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The Effect of Bioglass Coating on Microshear Bond Strength of Resin Cement to Zirconia

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A B S T R A C T

Objectives: Durable bonding to zirconia is a challenging issue in dentistry. This study aimed to assess the effect of bioglass coating of zirconia on the microshear bond strength of resin cement to zirconia and to study the effect of thermocycling on this bond.

Materials and Methods: This in-vitro experimental study was conducted on 60 yttria-stabilized tetragonal zirconia blocks in six groups (N=10) based on surface pretreatment and thermocycling. Surface pretreatments included no treatment control, alumina particle abrasion, and bioglass-coating of zirconia. Resin bonding was performed with Panavia F2.0 cements. Then, half of the specimens underwent a 24-hour incubation in 37°C water, while the other half were subjected to thermocycling (12000 cycles, 5-55°C, 60s for each batch) following the same incubation period. Subsequently, the microshear bond strength of the specimens was measured. Additionally, one block from each group was subjected to scanning electron microscopy and X-ray diffraction. The data were analyzed using Kruskal-Wallis and Mann-Whitney U tests.

Results: There was a significant difference between the bond strength values of different groups (P<0.001). Alumina particle abrasion and bioglass coating equally increased the bond strength compared to the untreated control group (P<0.001). Thermocycling caused significant decreases in bond strength in all the groups (P<0.001); however, the bond strength value of the thermocycled bioglass-coated group was significantly higher than that reported for the thermocycled alumina particle abraded group (P=0.015).

Conclusion: Despite the decrease in the bond strength values after thermocycling, the long-term efficacy of the bioglass coating of zirconia was promising.

Keywords: Dental Bonding; Zirconium Oxide; Resin Cements; Bioglass

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INTRODUCTION

One of the advances in dental ceramics is the development of high-strength zirconia, which has superior fracture toughness and durability compared to other available mineral and nonmetallic alternatives [1]. Zirconia ceramic is currently used in several dental applications, such as the fabrication of orthodontic brackets, intracanal posts, abutments, single-unit crowns, and fixed partial dentures [2,3]. Despite the superior mechanical properties, the conventional adhesive techniques often do not provide adequate resin bond strength, and a strong bond via both micromechanical interlocking and chemical bonding to the ceramic surface is required [4,5].

Bonding to zirconia has been the subject of numerous studies in recent years [6,7]. Creating a rough surface to provide mechanical retention is hard to achieve. On the other hand,

it has been documented that hydrofluoric acid etching and silanization are not effective for zirconia ceramics [1,6]. To date, no consensus has been reached on a standard surface treatment to maximize the bond strength to zirconia. The commonly used techniques for this purpose in the clinical setting include abrasion by burs or particles, use of phosphate monomers, and application of a tribochemical silica coating; however, the bond strength achieved through these techniques is much lower than that of conventional glass-ceramics [6].

In recent years, zirconia coating with ceramics has been suggested to enhance the resin bond strength. A recent systematic review concluded that ceramic coating of zirconia provides the highest long-term bond strength [8]. This layer serves as a chemically reactive surface that can be etched and primed [9,10]. Different types of ceramic coatings have been used for this purpose [8, 11-13], however, due to the lack of studies in this regard, further investigation of ceramic coatings on the inner surface of zirconia is encouraged [8]. In some studies, bioglass coatings on zirconia implants have been successfully used to benefit from the bioactive properties of bioglass and the mechanical properties of zirconia [14, 15]. In a previous study, the capability of this type of coating on zirconia for adhesive purposes was analyzed, and the results showed that 45S5 bioglass created a SiO_2-based coating layer with a minimal thickness (46.67±23.6μm) on the surface of the zirconia substrate, which could be etched by hydrofluoric acid. Furthermore, using a simple firing technique to create this intermediate layer on the zirconia surface is in favor of its efficacy for routine uses in dentistry [9].

On the other hand, it has been shown that aging decreases the bond strength of resin to many commercially available ceramic materials [16], so in order to simulate oral conditions, aging should be considered in bond strength studies [17]. Therefore, the present study aimed to evaluate the long-term efficacy of bioglass coating of zirconia by analyzing the effect of thermocycling on the bond strength of resin cement to bioglass-coated zirconia. The null hypothesis was that thermocycling would have no significant effect on the bond strength of resin cement to bioglass-coated zirconia.

