Influence of Surface Modification Protocol and Type of Luting Cement on Bonding of Monolithic Zirconia to Dentin Substrate

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ABSTRACT

Aim and objective: To assess microtensile bond strength (μTBS) of two types of luting cement to monolithic zirconia and dentin following various surface modifications and aging.

Materials and methods: Monolithic zirconia blocks were divided into four main groups. Group N: no surface modification; Group SB: sandblasted using 50 μm Al₂O₃ particles. Group GH: glazed with a thin film of low-fusing porcelain glaze and etched with 10% hydrofluoric acid for 60 seconds. Group CJ: sandblasted with CoJet sand. Surface-treated ceramic blocks were bonded to dentin using either self-adhesive cement or resin-modified glass ionomer cement and submitted to 3 or 150 days of water storage protocol with aging. The specimens were subjected to tensile force until de-bonding. Surface roughness (Ra, μm) was assessed after surface treatment. Analysis of variance (ANOVA) tests followed by Tukey's tests were used to analyze the data (α = 0.05).

Results: Surface modification using selective infiltration etching (SIE) showed significantly higher μTBS (p < 0.05) compared to tribochemical silica coating, sandblasting, and no treatment groups. The surface roughness of the SB and CJ groups were statistically higher compared to GH and N groups.

Conclusion: Selective glass infiltration etching was an effective method in altering the surface properties, creating a strong and durable bond to monolithic zirconia.

Clinical significance: Surface treatment procedures using SIE techniques combined with the use of universal 10-Methacryloyloxydecyl dihydrogen phosphate (MDP)-containing adhesives could establish a long-lasting and strong bonding to monolithic zirconia restorations. Resin-modified glass-ionomer cement (RMGIC) is an alternative luting cement for monolithic zirconia based on the assessment of its bond strength and bond durability.

Keywords: Bonding, Cement, Ceramic primer monolithic zirconia, Selective infiltration etching.

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application, altering the surface topography and producing similar bonding mechanisms like silica-based ceramics.\textsuperscript{21,22}

For bonding of ceramic restorations, several types of commercially available luting cements, including resin-modified glass-ionomer cement (RMGIC), composite cements (conventional and self-adhesive) are recommended.\textsuperscript{23-25} Counter to conventional composite resin cements, RMGIC and self-adhesive composite cements entail fewer pretreatment steps. This is because of their chemical affinities for both tooth substrates and restorative materials.\textsuperscript{23-25} Self-adhesive resin cement contains acid-functionalized monomers and conventional methacrylate monomers. Based on the functional acidic monomers, there are two popular groups; methacrylate monomers with carboxylic acid groups such as 4-META-based cements and PMGDM-based cements or with phosphoric acid groups such as 10-Methacryloyloxydecyl dihydrogen phosphate (MDP)-based cements and BMP-based cements.\textsuperscript{26-30}

A 3-year clinical study proposed that the clinical retention rate of zirconia restoration based mainly on the proper selection of luting cement, and except for zinc phosphate cements, most luting cement systems are expected to adhere to zirconia ceramic as a result of the presence of functional polymers or monomers in their composition.\textsuperscript{31} Although there is no report suggesting that phosphate ester monomers are a constituent of RMGIC, this does not prevent RMGIC from achieving a higher bond strength to Y-TZP compared to a conventional type of composite resin cement.\textsuperscript{24}

The development of innovative approaches to achieve optimal adhesion between different types of luting cements and zirconia is considered as one of the most important areas in dental materials research.\textsuperscript{14-17} The current status of adhesive cementation of monolithic zirconia has recently been the subject of a comprehensive review; however, no definitive approach for optimal adhesion has yet to be found.\textsuperscript{32}

The current study assessed the impact of various surface conditioning protocols, type of luting cement, and water storage periods with or without thermocycling on the bond strength of monolithic zirconia bonded to dentin substrate. The null hypothesis tested were: (1) no statistically significant difference in the microtensile bond strength (μTBS) would be found between zirconia bonded to dentin after surface modification using either airborne particle abrasion, selective infiltration etching (SIE), or tribochemical silica coating; the type of luting cement and the aging condition has no significant impact on in the μTBS of surface-treated monolithic zirconia bonded to dentin.

