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EFFECT OF USING SELF-ETCHING PRIMER VERSUS CONVENTIONAL ETCHING TECHNIQUE ON SHEAR BOND STRENGTH OF THREE GLASS CERAMIC MATERIALS TO RESIN CEMENT

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KEYWORDS

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ABSTRACT

Introduction: The success of ceramic restorations leans on establishing a strong bond between the resin cement and ceramic materials as well as between resin cement and tooth structure. **Aim:** This study aimed to evaluate the effect of self-etch ceramic primer compared to the conventional technique (hydrofluoric acid + silane) on the surface morphology, wettability, and the shear bond strength of glass ceramics to resin cement. **Material and methods:** Twenty rectangular shaped specimens were cut form 3 glass ceramic materials (IPS e.max CAD, Vita Suprinity and Obsidian); 10 large specimens (12x14x3mm) and 10 small specimens (5x5x3mm). The specimens were divided into three groups according to ceramic type, then each group was subdivided into two subgroups according to surface treatment protocol; hydrofluoric acid + silane or self-etch ceramic primer. The large specimens were bonded to their corresponding small specimens of the same ceramic material that had the same surface treatment. The samples were thermocycled for 5000 cycles, 5-55°C, 15 secs. Shear test was performed using universal testing machine. Furthermore, the effect of the two different surface treatments on the surface topography was evaluated using SEM. Contact angle of distilled water was measured on tested glass ceramic materials after different surface treatments. For this test, 6 additional large samples were assigned, two for each glass ceramic material. **Results:** The samples treated with hydrofluoric acid + silane combination recorded slightly higher mean shear bond strength values to resin cement than those treated with self-etch ceramic primer within the same tested material with a statistically insignificant difference. The wettability results showed more favorable contact angles with hydrofluoric acid + silane treatment than those obtained with self-etch ceramic primer. Data were analyzed by one way ANOVA and t-Tests at $p \leq 0.05$. **Conclusions:** The self-etch ceramic primer can efficiently replace the conventional surface treatment method.

INTRODUCTION

Dental ceramic restorations are highly appealing to dentists and patients owing to their combination of excellent mechanical and optical properties, in addition to their biocompatibility and chemical durability. A wide range of dental ceramic restorations are available including glass-based, oxide-based and resin-matrix ceramics [(1)].

The difference in the chemical nature of the materials of these restorations leads to variations in their mechanical properties and their bonding performance to different luting cements. The adhesive bonding of a ceramic restoration to tooth structure involves two distinctive interfaces that dictate and contribute to the success or failure of the restoration: tooth/resin interface and ceramic/resin interface. A successful restorative treatment requires optimization of both interfaces. This bond depends on understanding the internal structure of the restorative material and properly selecting the suitable surface treatment and resin adhesive [(2)].

The protocol of surface conditioning varies from one ceramic material to another. For glass ceramics, two main strategies are employed to enhance their bonding to the resin cement: chemical and mechanical. Etching with hydrofluoric acid (HF) selectively dissolves the glassy phase and makes the surface porous to achieve proper surface micro-roughness which improves the wettability and facilitates the mechanical interlocking of the resin cement [(3)].

Following the etching, a primer containing silane coupling agent is applied to the conditioned ceramic surface to provide chemical adhesion to the silica-containing ceramic substrate. Silane coupling agent contains two different reactive functional groups: one reacts with methacrylates to copolymerize with the organic matrix of the resin and the other is reactive towards silica in glassy structures to bond silicone dioxide with the OH groups of the ceramic surface [(4)].

Recently, one-bottle self-etch primers have been developed, these newly introduced one-component bonding systems contain ammonium polyfluorides that have been investigated as a possible etching media for dental ceramics as well as trimethoxypropyl methacrelate which is widely used in adhesive dentistry as a silane coupler. These primers enable etching and silanization in a single step [(5)].

These primers were introduced to replace the conventional treatment of hydrofluoric acid and silanization of glass ceramics. A one step etch and prime can reduce the time required and the technique sensitivity of etching and can avoid the possible hazardous effects of hydrofluoric acid (i.e., occupational and patient exposure to potential risks and biological damage from acid contact with living tissue) [(6)].

