceramic restoration through formation of micromechanical and chemical bonding at ceramic/tooth interface. However, they are complicated and need several steps during cementation. Therefore, self-adhesive resin cements were introduced to simplify bonding procedures[v].Therefore, the purpose of this study was to evaluate the effect of etching time and technique on the bond strength of machinable glass ceramic.

Materials and Methods

A total of 64 rectangular samples of partially crystallized machinable glass ceramics (IPS e max CAD) were constructed using CAD/CAM technology with the following dimension (12 mm x 10 mm and 3.5 mm thickness). Subsequently, all the ceramic samples were crystallized in a programat ceramic furnace (P500, Ivoclar-

Vivadent) in accordance with the manufacturer's recommendations**.** A total number of 64 composite resin discs were constructed using teflon pattern with central hollow (4mm internal diameter and 3mm thickness).

Surface conditioning of ceramic specimens

The conditioning surface of the specimens was cleaned with a steam cleaner. **Group (1)** : Etching with HF acid plus universal primer application (Monobond N). HF acid <5% was applied with microbrush on the bonding surface of the sample for 1 or 2 minutes, then acid was thoruoghly rinsed off with air–water spray for 30 sec. After that the specimens were cleaned in ultrasonic bath for 5 min using 95% ethyl alcohol then dried with oil-free air stream for 30 sec. A thin coat of universal primer was applied to conditioned bonding surface and allowed to react for 60sec. Subsequently, the excess was dispersed with a strong stream of oil-free air to ensure the solvent evaporation .

Group (2): Bonding surface was treated using self-etching ceramic primer. Samples were ultrasonically cleaned in a bath of 95% ethyl alcohol for 5 min and air-dried before surface conditioning. After that a self-etching ceramic primer was applied on the bonding surface using a microbrush for 1 or 2 minutes, then totally removed with a strong jet of air/water spray for 30 sec and dried with oil-free air for another 30 sec.

Cementation procedure

. Bonding procedures were performed according to the manufacturers' recommendations for each luting agent:.**Division** (1): Multi-step adhesive resin cement, self-curing, transparent, two-past adhesive resin cement (Multilink N). Equal amounts of base and catalyst were dispersed on waxed paper pad, mixed for 30 sec using a plastic spatula until a uniform mixture was attained. **Division** (2): Self-adhesive resin cement (G-CEM LinkAce was dispensed onto a paper pad at a ratio 1:1 and mixed for 30 sec using plastic spatula and applied to the bonding surfaces of the ceramic samples.

Finally using specially design device the composite discs were placed onto the conditioning surfaces of the ceramic samples after cement application. A static load of (2 Kg) was applied on the ceramic sample/composite disc assembly and the excess luting cement was removed using disposable minibrush. Bonded specimens were light-cured from different sides for 40 sec with a handheld light-curing device. The bonding assembly was kept under a static load for 5 minutes.

Artificial aging

One hour after cementation all specimens were stored in water bath at room temperature for 5 months and thermocycling for 10,000 cycles.

Shear bond strength evaluation

Shear test was done by applying compressive load at the ceramic/composite interface. A mono-bevelled chisel-shaped metal bar attached to the upper movable part of the testing machine moving at a cross-head speed of 0.5 mm/min was used till debonding occured. The load at which debonding occurred was recorded in Newton (N) and then transformed into Megapascals (MPa).

Failure pattern analysis

The bonding surface of the debonded samples were evaluated with optical reflection microscope at ×10 magnification and Scanning Electron Microscope (SEM) to determine failure pattern. The failure patterns were classified into (1) Adhesive pattern of failure : failure between resin cement and ceramics (at interface). (2) Cohesive pattern of failure : failure took place in the composite resin discs or in cement layer. (3) Mixed pattern of failure : involving cohesive failure of the cement and adhesive failure between ceramic and resin cement.

Statistical analysis

Statistical analysis was done by Social Package for Statistical Science (SPSS) software version 25.0. Statistical analysis was done with three-way ANOVAs, two-way ANOVA and one-way ANOVA at each level of the study followed by Post Hoc Tukey test.