**Materials and Methods**

This study was an *in vitro* study at which freshly extracted sound human mandibular molars (*N* = 80), free of cracks, caries, and restorations were selected. The teeth were collected with no associated patient identifiers in accordance with an institutional review board, Taibah University. The recruited teeth were scaled, stored in 0.1% thymol until used, within 6 months of extraction. To prepare a flat dentin surface, the occlusal surfaces of teeth were cut flat perpendicular to the long axis of each tooth with a diamond saw at low speed and water-cooling. Dentin surfaces were ground flat under running water using 600-grit silicon carbide papers to achieve a uniform smear layer.

Partially sintered zirconia blocks (inCoris TZI C, Sirona Dental, Germany) were cut under constant water cooling using a precision cutting machine (Isomet 4000, Buehler Ltd., Lake Bluff, IL, USA) to obtain blocks measuring 8 mm length, 8 mm width and 5 mm thickness. Zirconia blocks were sintered in a zirconia sintering furnace (Sirona inFire HTC, Sirona Dental Systems, GmbH, Germany) following the manufacturer’s recommendations. After sintering, zirconia blocks (6.4 mm length, 6.4 mm width, and 4 mm thickness) were sonically cleaned for 5 minutes in distilled water and air-dried.

Zirconia blocks were assigned into four main groups based on the surface treatment protocol used; Group N: control (no treatment). Group SB: the specimens were sandblasted with 50-μm Al2O3 particles at 0.1 MPa pressure from a distance of 20 mm for 15 seconds, followed by a 3-minute ultrasonic cleaning in 70% isopropyl and air-dried.

Group GH: MZ specimens were treated as follows; the glaze ceramic (Vita Akzent glaze) was used. Mixing of 0.035 g of ceramic frit with 0.2 mL of distilled deionized water, a thin slurry of glazing ceramic was formed and carefully applied with a clean brush to the zirconia surface, evenly distributed using a vibrating technique, and air-dried for 60 seconds. The glaze coating was fired (Vita Vacuumat 40 furnace, Vita Zahnfabrik, Germany) following the manufacturer’s instruction. The specimens were etched with HF gel (10% HF) for 60 seconds, washing under copious distilled water for 60 seconds. Group CJ: Chairside microblasting unit (Micro-Etcher II) was filled with 30 μm Al2O3 particles coated with silica (CoJet-Sand, CoJet system, 3M ESPE AG, Seefeld, Germany). Sandblasting was performed perpendicular to the bonding surface for 20 seconds from a distance of 10 mm at 2.8 bar pressure.

The surface roughness (Ra, μm) of the specimens after surface modification was assessed by the use of a profilometer (Mitutoyo Surf Test SJ 201 P/M; Mitutoyo Corporation, Tokyo, Japan). A tip radius of 5 μm of a diamond stylus with 0.25 mm/second driving speed was used. For each specimen, the surface roughness was assessed at three different locations and the average was then recorded.

Pretreated ceramic blocks were coated with an adhesive containing phosphate monomer (Clearfil Ceramic Primer; kuraray Noritake; Tokyo, Japan) that was applied to the treated surfaces with a microbrush and the excess was removed with an oil-free air stream.

The zirconia specimens were bonded to dentin surface using: a self-adhesive resin cement (Relyx Unicem, 3M ESPE, Seefeld, Germany) or a resin-modified glass ionomer cement (Relyx Luting Plus, Houston, TX, USA) according to the instructions of the manufacturer under a load of 750 g using a customized device. Resin cement was light-activated from each direction for 40 seconds using a halogen light (Hilux Ultra Plus, 600 mW/cm², Benlioglu Dental, Ankara, Turkey). Following the cementation procedure, the test specimens were stored in an incubator for 24 hours at 37°C under 100% humidity. All the procedures were performed by a single operator.

