

Comparison of the Effect of Surface Conditioning Methods on the Bond Strength of Different Zirconia Reinforced Lithium Silicate and Hybrid Ceramics to Resin Cement

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ABSTRACT

Objective: The objective of this study is to investigate the effect of surface treatment techniques on the shear bond strength (SBS) of resin cement bonded to CAD / CAM materials.

Methods: The zirconia-reinforced lithium silicate, Vita Suprinity (VS) and Celtra Duo (CD), and hybrid ceramics, Vita Enamic (VE) and Nacera Hybrid (NH), were used. Eighty specimens from each material were fabricated following the manufacturer's instructions and separated into 8 groups according to surface treatments. These were; K (control, no treatments), H (hydrofluoric acid), HS (H+ Silane S), A (abraded with 50-micron alumina particles A), AS (A+ S), C (abraded with 30-micron Cojet sand C), CS (C+S), S. The mean surface roughness (SR) of the specimens was evaluated. Surface treated specimens were cemented to the resin cement (Panavia F 2.0) for testing the adhesion using the shear bond strength (SBS) test. Mean SR and SBS were evaluated by 2-way ANOVA with the material type and surface treatments techniques as the independent factors.

Results: A and AS groups were observed to have the highest SR values. Both hybrid ceramics VE and NH showed the highest SR values among surface treatments. The highest SBS values were found usually on the H and HS treated surfaces. The highest values were observed on the CD material in the HS group (18.01MPa) and followed by the VE material (16.25 MPa) in the CS group. For CD and VS materials, adhesive and cohesive failures were found; and for VE and NH materials, adhesive and mixed cohesive failures were observed.

Conclusion: The surface treatment showed a significant effect on SR and SBS values. Although the SR values of the materials are high in the A and AS group, the highest SBS values were observed on H and HS treated surfaces.

Keywords: Shear Bond Strength, Surface Roughness, Hybrid Ceramic, Zirconia Reinforced Lithium Silicate Ceramic.

1. INTRODUCTION

Ceramics and resin-based composites are two different types of dental materials (1). Ceramics have good mechanical and optical properties and excellent biocompatibility due to their chemical stability (2). New ceramic materials in dentistry are increasing as stronger and tougher materials are developed with contemporary manufacturing techniques (3, 4). These include monolithic zirconia, zirconia reinforced lithium silicate ceramics and hybrid ceramics (3, 5, 6). Monolithic restorations have significant advantages such as decreased manufacturing time, improved cost effectiveness and elimination of the interface between the core and veneer materials (7, 8). Recently various monolithic glass ceramic materials also come into use currently with CAD/CAM systems for the application of fixed prosthesis (5, 9). This new zirconia reinforced lithium silicate glass ceramics are enriched lithium silicate glass ceramics (10% by weight) and combine the favorable material properties of zirconia (ZrO₂) and glass ceramic. The zirconia particles were added to reinforce the ceramic structure. (9, 10, 11, 12).

Hybrid ceramics introduced to the market to achieve a material with elastic modulus comparable to dentin have a number of advantages, including less crack propagation and better fracture resistance than few CAD / CAM systems (1, 6, 11, 13, 14, 15). Hybrid ceramics are composites of nanofiller and resins (6, 11, 16, 17). These materials composed of an organic polymer matrix reinforced by inorganic filler particles consist of porcelain glasses and ceramics (6).

The fracture resistance of ceramic materials can also be reinforced by the properties of the support materials or the bond strength. It was described that well-cemented specimens were generally more fracture resistant (13). For long term success, adhesion to a ceramic material is the one of the most important properties to evaluate the bond durability (14, 19). Another key factor for the clinical success of fixed prosthesis is the cementation procedure (20, 21). Luting cements and agents link prosthetic restorations with the supporting tooth structure and interfacial surface defects (13, 19, 20, 21). Retention loss

is the second reason why traditional fixed prostheses fail (21). Usually, ceramic restorations are pretreated physically or chemically, but are more likely made by a combination of both methods (22). The strength of the adhesion between the ceramic and the bonding agent determines the clinical success of a ceramic restoration. Also, clinical success partially depends on the adhesion techniques controlled by the surface treatments used (23). Various conditioning methods for ceramic surface pretreatment such as roughening the surface with a diamond bur, sandblasting with alumina or silica-coated alumina oxide particles, chemical etching with hydrofluoric acid (HF) are recommended to increase adhesion (4, 20, 23)

Acid abrasion resistant ceramics require some special surface treatment to optimize bonding to the resin-based composite. The most common surface treatments for this purpose are airborne particle abrasion and silica coating. (22). Air particle abrasion is also used to increase the roughness of the ceramic. Surface roughness (SR) allows interlocking between ceramic and resin cement. (24, 25). Ceramics with high glass content form a micro mechanical retention surface as a result of the effect of HF, and these ceramics are called acid-sensitive (12). During HF etching, some parts of the silicate ceramic surface are removed and a surface roughness occurs (4, 22). Application of silane coating agent is a chemical approach to bonding to ceramic and provides a chemical connection between ceramic and resin composite (4, 12, 22). These agents can create chemical connections between the inorganic phase of the ceramic and the organic phase of the resin (26, 27).

