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EVALUATION OF MICRO-TENSILE BOND STRENGTH OF TWO TYPES OF RESIN CERAMICS WITH DENTIN AFTER DIFFERENT SURFACE TREATMENTS

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ABSTRACT

Objective: The objective of this study was intended for evaluation the effect of different surface treatments on microtensile bond strength of two different luting resin cements to two CAD/CAM resin ceramic materials with dentin. **Materials and Methods:** A total number of 112 samples were used for microtensile bond strength test (μ-TBS). Samples were divided into two equal main groups according to type of resin ceramic used (Vita Enamic [ENA] and Lava Ultimate [LVU]). Then each main group was subdivided according to the surface treatment performed into four equal subgroups; Gr 1 (sandblasting (SB) with Al_2O_3 50μm), Gr 2(sandblasting (SB) with Al_2O_3 110μm), Gr 3 (plasma etching), and Gr 4 (Hydrofluoric acid (HF) plus Silane), then two types of resin cements (Rely X Ultimate, Panavia f 2.0) were applied to the treated ceramics in each group. μ-TBS test was performed and the data statistically analyzed. **Results:** The results of this study revealed that; LVU recorded statistically significant higher μ-TBS than ENA. Rely X Ultimate recorded statistically significant higher μ-TBS than Panavia F 2.0. **Conclusion:** LVU has higher μ-TBS than ENA with all surface treatments except with HF plus Silane ENA has higher μ-TBS than LVU.

Key Words: Resin ceramic, Resin cement, sandblasting, Plasma, Hydrofluoric acid.

INTRODUCTION

Most computer-aided design-computer-aided milling (CAD/CAM) blocks that are used for the manufacture of indirectly bonded tooth-colored restorations are made of glass ceramics: feldspar ceramic, leucite-reinforced glass ceramic, Lithium disilicate glass and zirconium reinforced lithium silicate glass ceramic [(1)]. Advantages of glass ceramic are their strength and superior optical properties. However, these are stiff, brittle materials with low fracture toughness and high susceptibility to fracture [(2)].

Due to the disadvantages of glass ceramics, the so-called hybrid ceramics and composite blocks with dispersed fillers have been introduced [(3)]. Hybrid ceramics are defined as material consisting of a filtered ceramic substructure with a polymer network. The attractiveness of both materials is based on ease of manufacturing and milling.[(4)] These materials less susceptible to milling errors during the grinding procedure which provides better marginal adaptation [(5)].

A durable bond between the three components of an adhesion complex (tooth tissues, resin cement, ceramic) is a key factor for the long-term clinical success of a restoration. Optimal adhesion is required to obtain a high bond strength. Adhesion of resin cement to resin ceramics is not only improved by acid etching and silanization. Airborne-particle abrasion is an alternative method for roughening the ceramic surface which provide mechanical adhesion. There are some possibilities for improving bonding to hybrid ceramics including modern techniques for surface treatments by plasma technology [(6)].

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The advantages of these resin-based materials are still the subject of research. In particular, the adhesion of these CAD/CAM resin-matrix ceramics to the tooth tissues and/or to the resin cement is of great importance in terms of the clinical success and longevity of an indirect restoration made with these materials.

So, hypothesis of this study was done that there will be an effect of different surface treatments on microtensile bond strength of two different luting resin cements to two CAD/CAM resin ceramic materials with dentin.

MATERIALS AND METHODS

Two types of CAD/CAM resin ceramic materials [Vita Enamic (ENA) and Lava Ultimate (LVU)] and luted to dentin using different resin cements (Rely x Ultimate and Panavia F 2.0) after four different surface treatments.

Samples grouping:

A total number of 112 samples were used for microtensile bond strength test. Samples were divided into two equal main groups according to type of resin ceramic used (LVU and ENA). Then each main group was subdivided according to the surface treatment performed into four equal subgroups; Gr 1 (sandblasting (SB) with Al_2O_3 50μm), Gr 2 (sandblasting (SB) with Al_2O_3 110μm), Gr 3 (plasma etching), and Gr 4 (Hydrofluoric acid (HF) plus Silane). Two types of resin cements (Rely X Ultimate, Panavia f 2.0) were applied to the treated ceramics in each subgroup.

