Clinical and Experimental Health Sciences

In Vitro Investigation of Shear Bond Strength of Titanium Alloy Bonded to Monolithic Zirconia Prepared Via Different Surface Roughening Methods Using Different Cements

Gülay Çakır, Zeliha Şanıvar Abbasgholizadeh, Yılmaz Umut Aslan

Marmara University, Faculty of Dentistry, Department of Prosthodontics, Istanbul, Türkiye.

Correspondence Author: Gülay Çakır
E-mail: glycakir@hotmail.com
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ABSTRACT

Objective: To evaluate the shear bond strength (SBS) of yttrium-stabilized tetragonal zirconia polycrystals bonded to titanium alloys via different surface treatment methods using four different cements.

Methods: Eighty titanium and monolithic zirconia discs were prepared with computer-aided design/manufacturing (CAD/CAM) technology. All titanium discs and 40 of monolithic zirconia discs were polished by using silicon carbide paper and sandblasted with 50 µm aluminum oxide (Al_2O_3). Tribochemical silica coating was applied to remaining 40 monolithic zirconia discs. The monolithic zirconia discs were divided into eight groups after surface treatment (n=10). Titanium discs were cemented using conventional glass ionomer cement (GIC), resin-modified GIC, self-adhesive resin cement, and dual-cure resin cement. The SBS test was performed using a universal testing machine. The failure patterns were examined by using a scanning electron microscope (SEM). Data were statistically analyzed with one-way analysis of variance (ANOVA), two-way ANOVA and Tukey's test (α<.05).

Results: The SBS values differed according to the surface treatment methods and cements used (p<.001). The highest and lowest SBS values were measured in the tribochemical-silica-coated G-CEM ONE (34.77±5.53 MPa) and Al_2O_3 sandblasted GC Fuji I (3.30±0.77 MPa) cement groups, respectively. Failure analysis revealed that 41.25%, 31.75% and 25% of the failures were cohesive, adhesive, and combined failures, respectively.

Conclusion: The SBS values between the monolithic zirconia and titanium alloy were significantly higher in the resin cement groups containing 10-methacryloyloxydecyl dihydrogen thiophosphate and 10-methacryloyloxydecyl dihydrogen phosphate (p<.05). While adhesive and combined failures were observed at high SBS values, cohesive failures were detected as the bonding values decreased.

Keywords: Cements, shear bond strength, surface roughness, monolithic zirconia, tribochemical silica coating

1. INTRODUCTION

Titanium, which is frequently used in the fabrication of dental implant abutments, has several advantages such as biocompatibility, resistance to abrasion, and sufficient mechanical durability (1). However, the metallic-gray color of the titanium alloys creates an aesthetic problem, especially in submucosal peri-implant tissues (2). Although zirconia abutments can overcome these aesthetic disadvantages, drawbacks such as failure of the implant-abutment junction area and wear at the implant connection limit their clinical use. To eliminate these issues and achieve more aesthetically pleasing results, hybrid abutments have been developed. A hybrid abutment consists of two components: a prefabricated titanium-based substructure, and a zirconia or lithium disilicate ceramic superstructure. The ceramic superstructure is bonded to the titanium base abutment using cement (1,2,3,4,5,6,7). Thus, the advantages of the two materials include a combination of the durability of titanium and the aesthetic properties of ceramic materials (8). According to previous studies, the clinical success of hybrid abutments depends on the cementation technique (4,7,8).

It has been reported that conventional and resin cements have been used in the cementation of hybrid abutments. (9,10). Resin cements are the material of choice for the cementation of fixed implant restorations due to their high bonding ability with metals and ceramics, wide aesthetic color options, favorable mechanical properties, high strength, superior retention, and low solubility in the oral cavity (9,10). Moreover, in the presence of multifunctional monomers such as 10-methacryloyloxydecyl dihydrogen thiophosphate (10-MDTP) and 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP), the surface wettability of the material increases and crosslinking occurs with the methacrylate groups of the resin cement (11).

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Various surface modification methods such as sandblasting, tribochemical silica coating, hydrophilic acid etching, and laser-based methods are recommended to form a strong mechanical and chemical retention between the resin cement and the ceramic (11,12).

Sandblasting creates a rough surface for the mechanical retention of the cement. Simultaneously, it increases the strength of monolithic zirconia and prevents the spreading of cracks by acting on the compressive stress layer (12). The tribochemical silica coating process not only produces roughness but also chemically activates the ceramic surfaces. As a result of the blasting pressure, the embedded silica and aluminum oxide (Al_2O_3) particles and the binding silane agent react chemically.