Fracture resistance of the ceramic-resin adhesion is controlled by the microstructure and surface treatment (28). Mechanical

laboratory tests can be used to show material selection and clinical recommendations for resin bonding to ceramics. (4, 19). Various methods can be used for the assessment of the bond strength: 3-point bending, the tensile and micro tensile and the shear and micro shear tests (4, 19, 28). The most common method is the shear bond strength (SBS) test (4, 17, 28, 29). The advantages of the shear bond strength (SBS) test performed by applying parallel force to the binding interface are easy specimen preparation and simple test protocol. (14, 16, 17, 19, 30, 31). Nevertheless, non-uniform stress distribution in the bonding surface and polymerization shrinkage of resin cement are not considered in this technique (28, 29, 30).

The objective of this study is to evaluate the effect of different surface treatments on surface roughness and shear bond strength of resin cement bonded to different ceramic materials. The null hypothesis in this study is that surface roughness and SBS are not affected by material variety and surface treatment technique.

2. METHODS

Two zirconia reinforced lithium silicate ceramics, Vita Suprinity (VS) and Celtra Duo (CD) and two hybrid ceramics, Vita Enamic (VE) and Nacera Hybrid (NH) were selected for the study. The materials, their manufacturers and compositions used in this study are shown in Table 1. All specimens (10 x 10 x 1 mm) in this study were prepared from prefabricated blocks. 320 specimens were separated into 8 subgroups (n = 10) for various surface treatments to be applied. The schematic diagram of the experimental groups is shown in Figure 1.

Table 1. Composition of the materials tested in this study.

| Material | Brand | Manufacturer | Lot No | Composition |
|--------------------------------------|-------------------------|--|-------------|---|
| Zirconia-reinforced Lithium Silicate | Celtra Duo CD | Sirona Dentsply, Milford, De, USA | 16000579 | SiO ₂ , P ₂ O ₅ , Al ₂ O ₃ , Li ₂ O, ZnO ₂ , Tb ₄ O ₇ , ZrO ₂ , CeO ₂ , pigments |
| Zirconia-reinforced Lithium Silicate | Vita VS Suprinity | Vita Zahnfabrick, Bad Säckingen, Germany | 59841 | ZrO ₂ , SiO ₂ , Li ₂ O, La ₂ O ₃ , P ₂ O ₅ , K ₂ O, Al ₂ O ₃ , La ₂ O ₃ , CeO ₂ , pigments |
| Hybrid Ceramic | Vita VE Enamic | Vita Zahnfabrik, Bad Säckingen, Germany | 63460 | SiO ₂ , Al ₂ O ₃ , Na ₂ O, K ₂ O, B ₂ O ₃ , CaO, TiO ₂ , UDMA, TEGDMA |
| Hybrid Ceramic | Nacera NH Hybrid | Doceram Medical Ceramics GmbH, Dortmund, Germany | 230516 | 50% Nano-Glass, 50% Polymer-Matrix |
| Hydrofluoric Acid | IPS Ceramic Etching Gel | Ivoclar Vivadent, Schaan, Lichtenstein | W31655 | <%5 Hydrofluoric Acid |
| Aluminium Oxide Sand | Korox | Bego, Bremen, Germany | 14361781112 | %99.6 Al ₂ O ₃ (50µm) |
| Cojet Sand | Cojet Sand | 3M ESPE, St. Paul, USA | 654604 | 30 µm silica coated sand |
| Silane Coupling Agent | Monobond N Silane | Ivoclar Vivadent, Schaan, Lichtenstein | W90335 | Alcohol based silane methacrylate, Phosphoric Acid Methacrylate, Sulfur Methacrylate Solution |
| Dual Cure Resin Cement | Panavia F 2.0 | Kuraray Medical Inc., Okayama, Japan | B70150 | Paste A: MDP, hydrophobic aromatic and aliphatic photoinitiator, dibenzoyl peroxide dimethacrylate, hydrophilic dimethacrylate, silanized silica, silanized colloidal silica, camphorquinone, catalysts, initiators |
| | | | AR0034 | Paste B: Hydrophobic aromatic and aliphatic dimethacrylate, silanized barium glass, sodium fluoride, catalysts, initiators, color pigments. |