Extracted human molars teeth (n=16) collected and then put in a soft mix of polymethyl-methacrylate (PMMA) resin was poured inside the plastic mold. Firstly, the occlusal enamel of the teeth was removed perpendicular to the long axis of the teeth using an Isomet Saw (Buehler, Lake Bluff, IL, USA) under water cooling to form flat occlusal dentin surfaces then rinsed with water.

Preparation of resin ceramic samples:

Sixthteen samples were prepared (4mm width, 4 mm thickness, 6 mm length) by using an Isomet Saw. All samples were wet polished sequentially with 600, 800, and 1000 (SiC) paper (3M, St. Paul, MN, USA) before surface treatments. Airborne-abrasion was applied evenly to the surfaces of the samples, by spraying (50-μm & 110-μm) Al_2O_3 particles (Quattro IS, Renfert, Hilzinger, Germany) for 20 second from a distance of 10 mm, at pressures of 0.1 MPa [7]. A low vacuum non-thermal-plasma chamber (Femto PCCE Zahntechnik, diener electronic GmbH und Co. KG, Ebhausen, Germany) was used for the plasma treatment. For this purpose, oxygen gas was used as the working gas in the plasma focus system and the condenser bank was charged to 12 kV. The energetic oxygen ion beam took the shape of fountain and spreaded upwards to bombard the facing samples [8]. HF gel 9. 6% (Micerium S.B.A. Via G. Italy) was applied to the surface of the samples for 60 s and rinsed with distilled water for 2 min.

After different surface treatments the adhesive systems were applied to the dentin surfaces according to the manufacturer's specifications. Following the adhesive procedure resin ceramics samples were put on the occlusal direction. After removal of excess cements light polymerized for 20 s. Each tooth was mounted on the cutting machine using an Isomet Saw and sectioned under water cooling to maintain numerous thin micro beam-shaped sticks (approximately 1.0 × 1.0 ×10 mm).

Microtensile bond strength test(µTBS):

Each beam was attached with its ends to a specially designed, modified version of Ciucchi's jig using the cyanoacrylate adhesive (Zapit). The attachment jig consisted of two aluminum articulating parts, one is fixed and the other movable. Then the sample was mounted on universal testing machine (Model 3345; Instron Industrial Products, Norwood, MA, USA). A tensile load with compression mode of force was applied at a crosshead speed of 0.5 mm/min. The applied tensile force resulted in debonding along the substrate-adhesive interface. The load required for debonding of each stick was recorded in MPa (Newton divided by the area). The micro-tensile bond strength δ (MPa) was calculated using the following equation: $\delta = L/A$, where L is the load (N) at failure of the sample and A is the interfacial area of the sample (mm^2) as measured with the digital caliper [9]. After performing the μTBS test, the interfaces were evaluated by a scanning electron microscope (SEM).

Independent-samples t-test of significance was used when comparing between two means. A one-way analysis of variance (ANOVA) when comparing between more than two means. Post Hoc test was used for multiple comparisons between different variables.

RESULTS

Regardless to different surface treatments or type of adhesive material, totally it was noted that, LVU recorded statistically significant higher μ-TBS mean ±SD value (48.871 ± 23.086 MPa) than ENA mean ±SD value (34.456 ± 16.161 MPa) as indicated by ANOVA test followed by pair-wise Tukey's post- hoc test ($P< 0.05$) as shown in figure (1).