MATERIALS AND METHODS

Surface treatment:

This in-vitro experimental study was ethically approved by the ethics committee of Shahid Beheshti University of Medical Sciences, according to the Declaration of Helsinki, under the code of IR.SBMU.DRC.1398.052. The study was conducted on 60 sintered yttria-stabilized tetragonal zirconia polycrystal blocks (ICE Zirkon, ZirkonZahn, Italy), measuring 10×2×7 mm. This material contains 4-6% Y_2O_3, < 1% Al_2O_3, max. 0.02% SiO_2, max. 0.01% Fe_2O_3, and max. 0.04% Na_2O [18]. All zirconia blocks were cleaned using 98% ethanol (Bidestan, Tehran, Iran) in an ultrasonic bath for 10 minutes.

The specimens were randomly divided into six groups (N=10) based on the surface pretreatments and subjecting to thermocycling. The groups were untreated zirconia without thermocycling as the control group (UC), alumina particle abraded zirconia without thermocycling (AA), bioglass-coated zirconia without thermocycling (BG), untreated zirconia subjected to thermocycling (UT), alumina particle abraded zirconia subjected to thermocycling (AAT), and bioglass-coated zirconia subjected to thermocycling (BGT).

A sample size of 10 was obtained using Power and Sample Size Calculation software (version 2.1.31), considering a significance level of less than 0.05 and a power of 80% to detect a difference of 4 units in bond strength values between groups, and a standard deviation of 3.

The zirconia blocks in the AA and AAT groups were abraded with 50μm aluminum oxide particles (True Etch, OrthoTechnology, FL, USA) under 40 PSI pressure at a 10 mm distance for 15s at an angle of 90° by a microetcher (Microetcher, Danville Engineering, CA, USA). Based on a study carried out by Zhang et al., the highest bond strength was achieved at 0.2 and 0.3 MPa; however, the mechanical properties were dramatically affected at high pressures. Due to the aforementioned findings, a pressure between the two values of 0.27 MPa or 40 PSI was chosen in the present study [18]. Then, the abraded specimens were cleaned in 98%

ethanol in an ultrasonic bath for 10 minutes in order to eliminate the loose particles on the zirconia surface following sandblasting.

For the surface treatment of the BG and BGT groups, bioglass slurry was prepared by mixing 500µg of bioglass (manufactured by the Materials and Energy Research Center of Iran), 1 cc of water, and 1 cc of polyvinyl alcohol binder (PVA, Merck, Germany), similar to a previous study [9]. The bioglass used in the present study was 45S5 bioglass (45 wt% SiO2, 24.5 wt% Na2O, 24.5 wt% CaO, and 6 wt% P2O5). The slurry was applied on the top surface of the zirconia blocks using a microbrush (TPC Advanced Technology, CA, USA). The samples were then heated in a furnace at a rate of 100°C/h up to 1,200°C and remained at this temperature for 2 h. Subsequently, the samples were cooled at a speed of 200°C/h. The surface of the coated blocks was etched with hydrofluoric acid (Ultradent Porcelain Etch, Ultradent Products Inc., UT, USA) for 60s, rinsed, and dried with an air spray for 90s. Silane was then applied on the surface as recommended by the manufacturer and allowed to dry.

After the pretreatments of the specimens, Tygon tubes (Norton Performance Plastics, OH, USA) with a 0.7 mm internal diameter were placed on the surface of the blocks for the application of Panavia F2.0 cement (Kuraray Medical Inc, Tokyo, Japan). The tubes were filled with cement and then light-cured with a diode light-curing unit (Radiplus, SDI Ltd, Australia) for 40s. Afterward, the Tygon tubes were gently removed, and the zirconia blocks with cement rods on their surfaces were immersed in distilled water and incubated at 37°C for 24h. After the initial incubation period, the specimens in the groups planned to receive thermocycling were placed in a thermocycler (Dorsa, Tehran, Iran) and subjected to 12,000 thermal cycles at 5-55°C for 60s per cycle in order to be artificially aged.

