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Shear Bond Strength of Universal Adhesives with Different Methods of Zirconia Ceramic Preparation after Thermocycling: A Laboratory Study

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Abstract

Objectives: The present study aimed to assess the level of shear bond strength (SBS) of universal adhesives (G-premio Bond Universal (GBU) and All Bond Universal (ABU) on zirconia without surface preparation, zirconia sandblasted by 50-micron aluminum oxide particles, and zirconia glazed by low-fusing ceramics after the thermocycling process.

Methods: Two Ceramill ZI zirconia blocks were used to prepare 18 cubes with dimensions of 10×10×10 mm. Then, based on the type of surface preparation, these 18 sintered zirconia cubes were classified into three groups of six, including the control group, sandblasted group, and glazed group. Each group was classified into two subgroups based on the type of universal adhesive (ABU/GBU). On each cube, four cylinders filled with Panavia F2 resin cement with a diameter of 1.5 mm and a height of 2 mm were attached by the universal adhesive of the related subgroup (n = 72). The samples were subjected to 5000 thermal cycles in the thermocycler, each sample was assessed to test SBS, and the type of failure observed in each group was finally observed under a stereomicroscope. The data were analyzed using a two-way analysis of variance (ANOVA) test, Tukey's post hoc test, and independent t-test, with a significance level of 0.05.

Results: There was a statistically significant difference in SBS between the control group and the other two groups (p-value < 0.05). At the same time, there was no statistically significant difference between the sandblasted zirconia group and the glazed zirconia group (p-value = 0.13).

Conclusion: Applying a low-fusing glazed layer to prepare the zirconia surface leads to an increased SBS of dual-cured self-etch resin cement, equivalent to sandblasting with 50-micron aluminum oxide after the thermocycling process.

Keywords: Zirconia shear bond strength; Universal adhesive; Glazed zirconia

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Introduction

Zirconia has become very popular among clinicians because of its desirable chemical features, extended durability, and ideal esthetics.[1] Zirconia falls in the category of polycrystalline ceramics, which lacks a glass structure in its composition. For this reason, the etching process with hydrofluoric acid does not affect it. In other words, the lack of silica in its structure hinders its bonding to the resin.[2] Therefore, techniques are used to create influential bonding, such as grinding, sandblasting (air abrasion) by aluminum oxide particles, tribochemical silica coating, selective etching, laser etching of the surface, and applying zirconia primer or adhesive.[3] The presence of the 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP) molecule in the primers and adhesives leads to zirconia adhesion to the resin composite.[2,4,5] Primers containing this molecule and phosphate monomers improve shear bond strength (SBS) to zirconia.[6-8] This monomer has two functional groups: A phosphate group that causes adhesion to the hydroxyl group of the zirconia surface and a carboxylic acid group that is light cured and bonds to the resin composite.[4,9,10] The strong chemical bond between zirconia and the MDP molecule is created by the chemical interaction between the MDP molecule's phosphate ester group and the zirconia surface's hydroxyl groups.[11-13] The use of primers containing MDP also culminates in increasing the SBS of resin cement with bisphenol-A-glycidyl methacrylate (Bis-GMA) to zirconia. [14] This monomer, despite its amphiphilic property, is still used as the most hydrophobic functional monomer in primers or adhesives, enhancing the SBS in the oral environment.[15] Universal adhesives are a new group of adhesives that can bond to enamel, dentin, metals, zirconia, and glass ceramics by all bonding techniques (total-etch, selective etch, and self-etch). These adhesives have MDP monomer, increasing the SBS.[16] Saliva, thermal changes, and acidity of the oral environment after consuming food give rise to a decrease in the mechanical stability of zirconia.[17] Currently, the most common method to elevate the bonding ability of zirconia is sandblasting, plus the use of resin cement containing MDP, such as Panavia.[18] This method and other methods mentioned above enhance the immediate bond to zirconia; however, when placed for a long time in the oral environment, which is a dynamic environment, they decrease the SBS.[19,20] A new method employed to improve SBS in zirconia is to create a silica-rich layer in the zirconia structure by preparing the zirconia surface by applying a low-fusing glazed layer or ceramic liner, which can also be resistant to aging.[21]

The present research aimed to assess the level of SBS of two universal adhesives on zirconia without surface preparation, zirconia sandblasted by 50-micron aluminum oxide particles, and zirconia glazed by low-fusing ceramics after the thermocycling process.