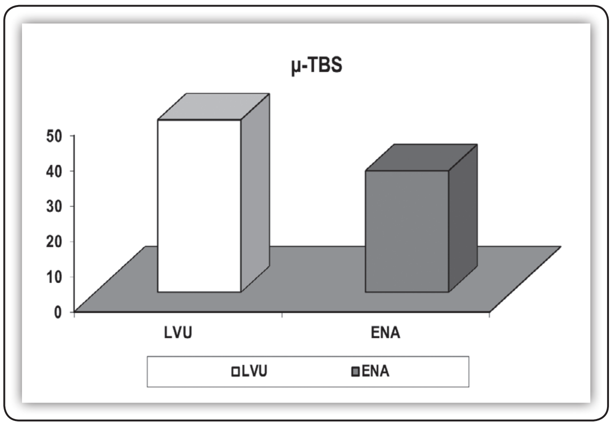

FIG (1) Bar chart of μ-TBS result mean values as function of ceramic material

Regardless to different surface treatments or type of ceramic material, totally it was noted that, Rely X Ultimate recorded statistically significant higher μ-TBS mean ±SD value (49.738 ± 22.04 MPa) than Panavia F 2.0 mean ±SD value (34.130 ± 17.386 MPa) as indicated by ANOVA test followed by pair-wise Tukey's post- hoc test ($P< 0.05$) as shown in figure (2).

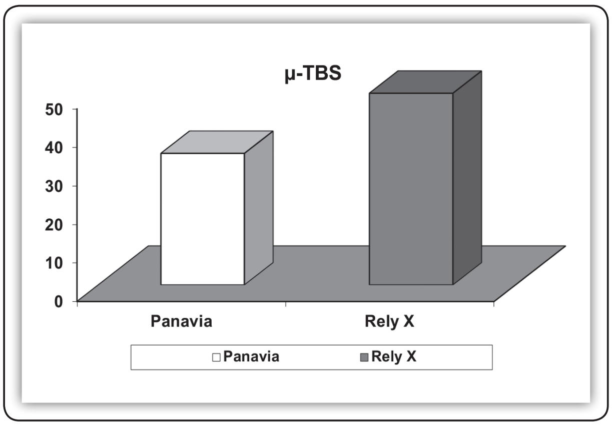

FIG (2) Bar chart of μ-TBS result mean values as function of adhesive material

The comparison between the mean μ-TBS of the two ceramic materials with Panavia F 2.0 and surface treatments are summarized in figure (3).

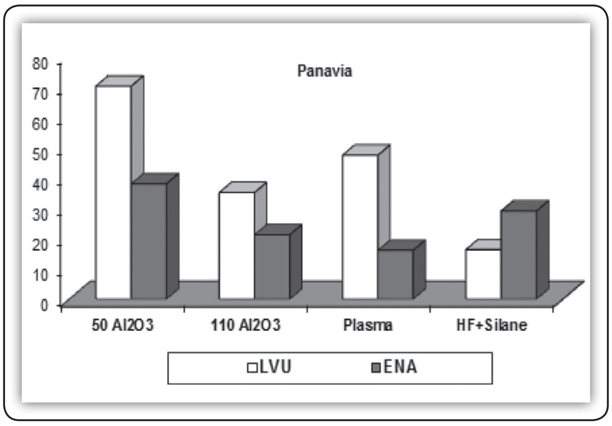

FIG (3) Bar chart of μ-TBS mean values for both resin ceramics materials as function of different surface treatments With Panavia F 2.0

LVU recorded higher μ-TBS mean value than ENA mean value except with HF plus Silane ENA recorded statistically significant higher μ-TBS mean value than LVU. In LVU, 50μm Al_2O_3 surface treatment recorded statistically significant highest μ-Tensile bond strength mean value (70.02014 ± 6.208681 MPa) followed by plasma surface treatment group mean value (47.33423 ± 4.671157MPa), then 110μm Al_2O_3 surface treatment group mean value (35.03201 ± 3.283663 MPa), while With HF plus Silane surface treatment group mean value group recorded statistically significant lowest μ-TBS mean value (16.27604 ± 6.20948 MPa).

In ENA, 50μm Al_2O_3 surface treatment recorded statistically significant highest μ-TBS mean value (37.89665 ± 4.193486 MPa) followed by HF plus Silane treatment group mean value (28.991387 ±2.66909), then 110μm Al_2O_3 surface treatment group mean value (21.15189 ± 1.818538 MPa), while plasma surface treatment group mean value group recorded statistically significant lowest μ-TBS mean value (16.04276 ± 2.586915 MPa) as indicated by ANOVA test ($p < 0.05$) followed by pair-wise Tukey's post- hoc test ($p < 0.05$).

The comparison between the mean μ-TBS of the two ceramic materials with Rely X Ultimate and surface treatments are summarized in figure (4).