The aim of this study was to evaluate the shear bond strength (SBS) between monolithic zirconia and titanium materials that were treated using different surface-roughening methods and to which traditional glass ionomer cement (GIC), resin-modified GIC, dual-cure resin cement and self-adhesive resin cement were applied in vitro. The null hypothesis in this study was that there would be no significant difference in SBS values between the tested cements. The second null hypothesis was that different ways of surface treatment would have no effect on adhesion.

2. METHODS

According to the results of an analysis based on G*Power version 3.1.9.2, (Heinrich – Heine-Universität Düsseldorf, Germany, power = 0.95, a = 0.05, b = 0.05) using on the data of a study (13), the number of speciments to be included in the study in each group was determined to be 10 (Fig. 1). Eighty monolithic zirconia discs with a height of 10 mm and a diameter of 7 mm (GC Initial Zirconia UHT, Tokyo Japan) were fabricated using the with CEREC inLab program (CERECMCX5 Software 18.1 DentsplySirona, Bensheim, Germany), and 80 titanium discs (CopraTi-5 Whitepeaks, Essen, Germany) with a height of 8 mm and a diameter of 12 mm were fabricated by using Dentifa PRO2 (Professional Dental CNC, Istanbul, Türkiye).

The titanium discs were polished using silicon carbide papers of different grit sizes, of P600, P1200 and P2400 (Minitech 233 Presi, Eybens, France), while the monolithic zirconia discs were polished by using P600, P800 and P1200 grit silicon carbide papers (Minitech 233 Presi, Eybens, France) with water cooling. Then, they were cleaned in an ultrasonic cleaner for 5 minutes (14,15).

All the titanium discs and 40 zirconia discs were sandblasted with 50 µm Al_2O_3 particles (Cobra Aluminum Oxide White, Renfert, Germany) under a pressure of 2 bar from a distance of 10 mm for 15 seconds and air dried for 10 seconds. A tribochemical silica coating (CoJet Sand, 3M ESPE, Seefeld, Germany) was applied to the remaining 40 zirconia specimen surfaces from a distance of 10 mm under a 2.8 bar air pressure for 15 seconds. Subsequently, silane (CoJet 3M-ESPE Sil, Seefeld, Germany) was applied for 15 seconds and allowed to dry for 5 minutes (16).

After the surface treatments, the specimens were divided into eight subgroups according to the cement type (n=10).

Conventional GIC (Fuji I, GC, Tokyo, Japan) was applied to the specimen surface (G4, G8) according to the instructions specified by the manufacturer. Resin-modified GIC (FujiCEM Evolve, GC, Tokyo, Japan) was applied to the center of the prepared surface using an automatic tip (G1, G5).

A dual-cure adhesive resin cement (G-CEM LinkForce, GC, Tokyo, Japan) was applied in combination with a universal primer (G-Multi Primer, GC, Tokyo, Japan). The primer was applied to both the titanium and the zirconia surfaces for 15 seconds and air dried for 10 seconds. Then, cement was applied using an automatic tip. (G2, G6).

Self-adhesive resin cement (G-CEM ONE, GC, Tokyo, Japan) was used in combination with an adhesive-enhancing primer (GC AEP, Tokyo Japan). Primers were applied to both surfaces for 10 s and air dried for 5 s. (G3, G7). The cement was applied by using an automatic tip. All resin cements were polymerized for 40 seconds using a 1200 mW/cm² light-emitting diode device (LED Rainbow Curing Light, MDD, Voco, Germany) (Fig.1). During the polymerization, all specimens were maintained under a constant force of 5 Newton (Table 1).