Methods

This study was registered by the Ethics Committee of Hamedan University of Medical Sciences (IR.UMSHA.REC.1399.739). Two Ceramill ZI (Amann Girrbach, Germany) zirconia blocks with a diameter of 98×87.5×12 mm were prepared, and then each block was scanned by a Ceramill map200 (Amann Girrbach, Germany) scanner and by the program defined in the software based on milling cubes sized 10 × 10 × 10 mm, milled in a Ceramill Mikro (Amann Girrbach, Germany) dry milling machine and finally sintered by a Ceramill Therm 3 (Amann Girrbach, Germany) furnace at a temperature of 1450℃ for 10 hours. Of each block, 14 cubes with dimensions of 10 × 10 × 10 mm were obtained by the milling machine, of which only 18 cubes were used for this study.

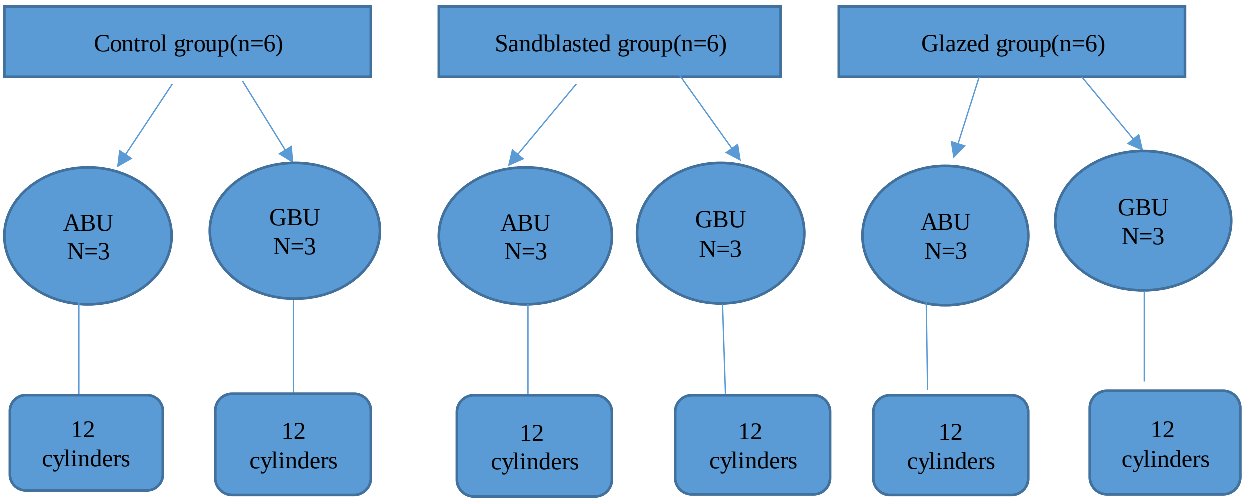
Preparation of Groups

The surfaces of all 18 sintered cubes were polished and finished by medium disk no.7601, medium wheel no.7648, fine disk no.7701, and fine wheel no.7748 related to DIACERA HP (EVE, Germany) zirconia polishing discs, respectively, and mounted inside self-cured acrylic resin (Acropars, Iran). Then, based on the surface preparation, the mounted samples were classified into three groups of six samples in each: 1- The control group without any surface preparation of zirconia, 2- the group sandblasted with 50 microns aluminum oxide, and 3- the group of zirconia glazed with low-fusing glaze powder and liquid. Also, each group was classified into two identical subgroups based on the type of universal adhesive: A: ABU and B: GBU. In each subgroup, there were 12 cylinders made of resin cement.