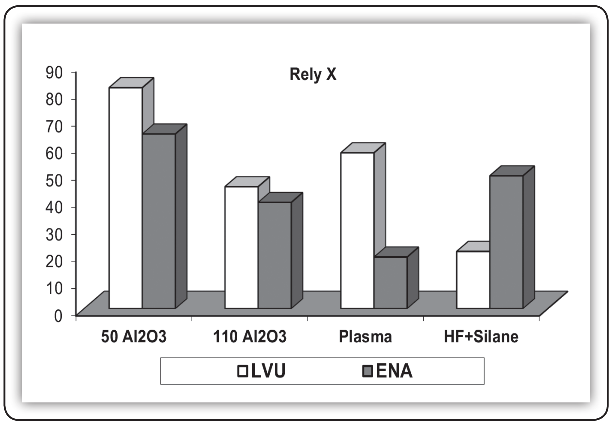

FIG (4) Bar chart of μ-Tensile bond strength mean values for both resin ceramics materials as function of different surface treatments with rely X

Lava Ultimate (LVU) recorded higher μ-TBS mean value than ENA material mean value except with HF plus Silane ENA recorded statistically significant higher μ-TBS mean value than LVU mean value. In LVU 50μm Al_2O_3 surface treatment recorded statistically significant highest μ-TBS mean value (81.56286 ± 8.412996 MPa) followed by plasma surface treatment group mean value (57.47019 ± 2.874582 MPa), then 110μm Al_2O_3 surface treatment group mean value (44.92765 ± 6.124959 MPa), while HF plus Silane surface treatment group mean value group recorded statistically significant lowest μ-TBS mean value (21.16367 ± 3.422152 MPa).

In ENA, 50μm Al_2O_3 surface treatment recorded statistically significant highest μ-TBS mean value (64.43001 ± 5.575096 MPa) followed by HF plus Silane surface treatment group mean value (48.95222 ± 3.363978 MPa), then 110μm Al2O3 surface treatment group mean value (39.10428 ± 4.486505 MPa), while plasma surface treatment group mean value group recorded statistically significant lowest μ-TBS mean value (19.08342 ± 4.475855 MPa) as indicated by ANOVA test ($p < 0.05$) followed by pair-wise Tukey's post- hoc test ($p < 0.05$).

Figure (5) represent the SEM images of the LVU and ENA resin-matrix ceramics after cementation with Relay X Ultimate and Panavia F2.0 with dentin which revealed that the interface analysis with air abrasion 50 μm Al_2O_3; show surface shallow and uniform surface irregularities. Also, show complete penetration of the unfilled resin into ceramic irregularities. But with air abrasion 110 μm Al_2O_3; show highly surface irregularities (deep &non uniform) and show surface microcracks. Also, show incomplete interaction between ceramic and resin cement (slight gab).

SEM interface analysis of LVU with plasma ttt; show little surface irregularities and show complete interaction between ceramic and resin cement. But with HF plus Silane show little surface irregularities (not deep & uniform). White arrow indicated an incomplete interaction between ceramic and resin cement (gab).But interface analysis of ENA with plasma ttt show little surface irregularities (not deep &

uniform) and show incomplete interaction between ceramic and resin cement. Continuity of the cement infiltration along the interfaces was not uniform (slight gab). But with HF plus Silane show little surface irregularities of ENA and complete interaction between ceramic and resin cement (uniform).