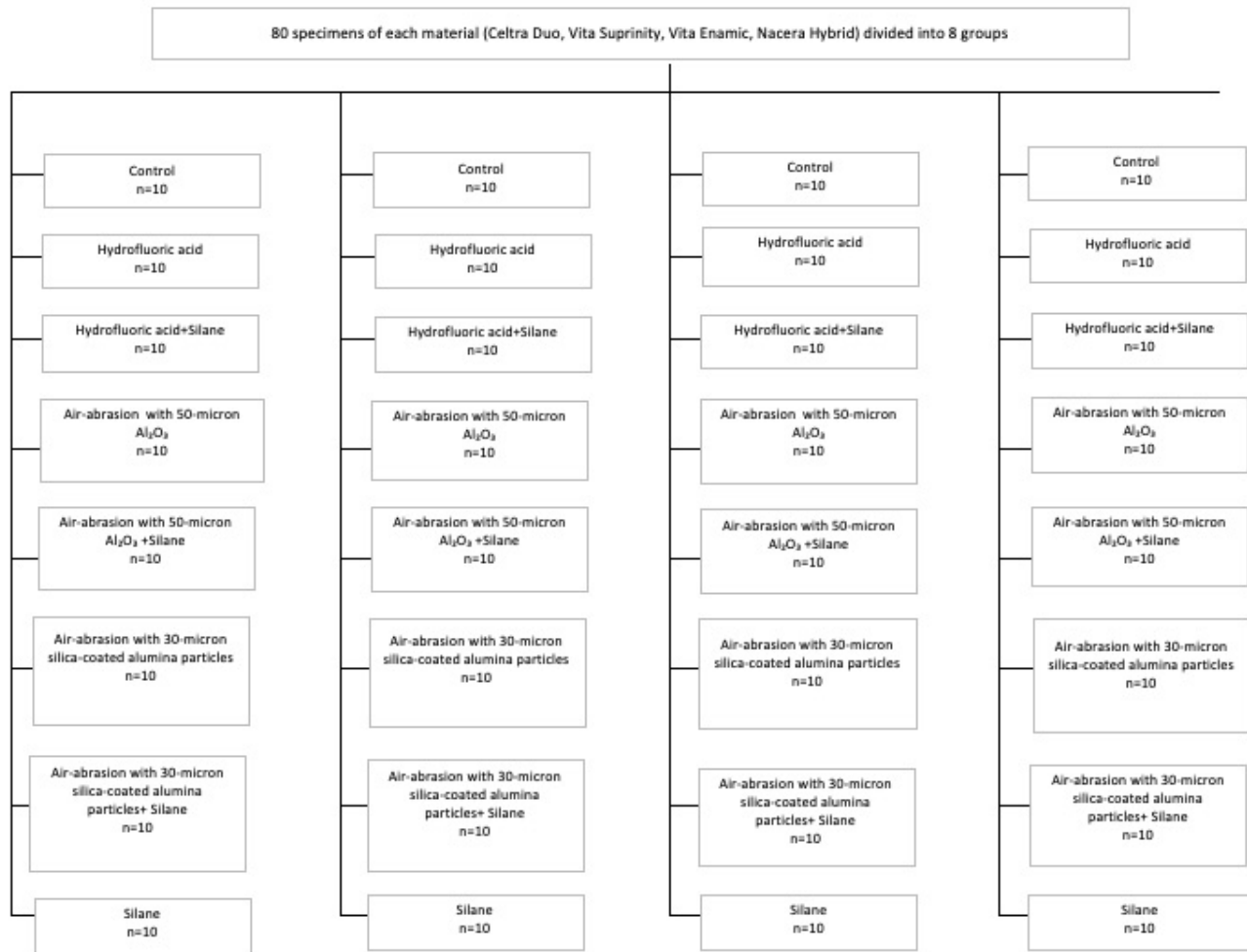


Figure 1. Schematic diagram of experimental groups.

All specimens were put into acrylic resin blocks and then polished under water cooling to obtain standardized surfaces. The specimens were separated into 8 groups according to the used surface treatments.

K: No treatment (control)

H: %5 Hydrofluoric acid application for 20 seconds

HS: %5 HF acid for 20 seconds + silane application for 60 seconds.

A: Air-abrasion with 50-micron Al_2O_3 at 2-5 bar pressure.

AS: Air-abrasion with 50-micron particles of alumina Al_2O_3 at 2.5 bar pressure + silane application for 60 seconds.

C: Air-abrasion with 30-micron silica-coated alumina particles with 2.5 bar pressure.

CS: Air-abrasion with 30-micron silica-coated alumina particles with 2.5 bar pressure + silane application for 60 seconds.

S: Silane application for 60 seconds.

All specimens were cleaned with ethanol and distilled water for 10 minutes in an ultrasonic cleaner after all surface treatments. A surface roughness (SR) profile was determined for each group using a profilometer (Mahr Surf M 300c,

Mahr GmbH, Germany). Three readings were taken from the surface of the specimens and a mean value was calculated.

After SR measurements, a dual-cure resin cement (Panavia F 2.0, Kuraray Medical Inc, Japan) was packed onto specimen surfaces using a mold which was 3 mm in diameter and 2 mm in depth cylindrical. Layers were added incrementally and cured using a light-curing unit (BA Optima 10 Boses 20, BA International Ltd. England). The application of cement in the specimen is shown in Figure 2. Then all specimens were kept in 37°C distilled water for 24 hours.