Results

The mean SBS of all test groups was compared across the following factors: surface treatment, etching time and luting agent. Three-way ANOVAs test showed that, surface treatment and luting agent had statistically significant effect on shear bond strength (p=0.012 & p=0.032, respectively). However, etching time had no statistically significant effect on shear stress (p=0.147). All combined effects between the studied independent factors had no statistically significant effect on shear bond strength (p>0.05). (Table 1)

Further analyses with serial 2-way ANOVAs were performed including the following factors: surface treatment ×etching time, surface treatment ×luting agent and etching time×luting agent. The interactions between surface treatment and etching time (p=0.218) and surface treatment and luting agent (p=0.250) and etching time and luting agent (p=0.284) were not significant. To determine which factor had the main effect on SBS ,further analyses with serial 1-Way ANOVAs test were used to detect the effect of each factor independently.

Furthermore, for multiple comparisons between different test groups, Post Hoc Tukey (HSD) test was used t to compare between each two test group. Post Hoc Tukey test showed that, a statistically significant difference between (self-adhesive cement at 2 minutes and multistep adhesive cement at 2 minutes within HF acid surface treatment test groups, p=0.01).

Failure mode analysis:

Failur pattern of debonded specimens showed mostly mixed failure pattern (44 specimens) followed by cohesive failure pattern (18) and the least was adhesive failure pattern (2 specimens).

Table 1: Summary of overall serial 3-way ANOVAs

Test group	Type III Sum of Squares	df	Mean Square	F	P-value
corrected model	85.649[a]	7	12.236	2.965	0.010
intercept	12756.629	1	12756.629	3091.051	0.000
surface treatment	28.157	1	28.157	6.823	0.012
etching time	8.903	1	8.903	2.157	0.147
luting agent	20.054	1	20.054	4.859	0.032
surface treatment * etching time	7.038	1	7.038	1.705	0.197
surface treatment * luting agent	5.896	1	5.896	1.429	0.237
etching time * luting agent	5.502	1	5.502	1.333	0.253
surface treatment * etching time * luting agent	10.099	1	10.099	2.447	0.123
error	231.109	56	4.127		
total	13073.388	64			
corrected total	316.759	63			

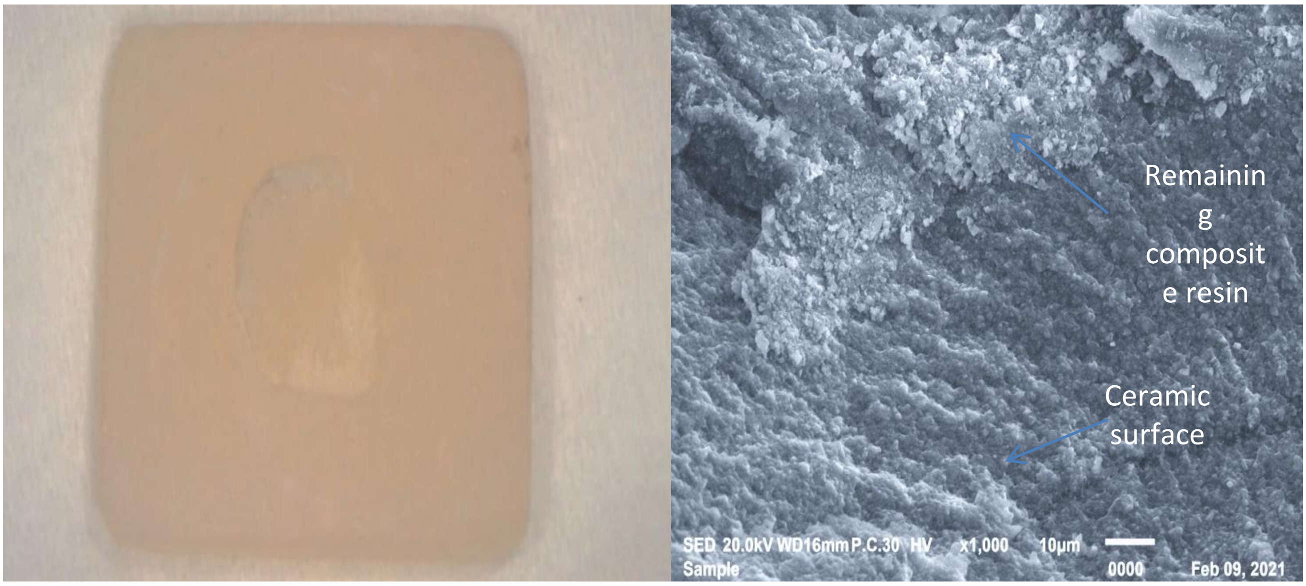

Figure 1: Showing Optical microscopic and SEM examination of group (HF-2M-MARC) with mixed failure mode

Discussion.