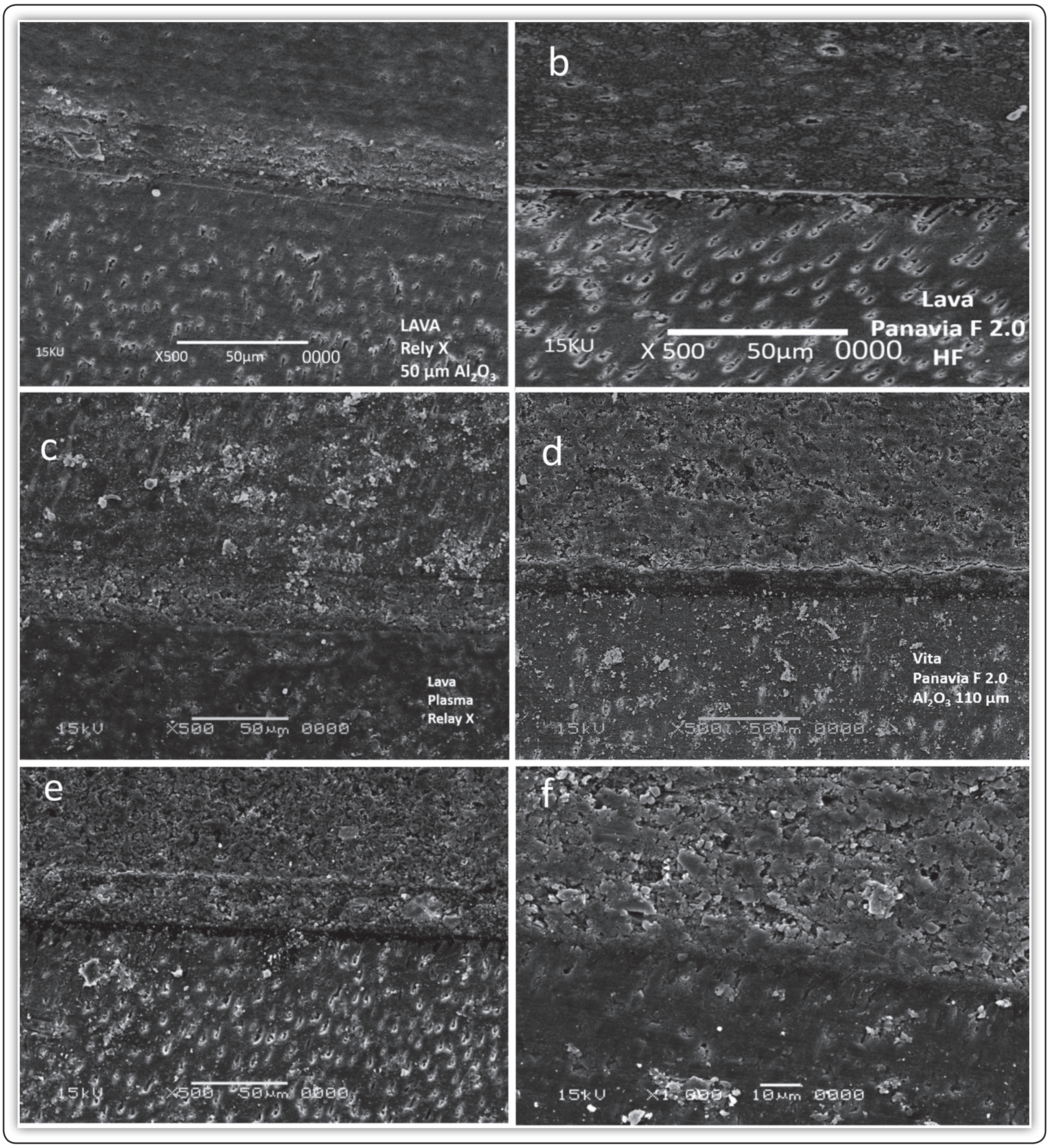

FIG (5) SEM images of LVU; (a) with Al_2O_3 50 μm and Rely X Ultimate, (b) With HF plus silane and Panavia F 2.0. (c) with plasma and Rely X Ultimate, Also, A SEM images of ENA; (d) with Al_2O_3 110 μm and Panavia F 2.0, (e) with plasma and Panavia F 2.0, (f) With HF plus silane and Rely X Ultimate

DISCUSSION

Recently, new resin ceramics CAD/CAM blocks were introduced to the dental field, composed of two matrices: a polymer and a ceramic networks[10]. This dual network structure reduced surface hardness of the material allowing easier milling in a shorter time[11].

The μTBS method was chosen as it provides a more uniform and homogeneous stress distribution during loading, and failure predominantly occurs at the adhesive interface due to the small bonded interfaces (approximately 1 mm^2) of the specimens used [12].