Each subgroup had four cylinders filled with dual-cured self-etch resin cement, cemented to the zirconia surface by universal adhesive. Figure 1 displays a zirconia cube mounted in an acrylic block along with a cylinder made with resin cement.

Figure 1: Zirconia cube mounted in an acrylic block with 4 cylinders made with resin cement

The ingredients used in the research are provided in Table 1.



1. Control Group

In the control group, no zirconia surface preparation was carried out. The surface of each block was treated with 37% phosphoric acid (Condac37, FGM, Brazil) for 10 seconds to clean the surface and enhance the surface's free energy. Then, the surface was washed for 15 seconds and completely dried by air syringe. In the next step, silane primer (Angelus, Brazil) was applied with a micro-brush for one minute and then uniformly spread by air syringe. Afterward, the ABU universal adhesive (Bisco, USA) was actively Rubbed on half of the samples by a micro-brush for 20 seconds, utterly spread on the surface for five seconds by air syringe, and cured for 10 seconds by the light-emitting diode (LED) light-curing device (D-Lux, Dia dent) with a power of 1600 mW/cm^2 in soft mode, and the GBU universal adhesive (GC, Japan) was applied on the other half of the samples similarly as before. Then, dual-cured Panavia F2 self-etch resin cement (Kurary, Japan) was placed into four Tygon tubes with a diameter of 1.5 mm and a height of 2 mm; each tube was placed on zirconia blocks and light-emitted for 20 seconds by the LED light-curing device according to the manufacturer's instructions and in soft mode, in such a way that the highest part of the device was placed on the top of the cylinder. After the curing process, the Tygon tube was separated from the resin cement by a scalpel. Finally, Oxygen_ inhibiting gel (oxyguard II, Kurary, Japan) was used, and it was placed for three minutes at the resin cement cylinder-zirconia block surface junction. After this duration and washing the gel residues, the cylinder-zirconia cube surface junction was light-cured again for 20 seconds.

Table 1- The ingredients used in the study

Material	Type of Material	Main Components
G-Premio Bond GC Corporation, Tokyo, Japan pH=1.5	Universal adhesive	10-MDP, 4-MET, MTDP, methacrylic acid ester, silica, acetone, water, photo-initiators
All_ bond universal Bisco, Schaumburg, IL, USA pH=3.1	Universal adhesive	10-MDP, 2-HEMA, BisGMA, ethanol, water, photo-initiator
Panavia F2 Kurary Noritake dental Inc, Japan	Self-etch, Dual cure dental adhesive	A paste: 10-MDP, hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, hydrophilic aliphatic dimethacrylate, silanated colloidal silica, silanated silica filler, catalusts, initiators B paste: hydrophobic aromatic dimethacrylate, hydrophobic aliphatic dimethacrylate, hydrophilic aliphatic dimethacrylate,silanated barium glass filler, surface treated sodium fluoride, catalysts, accelerators, pigments
Ceramill ZI Amann girrbakh, Germany	Zirconia block CAD material	$ZrO_2 + HfO_2 + Y_2O_3 \geq 99\%$ Y_2O_3: 4.5 – 5.6% $HfO_2 \leq 5\%$ $Al_2O_3 \leq 0.5\%$ Other oxides ≤ 1%
Silane Angelus, Brazil	Ceramic primer	Silane and ethanol
Hydrofluoric acid10% Maquira, Brazil	Porcelain Etchant	10% hydrofluoric acid, thickening agent, dye, purified water

2. Sandblasted Group

The samples of this group underwent a 10-second sandblasting process by 50-micron aluminum oxide particles from a distance of two centimeters with a pressure of two bar; then kept in distilled water at room temperature for 10 minutes, then completely dried by a strong power of air syringe. Afterward, 37% phosphoric acid (Condac37, FGM, Brazil) was applied for 10 seconds, washed for 15 seconds, and completely dried by air syringe. In the next step, silane primer (Angelus, Brazil) was applied with a micro-brush for one minute [22], and after this period of time, it was dried by an air syringe. Then, ABU was applied to half of the blocks, and GBU to the other half, similar to the instructions mentioned in the previous section. The cementation process was also carried out again in this step.