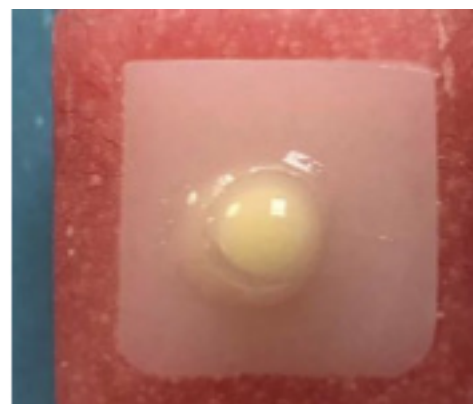


Figure 2. The application of dual-cure resin cement on the specimen.

The SBS was determined with a universal testing machine (Lloyd Instruments, Ametek Inc. Florida USA). SBS test with specimens is shown in Figure 3. The specimens were loaded at a crosshead speed of 0.5 mm/min. The maximum load (P) was measured when the resin cement was separated from the specimen. The SBS was measured from the formula below:



Figure 3. The specimen under the SBS test in the universal testing machine.

$$SBS \text{ (in MPa): } P \times 9.8 / r^2 \times \pi,$$

where P is the maximum load (in kg F) and r is the radius (in mm) of the resin cement.

After the SBS test, the interface of the specimens was analyzed using a loupe (Loupe opt-on Orange Dental, Biberach Germany) at 2.5 mm magnification. The fracture modes were classified as follows:

Type 1: Adhesive (between the surface of the both materials),

Type 2: Cohesive (within the ceramic material),

Type 3: Cohesive (failure within the material and resin cement) failure.

Statistical analyses were applied to the surface roughness and SBS values. Mean surface roughness and SBS results analyzed 2-way ANOVA with the material type and surface treatments as the independent variables. Multiple comparisons were made by Tukey's and Tamhane's tests. Statistical significance was set at the 0.05 probability level. The correlation between SR and SBS results was investigated using Pearson correlation and chi-squared analysis.

3. RESULTS

3.1. Surface Roughness

Multiple comparisons of the mean SR values of the materials used in this study and surface treatment methods were made with Tukey's and Tamhane's test.

Table 2. Mean and standard deviation (SD) of the surface roughness (Ra) values for the materials used in this study.

| Materials | Surface Treatments | Ra Values | | | One-Way ANOVA | |
|-----------|--------------------|-------------------|---------------------|------|---------------|-------|
| | | n | Mean | SD | F | p |
| CD | K | 10 | 0.19 ^d | 0.06 | 116.58 | 0.001 |
| | H | 10 | 0.35 ^c | 0.08 | | |
| | HS | 10 | 0.37 ^c | 0.11 | | |
| | A | 10 | 1.65 ^a | 0.26 | | |
| | AS | 10 | 1.45 ^{a,e} | 0.34 | | |
| | C | 10 | 1.15 ^{b,e} | 0.11 | | |
| | CS | 10 | 0.96 ^b | 0.14 | | |
| S | 10 | 0.18 ^d | 0.04 | | | |
| VS | K | 10 | 0.18 ^c | 0.05 | 125.152 | 0.001 |
| | H | 10 | 0.2 ^c | 0.07 | | |
| | HS | 10 | 0.15 ^{c,d} | 0.07 | | |
| | A | 10 | 1.5 ^a | 0.31 | | |
| | AS | 10 | 1.39 ^a | 0.29 | | |
| | C | 10 | 0.92 ^b | 0.13 | | |
| | CS | 10 | 0.95 ^b | 0.1 | | |
| S | 10 | 0.09 ^d | 0.03 | | | |
| VE | K | 10 | 0.18 ^d | 0.04 | 170.055 | 0.001 |
| | H | 10 | 0.39 ^c | 0.05 | | |
| | HS | 10 | 0.37 ^c | 0.04 | | |
| | A | 10 | 1.82 ^a | 0.19 | | |
| | AS | 10 | 1.79 ^{a,e} | 0.38 | | |
| | C | 10 | 1.21 ^b | 0.17 | | |
| | CS | 10 | 1.31 ^{b,e} | 0.13 | | |
| S | 10 | 0.18 ^d | 0.06 | | | |
| NH | K | 10 | 0.23 ^c | 0.08 | 384.525 | 0.001 |
| | H | 10 | 0.22 ^c | 0.07 | | |
| | HS | 10 | 0.19 ^c | 0.04 | | |
| | A | 10 | 2.18 ^a | 0.24 | | |
| | AS | 10 | 1.96 ^a | 0.2 | | |
| | C | 10 | 1.43 ^b | 0.11 | | |
| | CS | 10 | 1.34 ^b | 0.18 | | |
| S | 10 | 0.21 ^c | 0.05 | | | |

*Tukey HSD test, Tamhane's test. There is no statistically significant difference between the mean Ra values of the groups with common lowercase letters (p > 0.05)