Alteration of the surface topography by different etching techniques will result in changes in the surface area and on the wetting behavior of the ceramic substrate. This may also change the ceramic surface energy and its adhesive potential to resin [(7)].

Wettability is the ability of a liquid to spread over the surface of a solid and is usually estimated by the contact angle of a dispersion liquid on a substrate which is employed as an indicator of the substrate's total surface energy.

Bond strength testing of adhesive systems is regarded as a reliable predictor of dental restorations' lifespan. The shear bond strength test is the most widely used test for determining the bonding effectiveness of different adhesive systems. Shear tests provide several advantages, including ease of specimen preparation, a simplified testing technique, the ease of specimen alignment with the loading device and a lower rate of pretest failure [(8)].

It was hypothesized that self-etch ceramic primer surface treatment of lithium-based glass ceramics will yield bond strength values comparable to those obtained from the conventional technique (hydrofluoric acid and silane).

MATERIALS AND METHODS

Specimen preparation

A total of twelve rectangular shaped specimens (12x14x3mm) and another twelve smaller specimens (5x5x3mm) were cut from each of the three CAD/CAM glass ceramic blocks; IPS e.max CAD (Ivoclar Vivadent, Germany), Vita Suprinity (Vita Zahnfabrik, Germany) and Obsidian (Glidwell dental laboratories, USA). The blocks were sectioned in the pre-crystallized state using a low speed cutting saw (Isomet 4000, Germany) under constant water irrigation. Twenty specimens from each glass ceramic material (10 large and 10 small) were used for the shear test. Two small specimens and another two large specimens from each glass ceramic material were used for scanning electron microscope (SEM) evaluation and wettability measurements, respectively.

Specimens of each glass ceramic material were fired following its crystallization program recommended by the manufacturer. After crystallization, the specimens with large dimensions were embedded in epoxy resin blocks (Kemapoxy, Egypt) with only one side exposed to be bonded to the small specimens.

Specimen grouping:

The specimens were divided into three groups (n=12) according to the glass ceramic material:

Group I: IPS e.max CAD specimens.

Group II: Obsidian specimens.

Group III: Vita Suprinity specimens.

Each group was subdivided into two subgroups (n=6) according to the surface treatment protocol:

Subgroup A: Conventional technique (Hydrofluoric acid + silane)

Subgroup B: Self etch ceramic primer.

Surface treatment of the specimens:

All the specimens of the tested groups were subjected to two protocols of surface treatment:

Conventional technique (5% hydrofluoric acid + silane) was used to treat 6 large and 6 small specimens from each glass ceramic material. The 5% hydrofluoric acid gel (IPS ceramic etching gel, Ivoclar Vivadent) was applied on the bonding surface for 20 secs then thoroughly rinsed off with water spray and then dried. After that the silane coupling agent (Monobond Plus, Ivoclar Vivadent, Germany) was applied for 60 secs and gently blown with air to allow excess solvent to evaporate. Silane was not applied on the specimens that were assigned for SEM analysis.

Self-etch ceramic primer (Monobond Etch and Prime, Ivoclar Vivadent, Germany) was used to treat 6 large and 6 small specimens from each glass ceramic material. The primer was applied on the bonding surface using a micro brush, agitated for 20 secs, and left to react for 40 secs. After that the specimens were rinsed with water spray for 10 secs then blown dry with compressed air.

Surface morphology examination:

Two small representative selective specimens (5x5x3mm) from each glass ceramic material were evaluated using SEM (Tescan Vega, Czech Republic) for assessment of surface morphological changes and etch pattern produced by each surface treatment. Within each ceramic material one specimen was selected from each treated subgroup. The specimens were mounted on coded aluminum stubs, then coated with 6-nm gold layer (Au) for 90 s using Quorum techniques Ltd, sputter coater (Q 150t, England). The images were captured at 4000x magnification with an acceleration voltage of 20 kV at working distance 18mm.