3. Glazed Group

The low-fusing glaze powder (IPs, Ivoclar Vivadent, USA) was mixed with glaze liquid on a glass slab; a very thin layer was applied to the zirconia block surface using a very thin brush in such a way that it did not affect the zirconia internal compatibility, and it was then baked in a Programat EP 3000 (Ivoclar Vivadent, USA) furnace at 725°C for 20 minutes. Afterward, the samples were kept in distilled water at room temperature for 10 minutes, then they were completely dried by a strong air syringe. The 10% hydrofluoric acid (Maquira, Brazil) was used for one minute to etch the zirconia surface, washed by a strong stream of high-pressure air and water for 15 seconds, and completely dried by air syringe to check the etched surface. Afterward, 37% phosphoric acid (Condac37, FGM, Brazil) was applied for 10 seconds to remove the surface from the salts left by hydrofluoric acid, washed for 15 seconds, and completely dried by air syringe. In the next step, silane Primer (Angelus, Brazil) was applied with a micro-brush for one minute [22], and dried by an air syringe. Then, ABU was applied to half of the blocks, and GBU to the other half, similar to the instructions mentioned in the previous section. The cementation process was also carried out again in this step.

Thermocycling Process

All samples underwent thermocycling 24 hours later in a thermocycler (thermocycler-Mashhad) in distilled water at 55-5℃ for 5000 cycles with duration of exposure in cold and hot water and a delay time of 20 seconds.[23]

Evaluation of Shear Bond Strength

After the thermocycling process, the samples were placed in the universal testing machine (STM-50, Santam, Tehran, Iran) to evaluate SBS in such a way that the 26 mil (0.026 inch) brass orthodontic wire was pulled from the top of the machine as a loop covered each cylinder from its interface with the zirconia block surface at a speed of 0.5 mm/min and applied force until its failure. Finally, SBS was calculated by dividing the maximum stress failure by the cross-sectional area of each sample in Mpa.

In order to assess the types of bond failure, including adhesive (failure in the distance between cement and zirconia surface), cohesive (failure inside resin cement), and mixed (adhesive and cohesive failures), all samples were observed under a stereomicroscope (Olympus, Tokyo, Japan) with 40 times magnification.

According to the study objectives, the data were collected and analyzed using SPSS software version 26, and descriptive statistics methods and statistical tests, such as the two-way analysis of variance (ANOVA), Tukey's post hoc test, and independent t-test. The reliability of this test was considered 95%, and the test power was 80%. The significance level was considered to be 0.05 in all the assessed tests.

Results

At first, the data normality was assessed by the Kolmogorov-Smirnov test. Descriptive results of SBS of GBU and ABU universal adhesives in the control, sandblasted, and glazed groups are shown in Table 2.

Table 2- Mean and standard deviation of band strength in study groups based on Mpa

Groups	Mean and SD of GBU	Mean and SD of ABU	P_value between the control group and the other two groups	P_value between sandblasted and glazed group
Control	13.99 ± 2.51	13.17 ± 3.33	P_value<0.05	P_value=0.13
Sandblasted	20.95 ± 3.21	18.55 ± 2.94		
Glazed	18.27 ± 4.40	17.39 ± 3.71		

According to the one-way ANOVA and Tukey's post hoc test, there was a statistically significant difference between the control group and the other two groups (p-value < 0.05); however, there was no statistically significant difference between the sandblasted zirconia group and the glazed zirconia group (p-value = 0.13).

Based on two-way ANOVA, the type of adhesive (GBU-ABU) used in the groups did not have a significant effect (p-value = 0.94).

For two-by-two comparison of adhesives in each of the groups, the independent t-test was employed, which revealed no statistically significant difference between the type of adhesive used and the type of zirconia preparation (p-value = 0.66).

The frequency of failures observed in each subgroup is provided in Table 3, and the percentage of failure is shown separately for each group in Diagram 1.