The mean SR values of the materials used and surface treatments are presented in Table 2. The SR was affected significantly differently from the treatments for all materials, and the effect of surface treatment methods was evaluated for each material. Within all materials, the highest SR values were observed in the A and then AS group and the value difference between the two groups was not significant ($p>0.05$). The Mean values of A groups were; 1.65±0.26 MPa for CD, 1.5±0.31 MPa for VS, 1.82±0.19 MPa for VE, 2.18±0.24 MPa for NH. The mean values of AS groups were; 1.45±0.34 MPa for CD, 1.39±0.29 MPa for VS, 1.79±0.38 MPa for VE, 1.96±0.2 MPa for NH. Then, C and CS groups significantly followed these two groups. The lowest Ra values were found generally in K and S groups for all materials. Among the surface treatment groups, the highest Ra values were observed in the A group on NH material, in the C group on the VE material, and in AS and CS groups on NH and VE materials. Then, the lowest Ra values were obtained in H and HS groups in NH and VS materials.

3.2. Shear Bond Strength

Multiple comparisons of the mean SBS values and surface treatment methods were made with Tukey’s tests. The mean shear bond strength test values of the materials used and surface treatments are presented in Table 3. The SBS test values were significantly affected by surface treatment methods for all materials and the effect of the treatments was evaluated for each material. Within all the materials used, the highest SBS test values were observed in H and HS groups on CD, VS, NH materials and in the CS group on the VE material (16.25±5.83 MPa). CD material mean values are; 11.58±2.53 MPa for H group, 18.01±4.07 MPa for HS group. VS material mean values are; 18.84±4.37 MPa for H group, 11.60±3.09 MPa for HS group. NH material mean values are; 12.81±4.03 MPa for H group, 12.44±3.43 MPa for HS group. The lowest SBS values were generally found in the control group. Among the surface treatment groups, the highest SBS values were observed in C and CS groups on the VE material, and in A and AS groups on VE and VS materials. The lowest SBS test value was found in the K group at the CD material.

After the SBS test, the interface of the fracture specimens was analyzed and the fracture mode was classified as presented in Table 4. The failure type on the surfaces of all used materials differed according the material type and the surface treatments. Among the tested materials, when all surface treatments were evaluated, the Type 1 highest adhesive failure was observed in NH and then CD material, Type 2 cohesive failure was seen in VS and then CD materials

and Type 3 cohesive failure was primarily seen in VE material. Type 3 cohesive failure was not observed in CD and VS materials. Among the surface treatments, when all tested materials were evaluated, the highest Type 1 adhesive failure was observed in K and S treatment groups, Type 2 cohesive failure was seen in CS and then HS groups and Type 3 cohesive failure was observed primarily in HS treatment group. Type 3 cohesive failure was not observed in K and S groups.

Table 3. Mean and standart deviation (SD) of the Shear Bond Strength (SBS) (MPa) values of the materials used.

| Materials | Surface Treatments | SBS Values | | | One-Way ANOVA | |
|-----------|--------------------|------------|----------------------|------|---------------|-------|
| | | n | Mean | SD | F | p |
| CD | K | 10 | 2.45 ^e | 1.56 | 33.924 | 0.001 |
| | H | 10 | 11.58 ^{b,d} | 2.53 | | |
| | HS | 10 | 18.01 ^a | 4.07 | | |
| | A | 10 | 6.81 ^c | 2.66 | | |
| | AS | 10 | 8.55 ^{c,d} | 3.27 | | |
| | C | 10 | 5.37 ^c | 1.68 | | |
| | CS | 10 | 5.37 ^c | 2.64 | | |
| | S | 10 | 5.36 ^c | 1.74 | | |
| VS | K | 10 | 3.43 ^{c,d} | 1.97 | 11.709 | 0.001 |
| | H | 10 | 12.84 ^a | 4.37 | | |
| | HS | 10 | 11.6 ^a | 3.09 | | |
| | A | 10 | 8.7 ^{a,b} | 3.04 | | |
| | AS | 10 | 7.15 ^{b,c} | 4.15 | | |
| | C | 10 | 5.62 ^{b,c} | 2.31 | | |
| | CS | 10 | 5.99 ^{b,d} | 2.54 | | |
| | S | 10 | 4.3 ^{c,d} | 2.6 | | |
| VE | K | 10 | 8.87 ^b | 5.25 | 2.461 | 0.025 |
| | H | 10 | 13.17 ^{a,b} | 1.9 | | |
| | HS | 10 | 14.5 ^{a,b} | 3.33 | | |
| | A | 10 | 12.65 ^{a,b} | 5.22 | | |
| | AS | 10 | 11.25 ^{a,b} | 4.64 | | |
| | C | 10 | 13.99 ^{a,b} | 4.69 | | |
| | CS | 10 | 16.25 ^a | 5.83 | | |
| | S | 10 | 10.59 ^{a,b} | 5.82 | | |
| NH | K | 10 | 5.53 ^b | 1.37 | 7.506 | 0.001 |
| | H | 10 | 12.81 ^a | 4.03 | | |
| | HS | 10 | 12.44 ^a | 3.43 | | |
| | A | 10 | 5.76 ^b | 2.08 | | |
| | AS | 10 | 8.24 ^{a,b} | 3.3 | | |
| | C | 10 | 8.23 ^{a,b} | 2.41 | | |
| | CS | 10 | 8.26 ^{a,b} | 3.64 | | |
| | S | 10 | 8.69 ^{a,b} | 3.49 | | |

*Tukey HSD test, there is no statistically significant difference between the mean SBS values of the groups with common lowercase letters ($p>0.05$).