The hypotheses of this study should be partially rejected as both etching techniques and two luting cements used significantly affect SBS results, on the other hand etching time had no significant effect on SBS results.

Regarding the results of etching technique in the current study, the specimens that treated with HF plus Monobond N showed statistically significantly lower shear bond strength values than those obtained when treated with self-etching ceramic primer (MEP) in self-adhesive and multi-step adhesive resin cements after artificial aging. This may be due to, MEP contains trimethoxypropyl (responsible for chemical bond) methacrylate that can be attached to the lithium disilicate glass ceramic surface to form a permanent thin layer which is stable even after rinsing and drying.[vi]

These findings are in agreement with study by **prado et al. (2018)**[vii] who also reported that the new self-etching ceramic primer exhibited more bond strength values than hydrofluoric acid/silane when used for pretreatment of lithium disilicate ceramic after artificial aging (70 days water storage and 12000 thermocycling).

On contrast, the outcomes of this research not in agreement with El-**Damanhoury and Giantantzopoulou (2017)**[viii] and **Alkhudairy. (2018)**[ix] they showed that, etching with HF followed by Monobond plus application resulted in improved adhesion in comparison to pretreatment with self-etching ceramic primer.

On the other hand, the results of this in-vitro study showed that, etching time had no considerable effect on the bond strength between the resin cement and lithium disilicate glass ceramic. Moreover increasing the etching time did not significantly improved the bond strength. This might be due to the smaller pores and fissures that offer better mechanical interlocking sites than do the wider pores associated with long-term acid etching. Creating an adequately porous surface is a vital step for durable cementation of the indirect restoration, and this can be achieved by etching the surface for shorter etching cycles.[x]

This outcom was in agreement with **Verissimo et al. (2019)**[xi] who revealed that etching time factor had no significant effect on the bond strength of e.max CAD when etching for two different etching time. Other study was in agreement with our research work that showed by **Alshihri (2019)**[xii] who used MEP for different etching time and reported that there was no significant effect of the etching time on the bond strength and there was no additional benefit of increasing time of etching.

In the present study the type of the luting cement used had a significant effect on the shear bond strength. Results showed that, Multi-step adhesive resin cement regardless of technique of surface treatment showed higher SBS mean values than tested groups with self-adhesive resin cement. This low bond strength recorded for self-adhesive resin cement may be related to the initial low pH and higher viscosity of the self-adhesive cements, the low bond strength recorded for the self-adhesive resin despite the cement's limited ability to infiltrate the micropores with hydrolysis of the bonding interface. [9]

This outcome was in agreement with **Upadhyaya et al., (2019)**[xiii] who compared the shear bond strength (SBS) of multistep adhesive and self-adhesive resin cements used to bond the lithium disilicate restorations. They revealed that, there was statistically significant differences in SBS between multistep adhesive and self-adhesive resin cements. They reported that, multistep adhesive resin cement produced higher bond strength to glass ceramics when compared to self-adhesive resin cements

.The findings of this in-vitro study were not in agreement with **Aguiar et al. (2014)**[xiv] who noted that, a self-adhesive resin cement had good bond performance despite the surface treatment and the self-adhesive resin cement appears to be a good option for cementation. This might be due to they used natural teeth in their research with no thermocycling as artificial aging.

Considering failure pattern analysis of debonded specimens, the failure pattern were mainly mixed and cohesive failure pattern which indicated high bond strength values. These findings could be attributed to the difference in the microstructure of e max CAD, which led to better bond between resin luting materials and lithium disilicate in addition to higher intrinsic strength of this material.

Conclusions

1. The multi-step adhesive resin cement showed a superior bond strength in comparison to self-adhesive resin cement when used for lithium disilicate glass ceramic bonding regardless of etching techniques and times.
2. Using of Monobond Etch & Prime significantly increased bond strength to lithium disilicate glass ceramic compared to the combination of HF and Monobond N regardless of other factors.
3. Etching time did not had a significant effect on the bond strength to lithium disilicate glass ceramic.

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