Bond Strength Testing:

The specimens were transferred to a microtensile tester (Bisco Inc., IL, USA) for the measurement of microshear bond strength. By vertically soldering the cast cylinders to one jaw (compartment) of the device (Figure 1), the tensile load was converted to shear load [9]. The load was applied at a crosshead speed of 1 mm/min, and the load at failure was recorded. Then, the microshear bond strength was calculated using the following formula:

$S= F(N) / A(mm^2)$

where S is the shear bond strength, F is the load at failure in Newton, and A is the surface area in square millimeters.

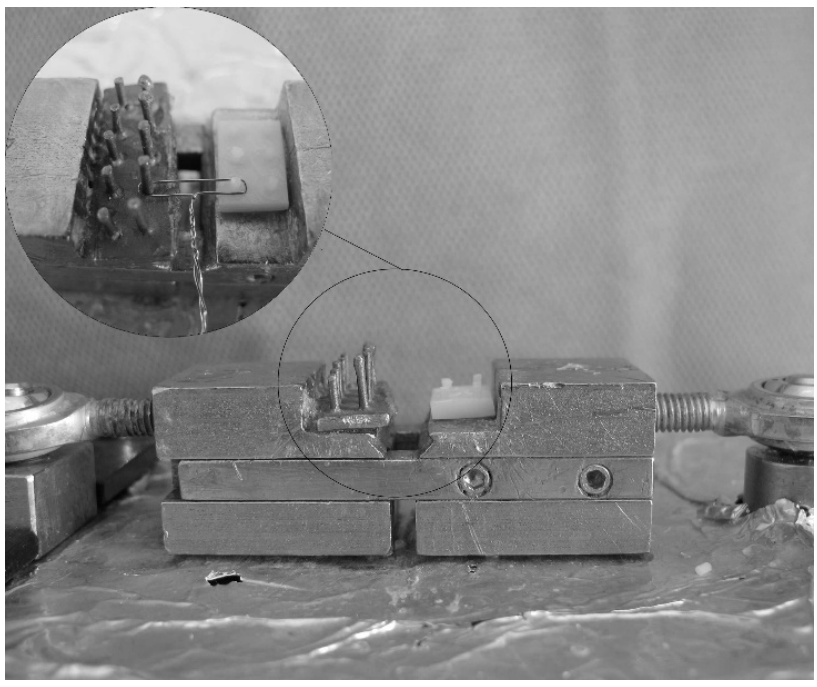

Fig. 1. Adaptation of the microtensile testing machine to function as a microshear tester

Scanning Electron Microscopy and X-ray Diffraction:

In each group, one block was subjected to scanning electron microscopy (SEM) and X-ray diffraction (XRD) analysis for the assessment of the surface and its crystalline structure.

Statistical Analysis:

The mean, standard deviation, median, and maximum and minimum values of microshear bond strength in different groups were reported. The distribution of the data was checked using the Kolmogorov-Smirnov test, which rejected normal distribution (P=0.021). Therefore, the non-parametric Kruskal-Wallis test was used to evaluate the difference among the groups, and then the Mann-Whitney U test was applied for the pairwise comparisons of the groups.

RESULTS

Bond Strength:

Table 1 tabulates the microshear bond strength values in the study groups. The Kruskal-Wallis test showed a significant difference among the groups (P<0.001). The pairwise comparisons of the groups by the Mann-Whitney U test are summarized in Table 1. In the comparison of short-term bond strength values, it was observed that both alumina particle abrasion and bioglass coating increased the bond strength, compared to that reported for the UC group (P<0.001). In addition, there was no significant difference (P=0.74) between these two groups (*i.e.,* AA and BG).

The groups subjected to thermocycling showed a significant decrease in bond strength. However, the bond strength of the BGT group was significantly higher than that of the UC (P<0.001) and AAT (P=0.015) groups. The bond strength of the AAT group was not significantly different from that reported for the UC group (P=0.912).

Figures 2 and 3 illustrate the SEM micrographs of the surface of the specimens in the six groups at ×1000 and ×5000 magnification, respectively. The SEM micrographs show the lines created by the abrasive in the UC group, the formation of a crack-free bioglass coating on the surface in the BG group, and a roughened surface in the AA group. In the groups subjected to thermocycling, no change was observed in the UT and AAT zirconia, compared to their counterparts without thermocycling. In the BGT group, some tiny crystals were seen in the coating layer.