Table 4. Failure modes of the experimental groups

| | | FAILURE MODE | | | | | | | | Chi Square Test | |
|--------------------|-------|--------------|-------|--------|-------|--------|-------|-------|------|-----------------|-------|
| | | Type 1 | | Type 2 | | Type 3 | | Total | | Chi Square | p |
| | | n | % | n | % | N | % | n | % | | |
| Materials | CD | 51 | 26.29 | 29 | 29.9 | 0 | 0 | 80 | 25 | 94.296 | 0.001 |
| | VS | 47 | 24.23 | 33 | 34.02 | 0 | 0 | 80 | 25 | | |
| | VE | 43 | 22.16 | 9 | 9.28 | 28 | 96.55 | 80 | 25 | | |
| | NH | 53 | 27.32 | 26 | 26.8 | 1 | 3.45 | 80 | 25 | | |
| | Total | 194 | 100 | 97 | 100 | 29 | 100 | 320 | 100 | | |
| Surface Treatments | K | 38 | 19.59 | 2 | 2.06 | 0 | 0 | 40 | 12.5 | * | 0.001 |
| | H | 27 | 13.92 | 12 | 12.37 | 1 | 3.45 | 40 | 12.5 | | |
| | HS | 12 | 6.19 | 20 | 20.62 | 8 | 27.59 | 40 | 12.5 | | |
| | A | 25 | 12.89 | 12 | 12.37 | 3 | 10.34 | 40 | 12.5 | | |
| | AS | 20 | 10.31 | 15 | 15.46 | 5 | 17.24 | 40 | 12.5 | | |
| | C | 21 | 10.82 | 13 | 13.4 | 6 | 20.69 | 40 | 12.5 | | |
| | CS | 13 | 6.7 | 21 | 21.65 | 6 | 20.69 | 40 | 12.5 | | |
| | S | 38 | 19.59 | 2 | 2.06 | 0 | 0 | 40 | 12.5 | | |
| Total | 194 | 100 | 97 | 100 | 29 | 100 | 320 | 100 | | | |

*Chi-Square Analysis, Chi-square analysis was performed with the Monte Carlo Simulation since 20% of the expected value in cells is less than 5.

4. DISCUSSION

This in vitro study evaluated the effect of different surface treatments on the SR and SBS of resin cement to/on? different CAD/CAM ceramic materials and demonstrated that the surface treatment methods have an influence on SR and SBS test values of the materials used and that these surface treatment methods significantly affected the results of this study. Consequently, the results of this study reject both null hypotheses.

Lithium-silicate based glass ceramics and hybrid ceramics have been recently used as materials for CAD/CAM techniques (5, 6). The newest generation glass ceramics VS and CD contains 10%wt. highly dispersed zirconia (9, 11). One of the two hybrid ceramics used in this study is a ceramic network consisting of approximately 14% resin embedded in 86% of a ceramic network VE material (15, 21, 26, 33). Another new CAD/CAM hybrid ceramic is the NH material for permanent restorations and contains 50% nano-glass and 50% polymer-matrix. This new hybrid ceramic has been introduced for manufacturing partial crowns, veneers and up to 3 units bridges (34).

To evaluate the adhesion of resin cement, tensile and micro tensile bond strength, pull and push tests are the other test methods. Sano et al. created the micro tensile bond strength test in order to eliminate the non-uniform stress distribution within the adhesive zone (35). However, the micro tensile bond strength test method is difficult to conduct, time consuming and highly technique sensitive because of the specimen preparation (28). The SBS test is simple and reliable. Hu et al. compared the difference between the two SBS tests for resin composite cements and concluded that the shear test is reliable to assess differences in bonding performance as long as shearing occurs at the interface with no fracture of the substrate (29). Therefore, the SBS test was used in the present study.

Achieving a chemical adhesion at the cement/ceramic interface may be essential for successful full bonds. Cement selection is a precondition for ensuring effective bond strength to indirect restorations. It has been found that bond strength is more effective when using dual-cured resin cement in indirect restorations (32). In the present study, only one resin cement was used to ensure standardization.