The bonded assemblies were cut with a diamond disk of the cutting machine (Buehler, Series 15LC Diamond, Buehler) along two axes (x and y) under water cooling. Non-trimmed beam specimens with a bonded area of 1.0 ± 0.1 mm², as measured with a digital caliper were produced. The initial section was discarded due to the probability of the presence of enamel and cement discrepancies that might falsify the results and only the inner beams were included in the study. The resultant beams were examined using a light microscope and only intact, crack-free beams were included.

The beam specimens obtained from each subgroup were assigned to one of the two aging conditions. While half of the
specimens were submitted to the microtensile test after a 3-day water storage at 37°C without artificial aging (NT), the other half were tested following 37,500 thermocycles over 150 days (distributed over five intervals of 7,500 cycles each, starting after 25-, 50-, 75-, 100-, and 125-day water storage) between (5 and 55 ± 2°C, dwell time: 30 seconds, transfer time: 5 seconds).

The beams were attached to the jig of the universal testing machine (Lloyd Instrument, Type 500, Hants, United Kingdom) with cyanoacrylate adhesive (Super Bonder–Flex Gel, Brazil) and stressed to failure in tension at a cross-head of 1 mm/minute and a load cell of 50 kgF. Microtensile bond strength data (MPa) were calculated by dividing the maximum tensile stress by the mean cross-sectional area of each beam. Test specimens that debonded spontaneously before tensile testing were assigned a bond strength of 0 MPa.

Failure pattern of all debonded specimens was examined under a stereomicroscope (Discovery V20, Zeiss) at 40x magnification and was classified as follows: adhesive (failure at the resin/dentin or resin/zirconia), or cohesive (failure exclusively within the resin cement) and mixed failure.

Three additional samples from each surface conditioning before tensile testing were assigned a bond strength of 0 MPa. Test specimens that debonded spontaneously before tensile testing were assigned a bond strength of 0 MPa.

Statistical Analysis
Levene test and the Shapiro–Wilk test were conducted to confirm the assumption of homogeneity and normality for the dependent variables (µTBS (MPa)) across all levels of the independent variables (surface treatment, luting cement, and storage time) indicated that, the data were normally distributed; therefore, parametric statistics were used. A three-way analysis of variance (ANOVA) was used to evaluate the overall statistical significance of differences among study variables and their interaction. Ra values (μm) were compared with one-way ANOVA. Multiple comparisons were made using Tukey’s post hoc test (α = 0.05). The modes of failure were analyzed with the Chi-square and Kruskal–Wallis tests to disclose the differences between the tested groups (α = 0.05). All statistical analysis was conducted using SPSS 20.0 software for Windows (SPSS Inc., Chicago, IL, USA).

Results
Mean values ± standard deviations (SD) of µTBS (MPa) are presented in Table 1.

All groups showed a reduction in µTBS over 15 days of water storage with thermocycling irrespective of the type of surface treatment or the luting cement used. A statistically significant difference in µTBS between 3 and 150 days was observed for groups SB and GH for both types of luting cement. However, the decrease in the bond strength after 150 days in the CJ group was not statistically significant (p > 0.05) for the resin cement group, while the RMGIC groups showed a significant impact of 150 days of water storage on bond strength values (Table 1).

Three-way ANOVA (surface modification protocol, type of luting cement, and aging process) revealed that all factors significantly influenced µTBS (surface treatment: (p < 0.001); luting cement: (p < 0.001); aging condition: (p < 0.001)), while no interaction between three main factors was found (p = 0.870) (Table 2). In addition, significant interaction was recorded between surface treatment and storage time (p = 0.023) and between surface treatment and cement type (p < 0.001) while no significant interaction was recorded between cement type and storage time (p = 0.329).

A statistically significant difference (p < 0.05) between different surface modification was recorded (Tukey’s test) with GH groups showing the highest µTBS values.

The self-adhesive resin cement showed µTBS higher than that recorded for RMGIC used except for the control group, irrespective of the surface treatment protocol and aging condition (Table 1).