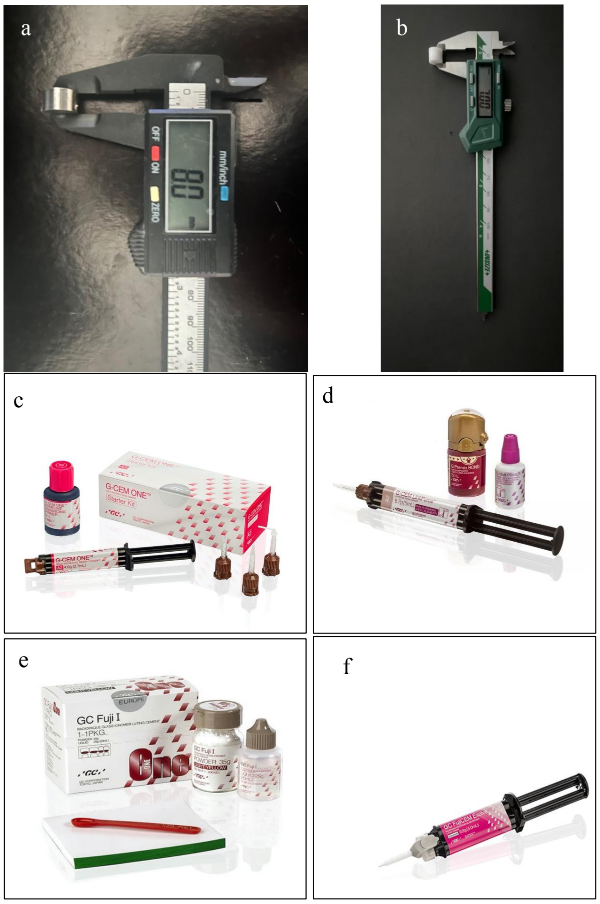

Figure 1. *Titanium disc (a), Zirconia disc (b), G-CEM ONE cement (d), G-CEM LinkForce cement (d), GC Fuji I cement (e), FujiCEM Evolve Cement (f)*

After cementation, all the specimens were placed in a dry-air incubator (Nüve EN 055, Akyurt, Ankara) with distilled water at 37 °C for 24 hours.

***Table 1**. Cements used in this study.*

Material	Main Components	Manufacturer
G-Cem LinkForce	Paste A: Bis-GMA, UDMA, DMA, initiator, pigments Paste B: Bis-MEPP, UDMA, DMA, initiator, Bis-EMA, dibenzoyl peroxide, BHT	GC Corp., Tokyo, Japan
G-Multi Primer	Vinyl silane, phosphoric methacrylate monomer, thiophosphoric ester monomer, methacrylic acid ester, ethyl alchol	GC Corp., Tokyo, Japan
G-CEM Adhesive-Enhancing Primer	Ethanol, 10-MDP, 10-MDTP 4-META, 2-hydroxy-1,3 dimethoxypropane, vanadyl acetylacetonate, 2,6-di-tert-butyl-*p*-cresol	GC Corp., Tokyo, Japan
G-CEM ONE	Paste A: Fluoroaluminosilicate glass, methacrylic acid ester, initiator Paste B: Silica filler, methacrylic acid ester, phosphoric methacrylate monomer, initiator	GC Corp., Tokyo, Japan
Fuji 1	Powder: fluoroalumino silicate glass Liquid: polyacrylic acid, distilled water, silica powder, polycarboxylic acid	GC Corp., Tokyo, Japan
FujiCEM Evolve	Paste A: HEMA, UDMA, Butyl hydroxytoluene, Stabilizer Paste B: Ytterbium trifluoride, Polyacrylic acid, Polybasic carboxylic acid, Quartz	GC Corp., Tokyo, Japan

Bis-GMA: bisphenol-A-glycidyldimethacrylate; Bis-EMA: ethoxylated bisphenol-A-dimethacrylate; MDP: 10-methacryloyloxydecyl dihydrogen phosphate; MDTP: 10-methacryloyloxydecyl dihydrogen phosphate HEMA: 2-hydroxyethyl methacrylate; 4-MET: 4-methacryloyloxyethyl trimellitate; MEPS: methacryloyloxyalkyl thiophosphate methylmethacrylate; UDMA: urethane dimethacrylate; DMA: N, N-dimethylacrylamide.

2.1. Shear Bond Strength Test

To fix the specimens during the SBS measurement, a plate of pure iron (30 mm ´ 10 mm ´ 10 mm), on which the titanium discs could be placed, was prepared (Fig. 2).

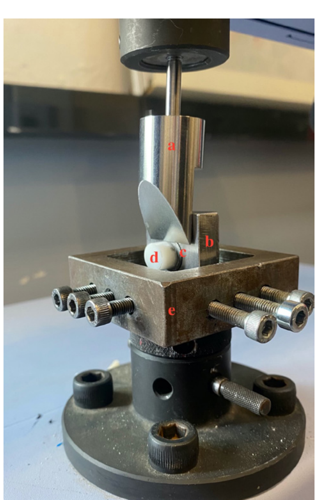

***Figure 2.** Shearing rod (a), The plate of pure iron (b), Titanium disc (c), Zirconia disc (d), Shear bond testing (e)*

The specimens were sequentially placed in a universal testing machine (Shimadzu Corporation, Tokyo, Japan). A blunt-tipped spacer was placed between the titanium and zirconia interfaces, and the specimens were loaded with a speed of 1 mm/min until the zirconia discs were separated from the titanium. The resulting values were obtained by dividing the applied force (N) by the bonded area (mm^2) and were recorded in megapascals.