Contact angle measurement:

Two large specimens of each glass ceramic material (12x14x3mm) were used for the wettability measurement. One specimen was selected from each treated subgroup following the previously mentioned protocols. The contact angle was measured using the sessile drop technique with a camera-based goniometer (OCA 15EC, Germany) connected to a computer with a special software. A micro syringe adapted to the goniometer deposited a droplet of distilled water of volume 1 μ onto the ceramic surface. After 5 seconds, images were taken with a camera coupled at a fixed distance of 30 cm. Then, the contact angle was recorded using a software program. For each specimen five contact angle measurements were recorded at different areas of the specimen.

Bonding of the samples:

For the shear test, 5 small specimens of each glass ceramic material were bonded to their corresponding large specimens (embedded in the epoxy resin blocks) which had the same surface treatment. The dual cure adhesive resin cement (Variolink Esthetic DC, Ivoclar Vivadent) was dispensed from the auto mix syringe according to manufacturer's recommendations and applied to the treated surface of the small specimens. A load of (3 kg) was applied using a specially designed device to ensure a standardized pressure during bonding. The resin cement was light cured using a light cure device (BlueLEX LD-M4, Monitex, Taiwan) for 2 secs per side for the initial setting, then the excess cement was easily removed with a scaler (Nordent, USA). The resin cement was then light cured for 20 secs per side.

Thermocycling:

All the bonded specimens of the tested groups were thermocycled between (5-55ºC) using a thermocycling machine (Suez Canal University, Egypt) for 5000 cycles. Each cycle took 40 secs and consisted of 15 secs immersion at 5ºC, 10 secs interval and 15 secs immersion at 55ºC.

Shear bond strength test:

The shear test was performed using a computerized universal testing machine (TIRA test 2805, Germany). The specimens were firmly attached to the lower fixed compartment of the machine using a specially constructed device so that the bonding interface was parallel to the long axis of the chisel (Figure 1). Compressive shear force was applied at ceramic/cement interface until debonding with the chisel travelling at a cross head speed of 1 mm/min with a load cell of 500 Newtons [9].

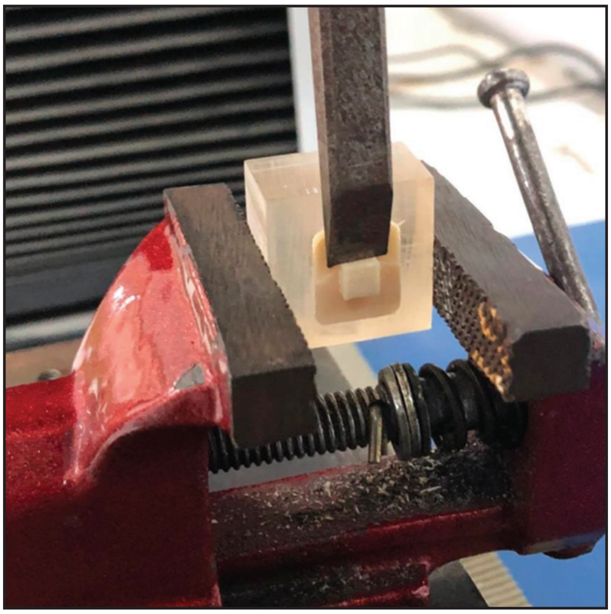

Fig. (1) Load applied at ceramic/cement interface.

Statistical analysis

Statistical analysis using one-way ANOVA and t-test with a statistical significance of P-value ≤0.05.

RESULTS

Surface morphology examination:

Scanning electron microscope evaluation showed the difference between the three glass ceramic materials with different surface treatments. The self-etch ceramic primer produced a superficial etch pattern, with limited micromechanical means of retention within the glassy matrix. On the other hand, hydrofluoric acid showed a homogenous and deep etch pattern with high surface irregularities of the treated surfaces (Figure 2).

Contact angle measurement:

The results showed that the ceramic samples treated with (hydrofluoric acid + silane) showed lower water contact angles (better wettability) compared to the samples treated with self-etch ceramic primer among the three glass ceramic materials (Table 1). The statistical analysis revealed that the contact angles differed significantly between the three ceramic materials and the two surface treatments.