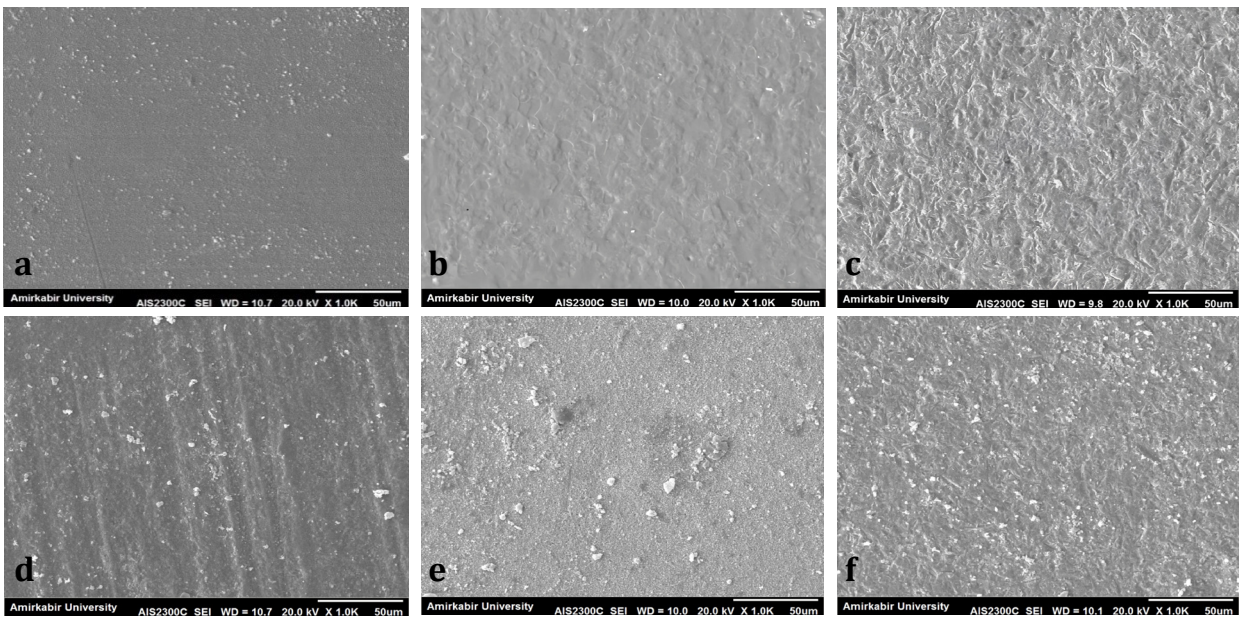

Fig. 2. Scanning electron microscopy micrographs of samples at x1000 magnification a. untreated zirconia without thermocycling b.bioglass-coated zirconia without thermocycling c. alumina particle abraded zirconia without thermocycling d. untreated zirconia subjected to thermocycling e. bioglass-coated zirconia subjected to thermocycling f. alumina particle abraded zirconia subjected to thermocycling

Table1. The microshear bond strength values (MPa) in the six study groups

Groups	Mean(Standard deviation)	Median	Minimum	Maximum
untreated zirconia without thermocycling (UC)[a]	8.6(4.5)	7.0	3.2	17.6
bioglass-coated zirconia without thermocycling (BG)[b]	35.8 (15.3)	35.2	11.2	60.8
untreated zirconia subjected to thermocycling (UT)	34.2 (9.5)b	33.6	21.2	49.6
alumina particle abraded zirconia subjected to thermocycling (AAT)[a]	-	-	-	-
bioglass-coated zirconia subjected to thermocycling (BGT)[c]	10.2(7.5)	8.4	2.4	26.8
bioglass-coated zirconia without thermocycling (BG)[b]	17.8(7.7)	17.6	8.4	37.6

*Groups marked with different superscript letters had significantly different bond strength values based on the results of Mann Whiteny U test ($p<0.05$).