The frequencies of the failure pattern of all tested variables are presented in Figures 1 and 2. A predominantly adhesive type of failure with a completely exposed zirconia or dentin surface has been identified in the control group.

However, regardless of the type of luting cement or storage period, SB groups displayed more adhesive than the mixed type of failures compared to the GH groups that showed a more mixed than adhesive failure mode. CJ group showed mainly an equal percentage of mixed and adhesive failure mode (Figs 1 and 2).

Scanning electron microscope evaluation of each surface pretreatment is presented in Figure 3.

For the control group, the surface topography analysis (Fig. 3A) showed a smooth surface pattern with no morphological alterations, whereas the SB group revealed uneven surface irregularities of the treated zirconia surface (Fig. 3B). Selective etching patterns with homogeneous surface irregularities have been found for the GH group (Fig. 3C). The CJ group (Fig. 3D) also displayed a surface topography comparable to that of the SB group (Fig. 3B).

The surface roughness data (Ra, μm) of the surface-treated monolithic zirconia are presented in Table 3. One-way ANOVA revealed that Ra recorded with sandblasting and tribochemical silica coating was significantly higher compared with the SIE and control groups (p < 0.001).

Table 1: Means and standard deviation of microtensile bond strength values (MPa) for the experimental groups. Different lowercase letters in the same column indicate a significant difference between groups (p < 0.05)

| Treatment | Cement       | Storage (days) | Mean  | Std. deviation |
|-----------|--------------|----------------|-------|----------------|
| N         | Resin cement | 3              | 10.7a | 2.5            |
|           |              | 150            | 9.5a  | 2.6            |
| Modified GIC | 3              | 9.4a           | 2.7   |
|           |              | 150            | 8.7b  | 3.6            |
| SB        | Resin cement | 3              | 24.06c| 3.9            |
|           |              | 150            | 20.9d | 4.0            |
| Modified GIC | 3              | 18.5d          | 2.9   |
|           |              | 150            | 15.6e | 3.3            |
| GH        | Resin cement | 3              | 30.5f | 3              |
|           |              | 150            | 25.1c | 3.2            |
| Modified GIC | 3              | 23.9c          | 3.0   |
|           |              | 150            | 20.5d | 2.7            |
| CJ        | Resin cement | 3              | 27.5f | 2.8            |
|           |              | 150            | 23.7c | 3.3            |
| Modified GIC | 3              | 20.4d          | 3     |
|           |              | 150            | 17.1g | 2.4            |
Table 2: Results of three-way ANOVA for microtensile bond strength (μTBS) values

| Source                        | Type III sum of squares | df  | Mean square | F       | Sig.     |
|-------------------------------|-------------------------|-----|-------------|---------|----------|
| Corrected model               | 10428.402               | 15  | 695.227     | 71.648  | <0.001   |
| Intercept                     | 87944.474               | 1   | 87944.474   | 9063.337| <0.001   |
| Treatment                     | 8159.118                | 3   | 2719.706    | 280.286 | <0.001   |
| Cement                        | 1338.593                | 1   | 1338.593    | 137.952 | <0.001   |
| Storage                       | 526.288                 | 1   | 526.288     | 54.238  | <0.001   |
| Treatment × cement            | 293.968                 | 3   | 97.989      | 10.099  | <0.001   |
| Treatment × storage           | 94.254                  | 3   | 31.418      | 3.238   | 0.023    |
| Cement × storage              | 9.283                   | 1   | 9.283       | 0.957   | 0.329    |
| Treatment × cement × storage  | 6.899                   | 3   | 2.300       | 0.237   | 0.870    |

Fig. 1: Graph of the failure pattern disruption within each experimental group

Figs 2A to D: SEM micrographs (1000x) of debonded surfaces showing: (A) Adhesive failure mode (dentin substrate); (B) Mixed failure mode at dentin-resin interface; (C) Mixed failure mode at zirconia resin interface of air abraded group; (D) Cohesive failure of resin-modified glass ionomer cement
Achieving durable bond strength with zirconia restorations is still a challenging procedure. Many adhesive strategies that combine mechanical and chemical pretreatment have been developed to improve the durability of bonding between resin cement and zirconia.34