Various surface treatment techniques are preferred depending on the characteristics of the material (32, 37). Elsaka SE has evaluated and confirmed the effect of surface treatments applied to CAD / CAM materials on micro tensile bond strength to resin cement (32). Similar results have also been reported for glass ceramics (26). Also, Kim et al. stated that the bonding strength varies between different types of materials even when implementing the same surface treatment method (24). In this study, different surface treatments were applied to different CAD/CAM material surfaces and SR and SBS values of the treated surfaces were evaluated. An important requirement for the clinically successful function of ceramic indirect restorations is adequate adhesion between the ceramic and tooth structure and the surface treatment prior to cementation could enhance the bond strength (38). Common physical surface treatment methods are roughening with a diamond bur, airborne particle abrasion with alumina or silica and etching with hydrofluoric acid (HF) (4). During HF etching, parts of the silicate ceramic surface are etched and result in the surface roughness. The HF etching time is important for adequate mechanical retention (4, 20). Della Bona et al. suggested that etching mechanisms change according to the type of the etchant and etching time and the ceramic microstructure and composition (39). In another study, Menees et al. found that HF etching for 20 seconds in concentrations varying from 5% and 9.5% is enough for etching. (40). In another study, Sato et al. concluded that etching with HF acid for 20 and 40 seconds was equally effective in producing stable resin bonding to a

zirconia reinforced lithium silicate ceramic (12). Therefore, 5% HF acid was applied for 20 seconds in the present study.

Airborne abrasion using alumina or silica particles is commonly recommended for luting resin composites to CAD/CAM blocks (4, 21, 25). The air abrasion system ensures air-particle abrasion with different particle sizes ranging from 30 to 250 micron between the ceramic and cement (41, 42). Generally, sand blasting pressure is recommended as 0.1-0.2 MPa. However, this pressure is lower than the pressure commonly used for ceramic restorations (25). In the present study, the surfaces were sandblasted in the air abrasion groups at 2.5 bars. The SR of the material was evaluated with a surface profilometer. In this study, Ra, which is the mean value of all absolute distances of the linear roughness profile, was used (43).

Regarding the influence of surface treatments between the four CAD/CAM materials, the A and AS groups produced significantly higher Ra values compared to the others and followed by the C and CS groups significantly. Untreated group K and S surfaces showed generally the lowest roughness values. In the present study, NH material showed the highest Ra values in A and AS groups followed by VE materials. Then, among the treated surfaces, the lowest values were observed in H and HS groups. Both NH and VE materials are hybrid ceramics and have different microstructures. The moduli of elasticity of NH and VE materials are 9.9 GPa and 30.0 GPa according to the manufacturer's information (34, 44). The composition of the NH new hybrid ceramic material matrix consists of 50% nano glass and 50% polymer matrix. According to the manufacturer's information, 100% silanized glass is permanently integrated into the polymer matrix (34). VE is based on a polymer-infiltrated ceramic network material that consists of a dominant network (86 wt.%) reinforced by an acrylic polymer network (14%). The two networks penetrate each other completely (45). This could possibly be attributed to the different compositions of these two hybrid ceramics with different filler contents, which have an impact on the much higher SR values. According to the SBS test results for the different tested materials, the H and HS chemical conditioning groups produced the highest bond strength values contrary to the SR test results.

Although the SR of the materials is high in A and AS groups in the present study, generally the highest SBS values were observed on H and HS treated surfaces. The lowest SR values in H and HS groups were found in VE and CS materials, but also the highest SBS values.

According to the SBS results, H and HS treated surfaces produced the highest bond strength and the highest values were observed on the CD material (18.01 ± 4.07 MPa) in the H group and followed by the VE material (16.25 ± 5.83 MPa) in the CS treatment group. Both ceramics are etchable ceramics. Then, these materials were followed by the VE material (14.5 ± 3.33 MPa) in the HS group, VS material (12.84 ± 4.37 MPa) in the H group and NH material (12.81 ± 4.03 MPa) in H and (12.44 ± 3.43) HS groups. As surface treatments, Frankenberger et al. suggested using HF acid for the VE