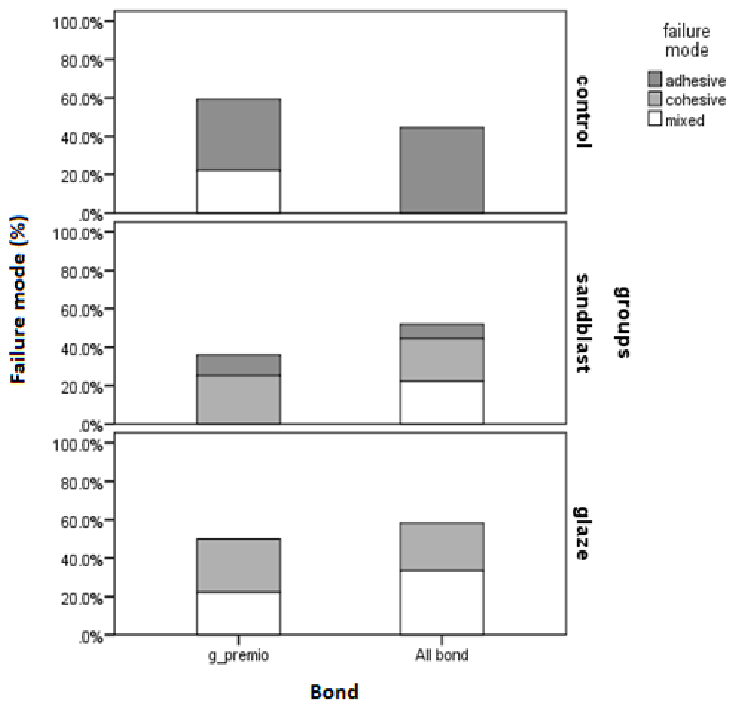


Diagram 1: The percentage of failure in each group

Table 3- The frequency of failures in each subgroup

	Control		Sandblast		Glaze		Total
	G_premio	All_bond	G_premio	All_bond	G_premio	All_bond	
Adhesive	10	12	3	2			27
Cohesive			9	8	10	9	36
Mixed	2			2	2	3	9

Discussion

The current research aimed to assess the SBS of two types of universal adhesive (GBU-ABU) on different zirconia ceramic preparation methods (sandblast, low-fusing glaze) after the thermocycling process. According to the results, universal adhesives containing MDP can increase SBS of dual-cured resin cement to zirconia ceramics prepared by different methods, and applying a low-fusing glazed layer on the zirconia surface equivalent to surface sandblasting can elevate

SBS of dual-cured resin cement. The results of this study are consistent with the studies conducted by Al Jeaidi et al. and Zakavi et al.[24,25] In Zakavi's study, it was concluded that the SBS of universal adhesives to zirconia was higher than the zirconia primer and the total-etch adhesive.[25] In Lumkemann et al.'s study, which is consistent with the present study, SBS of all universal adhesives, including GBU and ABU, to zirconia was reported to be high.[26] The results of this study match the results of Sharafeddin et al.'s [15] study. In their study, the zirconia primer had a higher SBS than ABU adhesive, the reason for which has been attributed to the synergistic effect between the acidic MDP and the carboxylic monomer present in the zirconia primer and the compatibility of this primer with various types of resin cement. They have also mentioned that the MDP monomer present in zirconia primer is different from universal adhesive.

In the current research, two types of Universal adhesive had MDP monomer, which is a Bifunctional molecule and is able to react with resin cement monomer and zirconia surface hydroxyl groups, leading to an increased SBS of the prepared zirconia compared to the control group. It is worth mentioning that following zirconia surface preparation using the sandblast, MDP performance increases [8,27], which was also confirmed in the present study. The chemical property of the MDP molecule alone is not effective for adhesion to zirconia and increasing its bonding, but the synergistic property of this molecule, plus the micromechanical adhesion property created by sandblasting, can result in enhancing SBS.[2,28] Similarly in the current study, the strength of Universal adhesives to sandblasted and glazed zirconia was higher than that in the control group.