2.2. Failure Analysis

After the specimens were failured and removed from the test apparatus, the failure sites were observed under a stereomicroscope at 30´ magnification to identify the type of bond failure. Failure types were grouped as adhesive failure (a) at the resin cement-titanium interface, cohesive failure (b) within the resin cement, and combined failure (c), which is a combination of adhesive and cohesive failure. To observe the topographic changes, the specimens were coated with gold (Quorum SC 7620 Sputter Coater, East Sussex, England) and examined using a scanning electron microscope (SEM; Zeiss Evo LS10, Germany) magnifications at 1000´, 2000´, and 5000´.

2.3. Statistical Analysis

Data were analyzed using the IBM Statistical Package for Social Sciences V23 software. Two-way analysis of variance (ANOVA) was used to compare the SBS according to the different etching methods and applied cements. One-way ANOVA was used for analysis of the failure type. Multiple comparisons were performed by using posthocTukey's significant difference test. The statistical significance was set at $\alpha < .05$.

3. RESULTS

The results of two-way ANOVA indicated that both surface treatment methods had a significant effect on the SBS values in the G2, G6 ($p = .013$) and G3, G7 ($p = .006$) cement groups ($p < .001$). A statistically significant difference was also observed between the mean SBS values of the different cement types ($p < .05$) (Table 2).

No significant differences were observed in the SBS values of the G1, G5 ($p = .821$) and G4, G8 ($p = 1.00$) cement types for the different surface methods ($p > .05$). Among all groups, G7 exhibited the highest SBS value (Table 2).

According to the failure-type analysis, 41.25% cohesive, 33.75% adhesive and 25% combined failures were observed (Fig. 3) (Table 3). While the cohesive failures were predominantly observed in G1, G4, G5 and G8 groups, adhesive and combined failures were observed in G2, G3, G6 and G7 groups. Cohesive failures were observed in GC Fuji I (G4, G8) cement when compared to G-CEM ONE (G3, G7) and G-CEM LinkForce (G2, G6) cements at a statistically significant level ($p < .001$) (Fig. 4 and Fig. 5).

Table 2. *Shear bond strength values (MPa)*

	Methods			
Cement	**Tribochemical Silane Coating**	**Aluminum oxide Sandblasting**	**p**	**Total Avarage**
GC FujiCEM Evolve	8.19 ± 1.96^{D,E} (G1)	5.85 ± 2.71^{E} (G5)	.821	7.02 ± 2.59^{d}
G-CEM LinkForce	18.55 ± 4.38^{C} (G2)	12.83 ± 2.29^{D} (G6)	.013	15.69 ± 4.49^{a}
G-CEM ONE	34.77 ± 5.53^{A} (G3)	28.64 ± 5.77^{B} (G7)	.006	31.70 ± 6.34^{b}
GC Fuji I	3.34 ± 1.14^{E} (G4)	3.30 ± 0.77^{E} (G8)	1.00	3.32 ± 0.95^{c}
Total Avarage	16.21 ± 12.7	12.65 ± 10.51	.025	14.43 ± 11.72

$^{A\_D}$ There is no difference between cements with the same letter; $^{A\text{-}E}$ No difference between cement and surface roughening method interactions with the same letter (p< .05)

Table 3*. Groups according to the failure type (n=10)*

	Failure Type		
Groups	Cohesive Failure	Adhesive Failure	Combined Failure
G1	7	0	3
G2	0	6	4
G3	0	7	3
G4	10	0	0
G5	7	0	3
G6	0	8	2
G7	0	6	4
G8	9	0	1
Total Average	41.25%	33.75%	25%

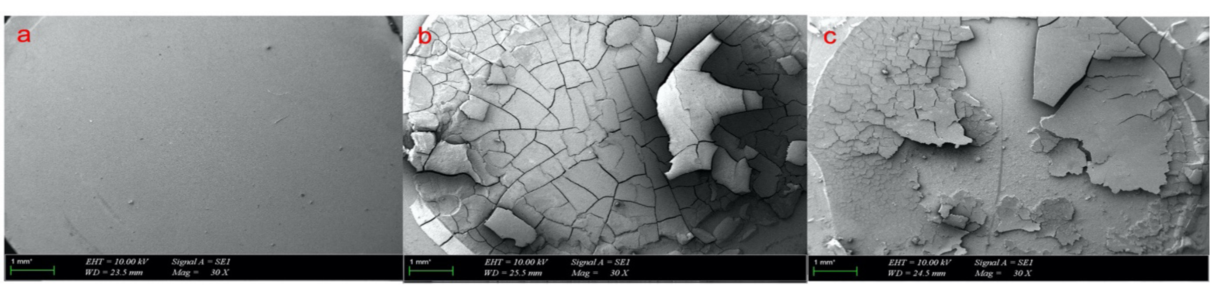

Figure 3. *Scanning electron microscope (SEM) images of failure types at magnification of 30´. (a) Adhesive failure; (b) Cohesive failure; (c) Mixed failure.*

