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JPOR 411 No. of Pages 9
ARTICLE IN PRESS
journal of prosthodontic research xxx (2017) xxx–xxx

Contents lists available at ScienceDirect

Journal of Prosthodontic Research

journal homepage: www.elsevier.com/locate/jpor

Original article

Self-etching ceramic primer versus hydrofluoric acid etching: Etching efficacy and bonding performance

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ARTICLE INFO

Article history:
Received 9 November 2016
Received in revised form 31 May 2017
Accepted 5 June 2017
Available online xxx

Keywords:
Adhesion
CAD/CAM ceramics
Hybrid ceramics
Bond strength
Surface treatment
Self-etching ceramic primer

ABSTRACT

Aim: This study assessed the effect of pretreatment of hybrid and glass ceramics using a self-etching primer on the shear bond strength (SBS) and surface topography, in comparison to pretreatment with hydrofluoric acid and silane.
Methods: 40 rectangular discs from each ceramic material (IPS e.max CAD;EM, Vita Mark II;VM, Vita Enamic;VE), were equally divided (n = 10) and assigned to one of four surface pretreatment methods; etching with 4.8% hydrofluoric acid followed by Monobond plus (HFMP), Monobond etch & prime (Ivoclar Vivadent) (MEP), No treatment (NT) as negative control and Monobond plus (Ivoclar Vivadent) with no etching (MP) as positive control. SBS of resin cement (Multilink-N, Ivoclar Vivadent) to ceramic surfaces was tested following a standard protocol. Surface roughness was evaluated using an Atomic force microscope (AFM). Surface topography and elemental analysis were analyzed using SEM/EDX. Data were analyzed with two-way analysis of variance (ANOVA) and post-hoc Bonferroni test at a significance level of α=0.05.
Results: Pretreatment with HFMP resulted in higher SBS and increased surface roughness in comparison to MEP and MP. Regardless the method of surface pretreatment, the mean SBS values of EM ceramic was significantly higher ($p < 0.05$) than those recorded for VM and VE, except when VE was treated with MEP, where the difference was statistically insignificant. Traces of fluoride ion were detected when MEP was used with VE and VM.
Conclusion: Under limited conditions, using MEP resulted in comparable SBS results to HFMP; meanwhile HFMP remains the gold standard for pretreatment of glass ceramics for resin-luting cementation.
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1. Introduction

Dental CAD/CAM has rapidly gained popularity among dental practitioners during the last decade. CAD/CAM generated dental restorations that may have several advantages over conventionally fabricated restorations; the ability to deliver the restoration in a single visit seems to be the most important advantage [1]. Other advantages of these systems include the uniform material quality, better physic-mechanical properties, the ability to reproduce the restorations and a significant reduction in production costs and time [2,3]. A wide range of CAD/CAM blocks are available for esthetic dental restorations including feldspathic glass ceramics, leucite-reinforced glass ceramics, lithium disilicate glass ceramics, aluminum-oxide and yttrium tetragonal zirconia polycrystals, composite resins [4] and hybrid ceramics [5]. The difference in the chemical nature of these materials leads to variations in their mechanical properties and their bonding performance to different luting cements [6].

The success of all-ceramic restorations leans on establishing a strong bond between the ceramic material and the tooth structure, especially for non-retentive restorations such as veneers and endocrowns [7]. This bond depends on understanding the internal structure of the restorative material and properly selecting the suitable surface treatment and resin adhesive. The main idea of ceramic pretreatment is inducing surface micro-roughness and then placement of a ceramic primer that facilitates the bonding to a more hydrophobic luting cement [8]. The method of surface pretreatment of ceramics before cementation plays a very important role in the success and longevity of ceramic restorations [9].

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http://dx.doi.org/10.1016/j.jpor.2017.06.002
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Please cite this article in press as: H.M. El-Damanhoury, M.D. Gaintantzopoulou, Self-etching ceramic primer versus hydrofluoric acid etching: Etching efficacy and bonding performance, J Prosthodont Res (2017), http://dx.doi.org/10.1016/j.jpor.2017.06.002

The protocol of pretreatment varies from one material to the other; while feldspathic glass ceramics and leucite-reinforced glass ceramics require 60 s of 5–10% hydrofluoric acid etching, lithium disilicate glass ceramics require 20 s only. Aluminum-oxide, zirconia based and composite resin are commonly treated with airborne-particle abrasion before adhesive cementation [10]. The novel hybrid resin-ceramics vary from one brand to the other, as some require hydrofluoric acid etching and others require micro-abrasion according to their composition and method of manufacturing [11,12].

The most common method for pretreatment of glass ceramics is etching with hydrofluoric acid followed by a primer containing organo-silane, where the acid reacts with the glass matrix that contains silica and forms hexafluorosilicates. This glass matrix is selectively removed and the crystalline structure is exposed. As a result, the surface of the ceramic becomes rough, which is expected for micromechanical retention on the ceramic surface [13]. This roughly etched surface also helps to provide more surface energy prior to combining with the silane solution [14].

Alternative methods like, micro-abrasion or air abrasion followed by tribochemical coating of the microblasted surface modified silica, laser etching and non-thermal plasma treatment have been investigated and promoted for pretreatment of metal ceramics [15,16].