In the present study, the SBS of the sandblasted and glazed groups was higher than that in the control group, and no statistically significant difference was found between the two groups under preparation. The present study results are contradictory to the results of Valentino et al.'s [22] study. They examined SBS of different groups of zirconia preparation as sandblast and glaze with and without silane to dual-cured resin cement and concluded that applying a low-fusing glazed layer on sintered zirconia, followed by hydrofluoric acid etching the surface, and also applying a low-fusing glazed layer and subsequent sandblasting of the glazed surface result in a considerable increase in the SBS compared to sandblasting alone. Moreover, in their study, applying silane compared to not applying it did not statistically increase the SBS. The reason suggested for this problem was that since the glazed layer is very thin, some of the surface containing glass particles may be removed by applying hydrofluoric acid or sandblasting, maintaining a small surface for the desirable bond. This issue is also true in the present research so that in the glazed zirconia group that had used hydrofluoric acid and silane, SBS was not statistically significant compared to the sandblasted group that had used silane. The difference between the present study and Valentino et al.'s study was assessing the SBS of two types of universal adhesives containing MDP in addition to assessing different methods of zirconia preparation on resin cement SBS. The use of silane in the glazed zirconia group should be carried out due to the presence of the glass phase, and if it is applied to the pure and even sandblasted zirconia, because of the absence of the glass phase in its structure, the zirconia cannot bond to it. The type of glass in various glaze powders may be different, which can influence the SBS of various types of glazed zirconia. It is worth mentioning that the glaze paste used in the present study contained alkali aluminosilicate glass, which induces a layer rich in silica on the zirconia surface. It has been mentioned in Derand et al.'s study that this design of adding low-fusing glaze should not have a thickness higher than five microns so as not to interfere with fixing the crown on the tooth, and the mechanism of this connection occurs in the form of electrostatic bonding and van der Waals forces on the zirconia surface.[29] Applying multiple layers can also impact the SBS, similar to Cheung et al.'s study, in which applying two glazed layers led to increasing resin cement SBS to zirconia compared to sandblasting alone.[18] The etching pattern type in glazed zirconia with different low-fusing glazed products available in the market can create different topographies and mechanical interlocking, subsequently impacting the resin cement SBS. Also, another essential point is that there should be a balance between the coefficient of thermal expansion (CTE) of low-fusing glaze and zirconia so that the bond between the two is not destroyed due to aging. In Sura et al.'s study, which matched the current study, adding a glazed layer followed by etching with hydrofluoric acid and applying silane compared to unglazed zirconia enhanced the SBS of resin cement after aging.[30] According to Cheung et al.'s research, which is not matched with the present study, the application of two glazed layers to sintered zirconia compared to the control, sandblasted, and Rocatec by 110-micron silica particle groups increased SBS of Panavia F2 resin cement after thermocycling.[18] Even Rocatec had a statistically significant increase in SBS compared to sandblasting, suggesting the great effect of the presence of glass phase compared to sandblasting with aluminum oxide in bonding improvement and enhancement. The zirconia hardness is created due to its phase change from the unstable tetragonal form to the stable monoclinic form. During this phase change, the energy absorbed by the zirconia matrix in the proximity of the expanding crack is consumed to turn into the monoclinic phase, which accompanies a volume increase of 3-4% and prevents crack expansion.[31,32] Sandblasting creates minor defects in zirconia and compressive stress points, changes its phase, and increases the bending strength of zirconia; however, it should be noted that this phase change can generally take place only once and is an irreversible process.[33,34] Ozcan suggests not using sandblasting with aluminum oxide particles above 50 microns to hinder phase change, and the minimum pressure required to spray the particles on the zirconia surface should be 2.5 bar. [33] This issue has not been confirmed by Aurelio [35], believing that sandblasting enhances the bending strength of zirconia, and also the size of the particles, the pressure used, and the sandblasting process duration have no effect on the phase change in zirconia. Therefore, the results, in this case, are contradictory, but it can be used as a An alternative solution to remove the sandblasting process due to the probable effects on the phase change in zirconia from new strategies, such as applying and baking low-fusing glaze or applying a ceramic liner for zirconia preparation in order to