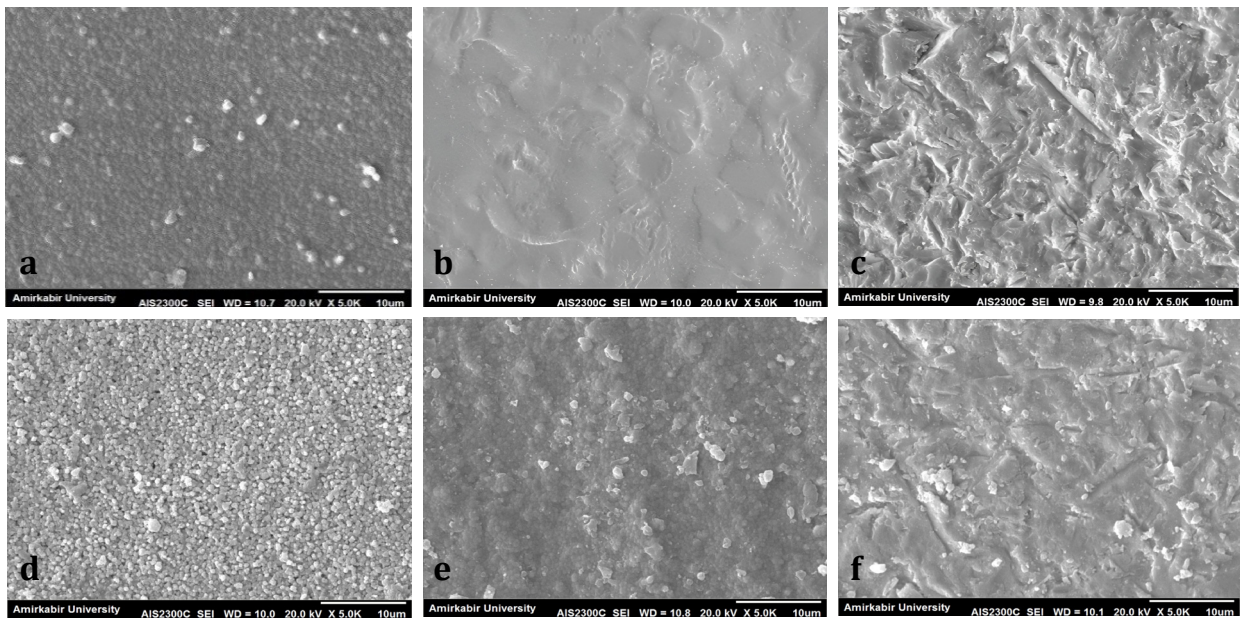

Fig. 3. Scanning electron microscopy micrographs of samples at x5000 magnification a. untreated zirconia without thermocycling b.bioglass-coated zirconia without thermocycling c. alumina particle abraded zirconia without thermocycling d. untreated zirconia subjected to thermocycling e. bioglass-coated zirconia subjected to thermocycling f. alumina particle abraded zirconia subjected to thermocycling

X-ray Diffraction:

The tetragonal phase was similar in all of the non-thermocycled groups with peaks at 20, 30, 34-36, 50, 51, 59-61, 63, 73, and 75. Similar peaks were noticed in the groups subjected to thermocycling, with the difference of an additional peak appearing at 2θ, 43. In the BG and BGT groups, the cubic phase peaks were reported at 2θ, 30, 36, 60, 63, and 74. The tetragonal phase was less in air particle abrasion and bioglass coating, compared to that reported for the control group. The amount of tetragonal phase in the UT and AAT groups was greater than that of the UC and AA groups, respectively; however, the amounts of the tetragonal and cubic phases were lower in the BGT group in comparison to those reported for the BG group (Figure 4).