material and also lithium disilicate ceramics. (46). In another study, Aboushelib et al. investigated the effect of surface treatments of two types of lithium disilicate ceramics on the micro tensile bond strength to a resin adhesive and found that the highest strength values were observed significantly in the CD material (34 MPa), which were also used and found in the present study (10). Also, they concluded that bond strength to lithium disilicate ceramics depends on proper surface treatment and on the chemical composition of the glass ceramics. Their bond strength methodology was not exactly the same as we used in this study, there was no micro tensile bond strength, but we used the SBS test. So, direct comparisons are not applicable. Sato et al. evaluated the effect of surface conditioning of the zirconia reinforced lithium silicate ceramic and resin cement on the micro tensile bond strength and observed that the silica coating was not efficient and etching with hydrofluoric acid for 20 seconds was effective for the stable resin bonding (12). In another study, Al-Thagaafi et al. (47) investigated the effect of surface conditioning protocols on the VS zirconia reinforced lithium silicate ceramic material with micro tensile bond strength and found that the bond strength of VS was 31.2 MPa in the HS treatment group and also has the highest bond strength values in sandblasting with Cojet sand. Pneumans et al. and Elsaka et al. investigated the micro tensile bonding performance of different glass ceramics and resin cement (11, 32). Pneumans et al. used H acid etching (5%) and silane combination as the surface treatment and they found that the best pretreatment for C (41.5) and VE (46.3) materials included etching with HF acid and observed that an application of S did not have any effect on the bond strength values (11). In contrast to the study of Pneumans et al., we observed the highest SBS values for the CD material in the HS (18.01 MPa) group compared to the H group (11.58 MPa). Elsaka et al. found the highest SBS value for the VE material (27.4 MPa) (32). The HS group showed higher values of bond strength compared to other treated surfaces, and the specimens including cohesive failure failed in a mixed mode. In this study, among all tested materials, the failure mode for the VE material was found between all tested materials the highest percentage of mixed cohesive failure. For CD and VS glass ceramics were observed more adhesive and cohesive mixed failures. Another published investigation measured the micro bond strength values of VE and CD materials and found 20.2 and 26.9 MPa (46). Both values are very close to the results of the present study in the HS group. Various in vitro studies established that H acid etching in combination with the use of silane is the finest surface treatment for lithium disilicate glass ceramics and the zirconia reinforced lithium silicate ceramics (11, 48, 49). According to the results of this study, as surface roughness increased through mechanical surface treatment as A or C, air-particle abrasion had less impact on the SBS test than chemical conditioning.

Concerning the mechanical bond strength, the present study used the SBS method to investigate the bond strength between different glass ceramic materials and resin cement. Hu et al. (29) evaluated and compared the adhesion of

different-cement combinations with the SBS test. The VS material (21.6 MPa) they found in the glass-based ceramic groups revealed significantly higher values than the VE material (14.7 MPa), and they also observed that on etched surfaces, VE exhibited primarily cohesive failures and the VS material exhibited primarily adhesive failures. According to the results of this study, considering all surface treatments, while adhesive and cohesive failures were found in the VS material, adhesive and primarily cohesive failures were observed in the VE material. This result was in accordance with the results of Hu et al and Elsaka et al. (29, 32). Also, similarly, some studies investigated the effect of the SBS test on hybrid ceramics (14, 30). Gungor et al. concluded that SBS was affected by surface treatments and found strength values for the VE material in HS treatment groups as 17.91 MPa (30).

Rohr et al. observed the highest SBS values for the VE material as 11.5 MPa and Schwenter et al. as 19.9 MPa. Similar results were obtained in the present study, which was in agreement with previous studies (14, 16, 20, 29, 30). In the present study, the highest SBS value with the VE material was determined in the CS group as 16.25 ± 5.83 MPa, not in the HS group. VE with CS treatment showed comparable bond strength to HS. This result is in agreement with previous studies (26, 32). In the study by Campos et al., C treatment group also resulted in the highest bond on the VE material before aging, but micro tensile bond strength values decreased significantly after aging (26). According to the study of Elsaka et al., for the groups etched with H and HS, the SR was lower than A, AS treatments, but the higher bond strength on the non-aged group for the VE material was found in the HS group and were comparable with the AS group (32).

Between the surface treatment groups, most of the specimens in the HS group showed mixed and primarily cohesive failures according to the present study and also the failure mode may also change with different tested material types. (32). The adhesive type of failure is typically associated with low bond strength values. Thus, mixed and cohesive failure modes are clinically preferable to total adhesive failure (50). Therefore, this finding is valuable data on the performance of the ceramic-resin bond where the resin cement is chemically bonded to the substrate material (20). Several studies reported mean SBS values between 15-25 MPa among the glass ceramic and resin for clinical applications, which is in agreement with the present SBS values data (14, 16, 20, 29, 30).

In this study, a single type of resin cement and silane coupling agent were used to ensure standardization. However, the sort of luting agent is the main determinant in the adhesion of dental materials.

Another limitation of the study is that thermal cycling was not included. Specimens in water at 37 °C were only short-term storage. Future in vitro studies should be conducted to investigate other factors such as different resin cement. Silane coupling agents and different test methodology should involve aging conditions together with the use of

thermocycling to provide a closer simulation of clinical situations.

5. CONCLUSION

With the limitations of this in vitro study, these conclusions can be emphasized:

In the present study, surfaces with A and AS treated groups increased the SR for all tested ceramics and are significantly followed by the C and CS surface treated groups.

Among all ceramic materials, the highest SBS values were observed in the HS group on the CD material and in the CS group on the VE material and in H and HS surface treatment groups on the VS and NH materials. The failure mode for CD and VS and NH materials was found to be the highest percentage adhesive and cohesive failures, however, adhesive and mixed cohesive failure was primarily observed for the VE material.

3. The higher SR will not always provide a higher bond strength value. SBS test results depend on the effect of surface treatments on the bond strength of the tested materials to resin cement.

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Conflicts of interest

The authors declare that they have no conflict of interest

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