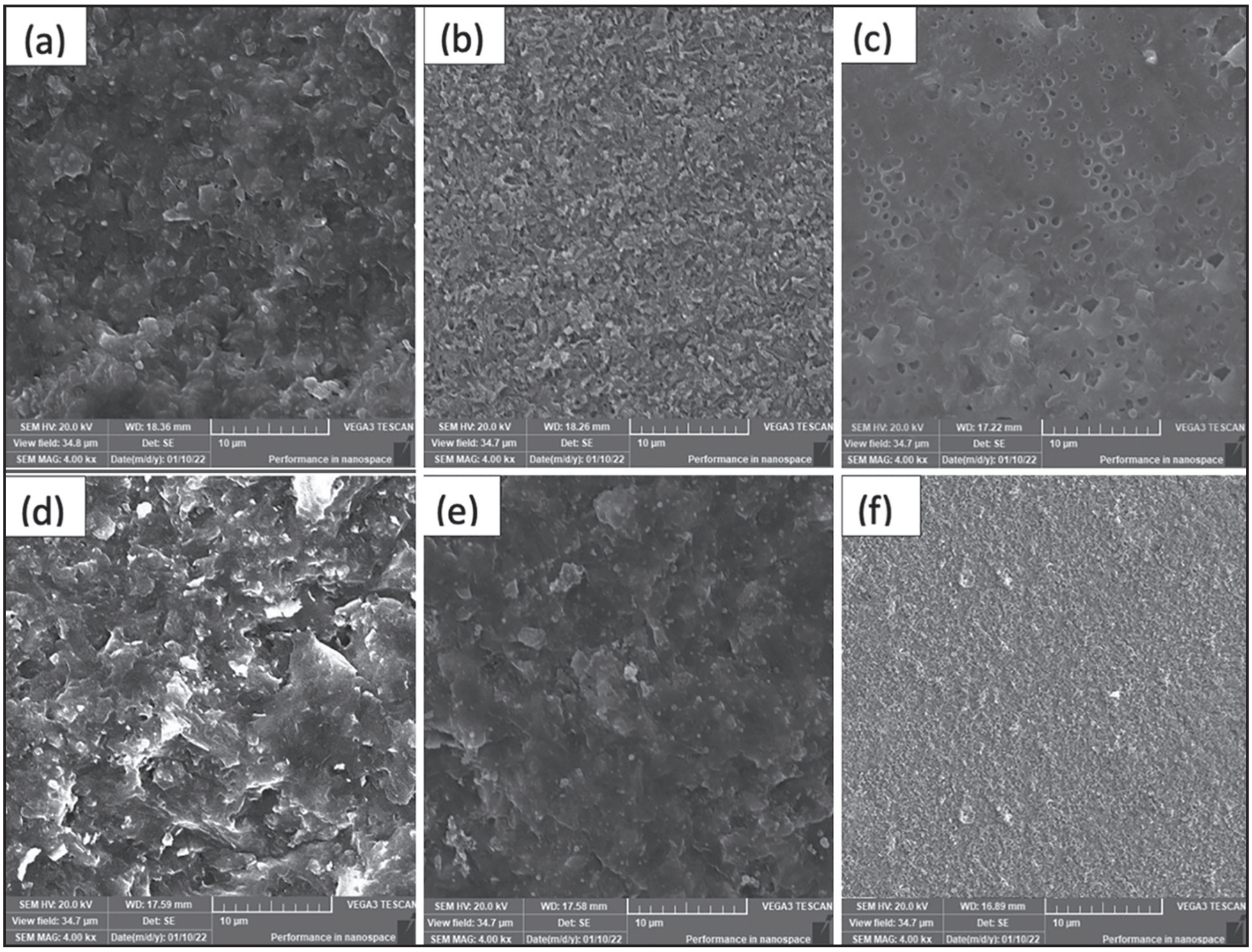

Fig. (2) (a) IPS e.max CAD etched with self-etch ceramic primer, (b) IPS e.max CAD etched with HF, (c) Obsidian etched with self-etch ceramic primer, (d) Obsidian etched with HF, (e) Vita suprinity etched with self-etch ceramic primer, (f) Vita suprinity etched with HF.