A self-etching ceramic primer (Monobond Etch & Prime, Ivoclar Vivadent, Schaan, Liechtenstein) has been introduced to the market as a single-component ceramic primer, alternative to hydrofluoric acid etching/silane coupling agent routine treatment. The novel material 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. Other than the internal data of the manufacturer, there are very few published research work about the newly introduced self-etching ceramic primer and the effect of its use on the bonding efficiency to different types of ceramics. Additionally, very little information is available in the literature about the bonding efficiency of the novel hybrid ceramics to luting resin cements [17]. Therefore, the aim of this study was to assess the effect of using self-etching ceramic primer on the shear bond strength and surface topography of different hybrid and glass ceramics, in comparison to the conventional technique of hydrofluoric acid etching followed by silane application. The null hypothesis was that pretreatment technique will have no significant influence on the surface topography or bonding performance of the CAD/CAM esthetic materials tested to the resin luting cements.

2. Materials and methods

The materials tested and their respective compositions are displayed in Table 1.

2.1. Specimen Preparation

CAD/CAM blocks from each tested material were used and each block was cut transversely using a low-speed diamond wheel saw (Isomet 1000, Buehler, Lake Bluff, IL) under water irrigation to obtain rectangular discs, 2 mm in thickness. After ultrasonic cleaning in a distilled water bath for 15 min, IPS e.max CAD (EM) specimens were fired following the crystallization program recommended by the manufacturer. All specimens were positioned in polyvinyl chloride (PVC) plastic rings and embedded in epoxy resin (Fastray, Harry J. Bosworth Co., Skokie, IL, USA) and wet polished with up to 600- grit silicon carbide paper discs in a semi-automatic polisher/grinder (MetaServ 250, Buehler, Lake Bluff, IL) for one minute. After polishing, the samples were ultrasonically cleaned in a bath of 80% ethyl alcohol for 15 min and dried to remove surface debris. 40 discs from each material were randomly selected and equally divided into 4 groups (n = 10) and assigned to one of the combinations of surface etching and priming methods below:

1. No treatment (NT). This group was used as negative control.
2. Monobond Plus (Ivoclar Vivadent) with no etching. The ceramic primer was applied with a microbrush and allowed to react for 60 s. Subsequently, the excess was dispersed with a strong stream of air to ensure the solvent evaporation (MP). This group was used as positive control.
3. Etching with 4.8% hydrofluoric acid for 60 s for VE and VM and 20 s only for EM. The acid was thoroughly rinsed off with a strong jet of air/water spray for 20 s and dried with oil-free air for 10 s, and application of Monobond Plus (Ivoclar Vivadent) following the same procedures mentioned above (HFMP).
4. Monobond Etch & Prime (Ivoclar Vivadent), a Self-etching glass-ceramic primer was applied on the adhesive surface using a microbrush, agitated into the surface for 20 s then allowed to react for another 40 s, thoroughly rinsed off with a strong jet of air/water spray for 20 s and dried with oil-free air for 10 s (MEP).

A special metal clip was used to fix a Teflon mold (Ultradent Inc, South Jordan, UT), with a cylindrical cavity of 2 mm width and 2 mm depth, to the pre-treated ceramic surface. Dual-cure resin cement (Multilink-N Automix, Ivoclar Vivadent) base and catalyst pastes were mixed using an auto-mixing tip and injected using an ultra-fine tip 1 mm in diameter into the mold. The excess cement was removed using a micro-brush and the luting resin was polymerized using LED light curing unit (Bluephase, Ivoclar Vivadent) operating at 1200 mW/cm^2 in standard mode for 20 s to fabricate cylindrical luting resin rods. The intensity of the curing unit was checked every 10 samples. The mold was disassembled and resultant rods were examined for any composite flashes, which were removed with a sharp blade. Each specimen was examined using magnifying loupes to identify specimens containing possible defects (bubbles or cracks in resin composite or flow of resin cement beyond the limits of the bonding area).

2.2. Shear Bond Strength Testing (SBS)

The samples were stored in distilled water at 37 °C for 24 h and thermos-cycled (TC) between 5 and 55 °C for 5000 cycles with 30-s dwell times before being tested for SBS using a table-top Shear Bond Strength Tester (Bisco Inc., Schaumburg, IL, USA). The semicircular metal attachment of the machine applied shear forces at the resin-ceramic interface, running at a crosshead speed of 1.0 mm/min, till complete failure of the resin composite and debonding. The force required for failure was recorded in Newton and was divided by the surface area (mm^2) to calculate the SBS in MPa.

The debonded specimens were examined under a stereomicroscope to determine the failure mode that was classified as adhesive between resin cement and ceramic (A), mixed (M), cohesive in resin cement (CR), or cohesive in ceramic (CC).

2.3. Surface roughness measurement

Nine ceramic discs for each material tested were prepared for surface roughness measurement and were divided into three subgroups; in the first subgroup specimen were left untreated and was considered as control, the second subgroup was treated with hydrofluoric acid with no application of silane, the last subgroup was treated with MEP following the same protocol mentioned previously. All specimens were washed with double distilled water,

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