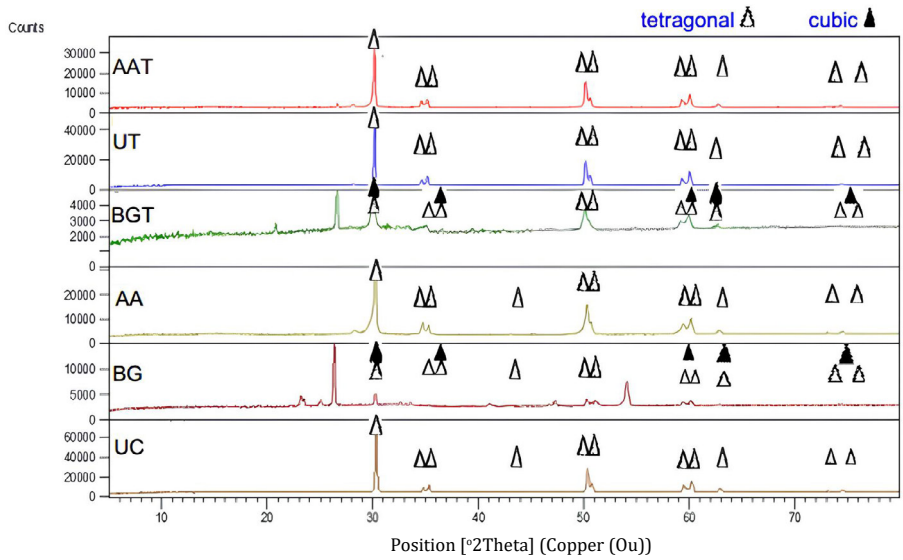

Fig. 4. X-ray diffraction: From top to bottom: alumina particle abraded zirconia subjected to thermocycling (AAT); untreated zirconia subjected to thermocycling (UT); bioglass-coated zirconia subjected to thermocycling (BGT); alumina particle abraded zirconia without thermocycling (AA); bioglass-coated zirconia without thermocycling (BG); untreated zirconia without thermocycling (UC)

DISCUSSION

Within the study findings, this bioglass coating of zirconia increased the 24-hour bond strength of resin cement to zirconia to the same level as air particle abrasion. Following artificial aging by thermocycling, the bioglass coating, like all other groups, showed significant decreases in bond strength values, so the null hypothesis was rejected.

In several studies, the bioglass coating of zirconia substrates has been applied to benefit from the properties of bioglass [14, 15]. Bioglass particles are expected to fill the gaps and porosities on the zirconia surface and decrease internal porosities, enhancing the mechanical strength and fracture toughness [19]. Considering the success of the bioglass coating of zirconia [9, 14, 15], this coating was used as an intermediate etchable layer in the present study, aiming to enhance the bond strength of resin cement to zirconia. In a previous study, two methods of applying bioglass on the top surface of the zirconia substrate in order to coat it were used, including the use of bioglass powder itself or the bioglass slurry. Since both methods had been successful in creating a coating layer after sintering, and since the slurry method led to a thinner layer of coating [9], the slurry method was used in the present study. Despite the thin coating thickness in this method, the uniform coating is still important for clinical applications. The CAD/CAM technology has the potential to consider the thickness of the internal coating to achieve a perfect fit and enhance the seating of the restoration [1]. However, other investigations are recommended to focus on the methods for applying a uniform coating with minimal thickness and the effect of this layer on the mechanical strength of zirconia.

In this study, the effect of bioglass coating was compared to that of alumina particle abrasion, which is a commonly used technique in most relevant studies [21]. The findings revealed that the short-term bond strength was comparable in alumina particle abraded and bioglass coated groups, and both values were higher than the value in the control group. The same increase in bond strength has also been reported in some other studies using intermediate layers, such as a glaze layer [22, 23], glass micro-pearls [24], and hydroxyapatite [13].

In the present study, thermocycling caused a significant reduction in the bond strength of all the groups; accordingly, all the samples of the control group failed prior to the measurement of bond strength. Nonetheless, the bond strength of the BGT group was still significantly

higher than that reported for the UC or AAT groups. In a study carried out by Vanderlei et al., the groups receiving glaze and silane coatings showed a significant reduction in their bond strength after aging; however, they still had higher bond strength values, compared to the groups with other surface treatments [23].

In a study conducted by Cura et al., the bond strength of glazed and silanized zirconia did not demonstrate a significant reduction after 5,000 thermal cycles [22]. Despite the difference in the types of coatings used in previous studies and the current study, it may be stated that a more durable bond strength may be due to the presence of SiO2 in the formulation of bioglass and the positive efficacy of silanization in increasing the durability of the bond.