Table (1) *The mean contact angle and SD (in °) of each ceramic material with two different surface treatment protocols.*

Surface treatment \ Ceramic material	IPS e.max CAD Group (A)	Obsidian Group (B)	Vita Suprinity Group (C)	ANOVA P-values
HF + silane	23±1.4[c]	31.1±0.87[b]	45.1±0.59[a]	<0.001**
Self-etch	67±0.8[b]	57.8±0.81[c]	81±1.15[a]	<0.001**
P-values	<0.001**	<0.001**	<0.001**	

*-**; and different letters mean significant difference between groups (same row) using one way ANOVA at (P<0.05).*
; means significant difference between subgroups (same column) using T-test at (P<0.05).

Shear bond strength results:

The results showed that the samples of the three ceramic materials treated with (hydrofluoric acid + silane) recorded slightly higher mean values than those treated with self-etch ceramic primer with a statistically insignificant difference (Table 2). Moreover, the statistical analysis showed a significant difference between the three glass ceramic materials for each surface treatment.

Table (2) *Mean shear bond strength ± SD in MPa after thermocycling.*

Surface treatment \ Ceramic material	IPS e.max CAD Group (I)	Obsidian Group (II)	Vita Suprinity Group (III)	ANOVA P-values
HF+silane	26.53±1.03[a]	18.65±0.86[c]	21.5±1.1[b]	<0.001**
Self-etch	25.11±0.96[a]	17.33±0.97[c]	20.06±0.92[b]	<0.001**
P-value	0.055 Ns	0.057 Ns	0.056 Ns	

*-**; and different letters mean significant difference between groups (same row) using one-way ANOVA at (P<0.05).*
-Ns; means no significant difference (along the same column) using T-test at (P<0.05).

DISCUSSION

Dental ceramic restorations have been widely used for aesthetic and functional improvements because of their outstanding optical qualities, biocompatibility, and longevity(1). The category investigated in this study was glass-based ceramics. A successful restorative treatment requires optimization of ceramic/cement interface and its reliability and longevity which is affected by adhesive material and surface treatment protocols(2). This study assessed the influence of two surface treatment protocols; self-etch ceramic primer compared to the conventional technique (hydrofluoric acid etching and silane application) on the shear bond strength of adhesive resin cement to three different types of glass ceramics.

In this study thermocycling was used as an artificial aging technique. So, all specimens in this study were subjected to thermocycling for 5,000 cycles, which simulates conditions equivalent to 6 months of clinical use.

Shear bond strength test has been frequently employed in research in the field of bonding. This test not only evaluate the bond strength of adhesive/substrate combination but also the effectiveness of the surface treatment of the substrate on the bond [(8)]. An important aspect to use the shear bond strength test, is the high amount of shear stress concentration that occurs during chewing in an adhesively luted indirect restoration [(10)].

The contact angle of a dispersion liquid on a substrate is employed as an indicator of the substrate's total surface energy and wettability [(11)]. The sessile drop technique was used in this study to measure wettability since it has been widely used in many investigations.

In this study, the specimens treated with (hydrofluoric acid + silane) combination recorded slightly higher mean shear bond strength values to resin cement than those treated with self-etch ceramic primer within the same tested material with a statistically insignificant difference.

The difference in the results might be attributed to the different etching effects of hydrofluoric acid and the self-etch ceramic primer on the glass ceramic surface. Hydrofluoric acid etching results in removing the silica matrix and exposing the underlying crystalline structure, this produced high surface roughness, hence increased bonded surface area. On the other hand, ammonium polyfluoride contained in the self-etch ceramic primer is an acid salt with a milder acidic potential (pH=3.7) in comparison to hydrofluoric acid (pH=2) which might have resulted in less prominent etching effect on the treated ceramic specimens.

This explanation was confirmed by the scanning electron microscope findings in this study. Examination of the hydrofluoric acid etched surfaces of all the tested materials showed significant changes in surface topography, high surface irregularities, and micropores. Meanwhile, specimens etched with self-etch ceramic primer revealed less surface roughness and a smoother etch pattern compared to that produced by hydrofluoric acid etching [(12)].

Furthermore, wettability should be considered as an important factor that might affect the bond strength of resin cements to ceramics [(7)]. A higher wettability of a liquid maximizes its contact capability and attractive forces towards the substrate, which is an important requirement for a strong adhesion [(13)]. Wettability is influenced by the surface energy of the substrate that was affected by the different treatment protocols. Ceramic surface energy was increased after hydrofluoric acid etching. A high surface energy could be responsible for spreading of the silane primer, adhesive system and resin cement across the ceramic surface [(14)].