This study was conducted to assess the impact of different surface conditioning, luting cement type, and aging conditions on μTBS of monolithic zirconia bonded to dentin substrate. The results led to the rejection of the null hypothesis that: (1) no statistically significant difference in the μTBS of a monolithic zirconia bonded to dentin substrate would be found following surface modification using either airborne particle abrasion, SIE, or tribochemical silica coating; (2) the null hypothesis that the type of luting cement and the aging condition has no significant impact on in the μTBS of surface-treated monolithic zirconia bonded to dentin substrate.

The results of the current investigation showed that, regardless of the type of luting cement, SIE followed by primer application produced the highest μTBS values. This could be related to the intergranular porosity which leads to mechanical interlocking with the luting cement. The SIE involves only the exposed surface grains to the molten glass, with the resultant control of the area to be selectively etched.20–22

A study conducted by Salem et al. concluded that selective infiltration etching improves the bond strength to high-translucency zirconia and the resultant bond is stable, strong, and durable.22 Although the results were comparable to those of the current investigation, the authors used composite resin blocks as a substrate which could reveal different failure mode results than that recorded in this study.

Additionally, this finding was also supported by the results of Bottino et al.34 who concluded that both SIE and the tribochemical silica coating improve the bond between the resin cement and zirconia while they reported a non-statistical significant difference.
between both types of surface modification which was contradicted by the results of the current investigation. The explanation of this finding could be related to the use of different types of luting cements and the primer type.

Based on the finding of this study, surface roughness alone is not enough for enhancing the bonding process. Although the SIE group showed less surface roughness than showed by either SB or CJ groups, it demonstrated the higher bond strength values. The alteration in the surface properties following SIE is related to the ability of HF to chemically attack the applied silica oxide layer increasing its roughness, along with enhancing hydroxylation on the treated zirconia surface.20

Conversely, Cheung and Botelho reported that stable and strong bond strengths were not achieved following SIE of zirconia. Furthermore, a reduction of bond strength by 58.5% following thermocycling were recorded.23 This finding could be attributable to the use of a different type of zirconia in their study.

Higher bond strengths values recorded for the specimens subjected to tribochemical silica coating (CoJet system) followed by universal primer application compared with sandblasting (airborne particle abrasion with Al2O3 particles) and control groups for both types of luting cement/aging protocols, could be related to the role of silica particles in abrading the inner surface and its ability to react with the adhesive monomer in the ceramic primer.19,35

Additionally, tribochemical coating of silica surface treatment did not reduce bond strength values after 150 days of water storage when used with resin cement. That may suggest a more stable bonding, which would have a benefit over the other treatment methods used in this research.

The results of the current investigation record higher μTBs of a sandblasted group compared with the control group. This finding could be explained by the ability of airborne particle abrasion with aluminum oxide particles with a particle size of 50 μm to produce micromechanical roughness and to increases the surface energy of the zirconia surface area. Although the procedure is recommended by many investigators,5,12,35 some previous reports concluded that the use of sandblasting procedure to increase the surface roughness of zirconia has a negative impact on the durability of Y-TZP restorations as a result of the formation of structural defects and sharp cracks which render the susceptibility of zirconia framework to radial cracking during the function.16,17

The bond strength values achieved on zirconia after sandblasting and the remarkable percentage of adhesive failures showed that this type of surface modification did not support the development of enough micromechanical pores to improve the bond strength.