The comparison between the mean μ-TBS of the two ceramic materials LVU obtained significant higher μTBS values than ENA. The differences in the moduli of elasticity among the materials could play a role in the bond strength results. The modulus of elasticity of ENA higher than LVU Moreover, LVU have similar modulus of elasticity to dentin and resin cements. This agreement with Bellan et al [13] whose found that the more modulus of elasticity of material (ENA) tend to start the fracture at the adhesive interface at lower values than the more resilient materials (LVU), which could explain the lower μTBS values of ENA.

Also, this may be due to phosphate ester group (MDP) that found in both resin cements could bond directly to zirconium oxides that found in LVU, thus creating chemical bonds between the resin composite cements and zirconia.[14] This is in disagreement with Dadjoo et al [15] whose found that LVU contain zirconia which has a low surface energy, limiting adhesiveness following cementation, even when resin cements are used, so the surface should be treated with acid or airborne-particle abrasion plus primer application.

Rely X Ultimate obtained significant higher μTBS values than Panavia F 2.0. During manipulation we noticed that rely X had lower viscosity than Panavia, lower viscosity of the adhesive is required to allow its easy flow on the surface of the ceramic to provide good wettability and adhesion. Some authors describe the poor bonding performance of Panavia to a polymerization inhibition effect exerted by acidic monomers present on the ED Primer formulation [16]. According to Salza et al [17] the slow polymerization of this cement is related to an incompatibility of the peroxides with the acidic components of the luting cement paste.

In the present study 50 μm Al_2O_3 surface treatment showed significant higher micro tensile bond strength than 110 μm Al_2O_3. This results are supported by SEM photomicrographs that showed microcracks at the surface between resin ceramic and resin cements. Air abrasion enhanced the surface energy through creating surface irregularities and mechanical interlocking. But aggressive sandblasting by 110 μm Al_2O_3 does not seem to be the best surface treatment for etchable ceramics, since it may cause microcracks in the ceramic surface, which may lead to premature failures [18]. This is in agreement with Addison et al [19] who reported that aggressive air abrasion generates critical-size flaws and defects, which may subsequently affect the clinical viability of resin ceramic restorations.

Nonthermal oxygen plasma was found to increase the bond strength to a similar extent to that produced by the standard treatment with airborne-particle abrasion with LVU, but without altering surface properties, thus reducing the risk of fissures and cracks. Plasma treatments are capable of enhancing the surface energy by mild etching and chemical functionalization, without changing the bulk properties of the substrates. It is known that the gas of the plasma device promotes the formation of active peroxide radicals (R–O–O–), and incorporates additional functional groups (C–O, C–OH) into the upper layer of the treated surface, which initiate chemical surface element as zirconia [20]. This is confirmed by Lopes et al [21] whose reported the same findings for ceramics containing zirconia.

These results were supported by SEM observations which showed little surface irregularities (not deep & uniform) of LVU when treated with plasma but show complete interaction between ceramic and resin cement.

The effect of HF plus Silane on the bonding surface of ENA CAD/CAM restorative material evidently provided significant higher bond strength than LVU. Surface treatment with HF modifies the microstructure of the treated surface by partial dissolution of the glassy phases of the restorative material, thus confirmed by Elsaka et al[12] whose revealed that HF acid forms micro porosities and enhances the establishment of mechanical interlocking with luting resin. Also, Silane is a bifunctional molecule that hydrolyzes to form silanol (-SiOH) groups. These groups can react with silica available on the surface of ENA to form siloxane (-Si–O–Si–O-) networks [22].

This results supported by SEM observation which showed interface between ceramic and resin cement. Continuity of the cement infiltration along the interfaces was more uniform for ENA when treated with HF plus Silane than LVU which show incomplete interaction between ceramic and resin cement gap.

The hypothesis of this study was that there will be a different in surface treatments on microtensile bond strength of two different luting resin cements to two CAD/CAM resin ceramic materials with dentin was accepted.

CONCLUSION

Within the limitations of this study the following conclusions might be drawn:

1. Lava Ultimate has higher μ-TBS than Vita Enamic with all surface treatments except with HF plus Silane Vita Enamic has higher μ-TBS than Lava Ultimate.
2. Sandblasting with 50μm Al_2O_3 particles have higher μ-TBS than other surface treatments.

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