On the other hand, surfaces treated with self-etch ceramic primer were found to have fluoride remnants even after washing and the presence of fluoride is known for lowering the surface energy thus diminishing ceramic wettability, this could justify the finding that surfaces treated with self-etch ceramic primer were less susceptible to wetting[(15,16)].

Moreover, a significant and positive correlation was found between wettability and surface irregularities [(17)]. The deep etch pattern created by hydrofluoric acid, in turn increased surface area, liquid penetration, and eventually the wettability making it more receptive to the resin cement and thereby could be responsible for creating superior bond strength. Meanwhile, in case of self-etch ceramic primer the superficially produced etch pattern could be held responsible for decreasing the ceramic wettability.

Although surfaces treated with self-etch ceramic primer exhibited a less pronounced etch pattern and a higher contact angle than those exhibited by (hydrofluoric acid + silane) treatment protocol, however, they showed a relatively high bonding efficiency, and the differences between the two surface treatment protocols were statistically insignificant. This might indicate that the main mechanism of the bonding process with self-etching primer was the chemical bonding through the silane agent between the glass-ceramics and resin cement [18].

Moreover, the shear bond strength values of resin cement to ceramic treated with self-etch ceramic primer might be due to the chemical composition of the primer. According to the manufacturer, it contains functional monomers such as methacrylated phosphoric acid easter, which reacts with the ceramic ions. The presence of these monomers together with the methacrylate silane might increase the potential for chemical interaction[19]. It also contains a silane crosslinking agent (Bis (triethoxysilyl) ethane) that when combined with organofunctional silanes could enhance the crosslinking capacity of the siloxane network and subsequently might improve the adhesive properties under wet conditions [20].

Furthermore, a reliable bond was reported between silica and fluoride, thus, the chemical affinity between silica in glass ceramics and ammonium polyfluoride in self-etch ceramic primer could be held responsible for creating a bond strength.

Thermocycling was reported to be responsible for hydrolytically deteriorating the bond strength of adhesively bonded materials [21]. This could be attributed to temperature fluctuations that could induce stresses at the bonded interface due to the difference in the coefficient of thermal expansion between the bonded ceramic and the resin cement. However, in the current study thermocycling seemed to have a reduced detrimental effect on the ceramic/cement interface with self-etch ceramic primer compared to hydrofluoric acid. This might be related to the fluoride identified on the self-etch ceramic primer treated surfaces which was thought to increase the water contact angle (indicating low wettability), the hydrophobicity of the ceramic substrate and subsequently, the hydrolytic stability of the ceramic/resin cement interface [22]. Based on the previous scientific data it could be concluded that a stable chemical bonding was established between resin cement and the surfaces treated with self-etch ceramic primer that compensated for the low micromechanical retention. These results came in agreement with ***Vichi et al*** [23].

The results of the current study revealed that the effect of surface treatments on the shear bond strength of the resin cement to the CAD/CAM ceramic materials was material dependent. This is owing to differences in chemical composition, glass content and crystalline configuration between the three ceramic materials investigated in this study. With IPS e.max CAD, the lithium disilicate crystals form a needle-like or tetragonal structure. With Obsidian, the nano-sized lithium silicate crystals are spherical or monoclinic in shape, so that after removal of the glassy matrix the needle like crystals will project sharper and more prominent than the other spherical lithium silicate crystals [24]. Also, the lithium disilicate crystals with IPS e.max CAD are larger in size than lithium metasilicates and lithium orthophosphates crystals with Vita Suprinity, thus the varied molecular distribution might be responsible for giving various etch patterns on the surfaces [25].

CONCLUSION

Within the limitations and conditions of this in vitro study, it was concluded that simplification of the pretreatment protocol using the self-etch ceramic primer would give shear bond strength results comparable to the recommended hydrofluoric acid + silane pretreatment procedures.

Suggestions for further studies

It would be beneficial to support these findings with clinical studies. More investigations are still required considering other parameters like cement type and thickness before a clear recommendation for one surface treatment protocol can be made.

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