The μTBS of the control group display pretesting debonding even after 3-day water storage. This observation is supported by the finding of Yang et al., who reported a significant decrease in the bond strength to 0 MPa even using 10-MDP containing Primer.19 On the contrary, a study conducted by Aung et al. related the absence of pretesting debonding of untreated zirconia to the finding that both self-adhesive resin cement and primer containing 10-MDP effectively bonded to polished, but not air-abraded, zirconia.21

Independent of the surface treatment procedure and luting cements used in the current study, the water storage combined with thermocycling reduced the bond strength except for the control group. This could be attributable to the combination of hydrolytic degradation, water diffusion into the interfacial layer, and thermal irradiation during thermocycling favors degradation of the interface. Additionally, the use of the dentin substrate to simulate oral condition affects, as the bond between luting cement and dentin appears to be more liable to the hydrolysis following the thermocycling aging process. In addition, after the thermocycling aging process, the interfaces between the resin cements and the zirconia surfaces showed an increase in adhesive failures.28,36

Regarding the type of luting cement, self-adhesive resin cement revealed statistically higher bond strength values than recorded with the RMGIC except for control and air-abraded groups. This could be partly attributed to the incorporation of the hydrophilic dimethacrylate monomer urethane dimethacrylate (UDMA) that tends to absorb water which is responsible for the adhesive failures observed with RMGIC groups. Most adhesives underwent a degree of dissolution in water which increased when exposed to other media, e.g., acids and bases.25

This finding was supported by the study conducted by Yang et al. who studied the impact of the type of luting cement on the shear bond strength to zirconia, they concluded that self-adhesive composite cement showed a higher μBS than RMGIC, suggesting that the improved bonding properties of the self-adhesive composite cement to Y-TZP are dependent on the phosphate ester monomer type or brand.24

These results could be related to the high content of acidic phosphate functional monomers in self-adhesive resin cement which could increase the hydrophilicity of the cement resulting in hydrolytic degradation due to the higher water sorption.37 Tanış and Akçabay18 have shown that the use of MDP-containing resin cement has increased the bond strength with the airborne particle abrasion group. Another study reported that abraded zirconia specimens bonded to methacrylate-based cement showed lower bond strength than those bonded to MDP-containing resin cement as MDP monomers produce chemical interactions with zirconia.37,39

On the contrary, a study conducted by Ernst et al. reported that the retentive strength of RMGIC had the same level of retentive strength as the self-adhesive resin cement to zirconia ceramics.40

The high percentage of adhesive failures and the low bond strength values recorded in the control group confirmed that no interaction occurred between cement material and the zirconia substrate which is related to the nature of the zirconia ceramics that is a relatively inert material with low surface energy and wettability. It has been proposed that the minimum required strength for clinical bonding is between 10 and 13 MPa.41,42 In the present study, the control group does not have sufficient strength to provide stable bonding to monolithic zirconia.

In the present study, debonded specimens in SB groups displayed both adhesive and mixed failure mainly at the cement–ceramic interface. These observations proposed that the established bond at the interface might be restricted to weak intermolecular attractions, such as, the polar hydroxyl groups on the zirconia and the hydrogen bonding between the polar functional groups of the polymers or monomers in the cement.11,12 While for groups SIE and CJ, the mixed type of failure was recorded.

These results are in agreement with previous studies.33,34 It has been recorded that the mixed and cohesive modes of failure are clinically preferred to total adhesive failure which is mainly related to low bond strength.10,11,25,27

The results of the current investigation have certain limitations. Only one brand of monolithic zirconia was tested. In addition, two types of luting cement with one primer were used. Therefore, further studies are needed to evaluate the impact of different surface...
treatment protocols combined with different types of primer on the bond strength durability to different types of monolithic zirconia ceramics of different shades. In addition, the selection of fluoride-releasing luting cements based on their antibacterial activity; while the influence of fluoride in bonded interfaces is not tested. Also of interest would be the influence of monolithic zirconia sintering protocols combined with thermocycling on the bonding durability of resin cement to monolithic zirconia.

**Conclusion**

The pretreatment method and type of luting cement influences the effectiveness of the bond strength of monolithic zirconia to dentin substrate. Selective glass infiltration etching was an effective method in altering the surface properties, creating a strong and durable bond to monolithic zirconia. Resin-modified glass-ionomer cement could be considered as an alternative luting cement for cementation of monolithic zirconia restorations.

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