improve SBS of this material. Moreover, since the current research showed that the SBS of sandblasted and glazed zirconia had no statistically significant difference, this strategy can be employed for adhesion under clinical conditions. Due to the low SBS in the control group, it can be concluded that although the resin cement containing MDP establishes a chemical bond with zirconia and other metals, this bond lacks enough strength to deal with thermocycling conditions and simply causes the sample to debond. Thus, in order to create optimal bonding in the intraoral environment that has been simulated by thermocycling in laboratory conditions, in addition to chemical bonding, micromechanical bonding must also be established. Everson et al. investigated the effect of applying five different types of ceramic glazes with a coefficient of thermal expansion (CTE) in the range of zirconia coefficient of thermal expansion on the zirconia surface and compared their effects with the zirconia group under the tribochemical process by CoJet on SBS of resin cement and concluded that after thermocycling, SBS of all five glazed zirconia groups was higher than that of the tribochemical group and no statistically significant difference was observed between different glazing materials. [36] Their study is inconsistent with the present study. One of the reasons for this inconsistency may be the different SBS testing methods, such as using a flat blade to apply force and cementing the composite cylinder on the zirconia surface, not using bonding in their study, and the use of self-adhesive resin cement. Notably, the use of various tools for applying force, such as chisels, looped wire, etc., as well as the amount of force applied for failure, leads to different results in the studies. In their study, RelyX Unicem resin cement was used, which lacks MDP molecules in its structure. However, this issue had no effect on the results of this study, and although this cement was used in all groups, SBS was higher in the glazed group than in the tribochemical group. It may be concluded that the impact of the micromechanical bond formed in zirconia preparation is much greater than the chemical bond formed by resin cement. Concerning the silane effect, results similar to those of resin cement can be achieved. In the current research, the Synergistic effects of silane and Panavia cement, which contains MDP, were investigated in the control, glazed, and sandblasted groups, and it is not clear whether the increased SBS obtained is due to the micromechanical bond stemming from glaze and sandblast or due to the chemical bond created by the interactions of silane and the presence of MDP molecule in Panavia resin cement. It may be concluded that the Synergistic effect of mechanical and chemical preparation methods for the zirconia surface can enhance SBS. In order for the glaze to be better accepted by the zirconia surface, the coefficient of linear thermal expansion of both materials should be consistent with each other, as the coefficient of linear thermal expansion of the zirconia block in the present study was 10.4±0.5×10-6/k (25-500°C) and the coefficient of linear thermal expansion of glaze paste was 9.3±0.5×10-6/k(25-TG°C); accordingly, both materials were almost in the same range and were consistent, which is also observed in Everson et al.'s [36] study. In current research, the surface in the control and sandblasted groups was cleaned with phosphoric acid for 10 seconds to remove probable debris. This action can give rise to increasing the surface free energy and increasing the surface acceptance for better bonding and wetting; however, as seen, although universal adhesives and Panavia F2 cement, which contain MDP in their composition and have the ability to bond to zirconia, were used, the obtained results demonstrated that the chemical bond itself is not enough to bond resin cement to zirconia, but other factors are also involved

Conclusion

Applying a low-fusing glazed layer to prepare the zirconia surface culminated in enhancing the SBS of dual-cured self-etch resin cement, equivalent to sandblasting by 50-micron aluminum oxide, after the thermocycling process. No statistically significant difference was observed between the GBU and ABU universal adhesives in increasing the resin cement SBS.

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Author Contributions:
E.Y. conceptualization, data curation; S.K. formal analysis, investigation; S.K. and E.Y. Methodology; E.Y. project administration; S.K. resources; E.Y. Supervision; S.K. validation; E.Y. visualization; S.K. writing – original draft, writing – review & editing.
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Ethical Approval Code: This study was registered by the Ethics Committee of Hamedan University of Medical Sciences (IR.UMSHA.REC.1399.739).
Informed Consent Statement:

This article is in_vitro study and it has no informed consent.

Data Availability Statement:

The data are original and we cannot share them for ethical reasons. Upon request from the corresponding author, we can share the data with reasons.

Conflict of Interest: No Conflict of Interest Declared ■

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