It has been shown that the bond strength of conventional resin cements containing bis-GMA significantly decreases after thermocycling of abraded zirconia [25,26]. The abrasion of zirconia, compared to that of metal, creates a rough surface with fewer undercuts, and the conventional resin cement bonding cannot resist thermocycling [26]. Nonetheless, some studies using MDP-containing resin cements, such as Panavia F2, reported that the bond strength to abraded zirconia did not significantly decrease after thermocycling [27] and even increased in some cases [25]. This finding indicates that MDP chemically bonds to zirconia, and this bonding is enhanced by an increase in temperature. However, this result should be interpreted with caution since it may be due to a lower number of cycles in those studies. Based on the evidence, it has been demonstrated that this chemical bond is not stable in longer cycles, and finally, a reduction in bond strength may occur even in the use of MDP-containing cements. Valandro et al. showed that the chemical bond of hydroxyl groups of zirconia to MDP is not stable after 1,200 thermal cycles [28]. In the present study, despite the use of an MDP-containing cement, the bond strength of resin cement to alumina particle abraded zirconia was significantly lower in the thermocycled one, which is in line with the findings of a study by Attia [29]. In a study carried out by Ozcan et al., the bond strength of Panavia F2 resin cement to alumina particle abraded samples decreased to 0 after 6,000 thermal cycles [30]. The difference in the results of studies may be due to the number of cycles, different degrees of roughness created by particle abrasion methods, and the hardness of zirconia samples used in different studies.

According to the results of XRD, trace amounts of the cubic phase were observed in the bioglass-coated samples. The cubic phase is observed in zirconia samples sintered at 1400-1550°C or higher temperatures. It is a yttrium-rich phase and decreases the amount of yttrium in the adjacent tetragonal zirconia [31]. In addition, zirconia samples sintered at temperatures higher than 1400°C indicate a biphasic cubic and tetragonal structure [32]. The presence of the cubic phase, since it collects yttrium, results in higher susceptibility of the adjacent tetragonal phase to transformation. As a result, high-temperature treatments are sometimes recommended to produce tough and strong zirconia [31]. Nonetheless, higher amounts of the cubic phase increase the transformation nuclei [33] and result in eventual aging [34]. Along with this, it is reported that the variable amount of cubic phase in the zirconia structure is related to lower strength and toughness [35], so the presence of the cubic phase in bioglass-coated groups may make the structure susceptible to aging and lower mechanical strength.

Furthermore, comparing the alumina particle abraded and bioglass-coated groups with the control group revealed a reduction in the tetragonal phase in the former two groups, indicating stress application and induction of tetragonal to monoclinic phase transformation in both groups; therefore, it is required to carry out further studies in this regard. Hjerppe evaluated the effect of thermocycling and observed that the amount of the monoclinic phase increased with an increase in thermal cycles in the glazed group. However, no phase transformation was observed when non-coated samples underwent thermocycling [36]. Some other studies reported tetragonal to monoclinic phase transformation and reduction of the tetragonal phase after alumina particle abrasion [18,37,38]. It was shown that after thermocycling of abraded zirconia at 1,200°C, this phase transformation gradually reverses,

and monoclinic phase content decreases [37]. This finding can partly explain the increase in the tetragonal phase in the UT and AAT groups in the present study. In a study conducted by Perdigao et al., an increase in the monoclinic phase occurred after thermocycling. This difference can be due to higher thermal cycles and lower yttria content in the aforementioned study, compared to those reported for the current study [39].

The assessment of the effect of thermocycling on the phases of bioglass-coated zirconia revealed a reduction in both the tetragonal and cubic phases. Regarding the limitations of the present study, it may be assumed that in the coated groups, due to the presence of two layers and differences in the coefficients of thermal expansion, thermal cycles could have induced stress and resulted in a monoclinic phase transformation. Despite positive results in terms of bond strength, the presence of the cubic phase and reduction of the tetragonal phase induced by thermocycling of coated zirconia can enhance zirconia aging. This finding necessitates performing further studies in this regard. The SEM assessment of the coating layer after thermocycling showed a crystalline structure, which can be due to the formation of crystals as a result of thermal cycles.

CONCLUSIONS

Within the limitations of this study, bioglass coating of zirconia increased the 24-hour bond strength of resin cement to zirconia to the same degree as did alumina particle abrasion. Following artificial aging by thermocycling, bioglass coating yielded higher bond strength than did alumina particle abrasion.

CONFLICT OF INTEREST STATEMENT

None declared.

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