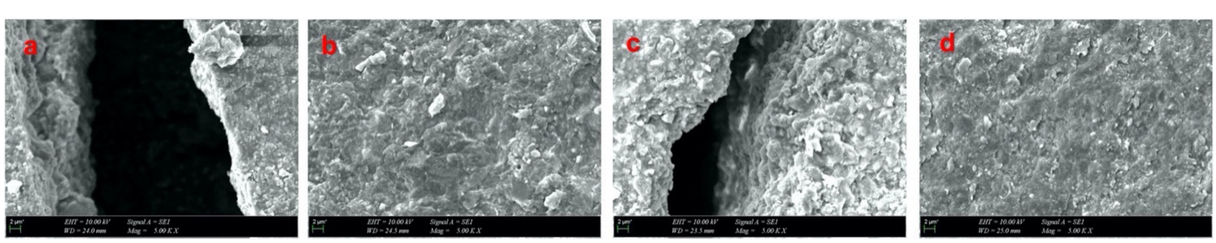

Figure 4. *Scanning electron microscope (SEM) images of Al2O3 sandblasted roughened titanium surfaces at a magnification of 5000´. (a) GC FujiCEM Evolve; (b) G-CEM LinkForce; (c) GC Fuji I; (d) G-CEM ONE.*

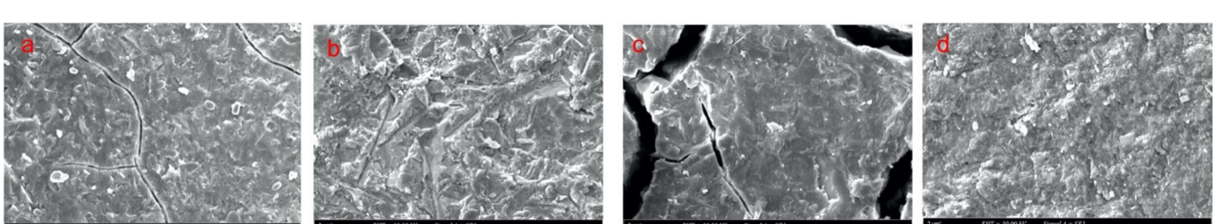

Figure 5*. Scanning electron microscope (SEM) images of titanium surfaces treated with CoJet at 5000´ magnification. (a) GC FujiCEM Evolve; (b) G-CEM LinkForce; (c) GC Fuji I; (d) G-CEM ONE.*

4. DISCUSSION

The study focused on the SBS of Y-TZP bonded to titanium alloys via various surface treatment techniques and cements. Considering the results obtained in this study, the first null hypothesis, that different types of cements do not affect the bonding to the titanium surface, was rejected. Tha statistical analysis revealed a significant difference between the study groups (p< .001). The second null hypothesis was rejected because different surface treatment methods affected the bond strength. The CoJet system was significantly more effective in the 10-MDTP and 10-MDP adhesive resin cement groups (p< .001).

Al_2O_3 particles (50 µm) have been used in many studies to increase surface roughness and increase the retention of titanium abutments with cements (6,12,17,18,19). Based on these studies, 50 µm Al_2O_3 particles were used for the surface treatment of all titanium surfaces in this study.

The strong and long-term bonding between cement and titanium depends on the constituents, properties, and bonding ability, as well as on the surface properties of the titanium. Therefore, four different categories of luting cements were used in our study.

The results of this study revealed that the SBS values of both Fuji I (G4, G8) and FujiCEM Evolve (G1, G5) groups were significantly lower than those of the adhesive resin groups (p< .001). Fuji I, a brand of GIC, provides advantages such as a low cost, relatively improved biocompatibility, fluoride release, and ease of manipulation (20). The results of our study are similar to the results of the study by Fawzy et al (6), where the Fuji I cement primarily exhibited cohesive failure and low SBS values. The FujiCem Evolve cement also showed similar results as the Fuji I cement in our study.

Sandblasting increases the bond strength by enhancing the surface area and roughness. Zhang et al. suggested that sandblasting reduces the strength of zirconia by causing microcracks. However, research has demonstrated that the resin cement infiltrates into the microcracks, thereby significantly increases the strength of the ceramic (12). Moreover, in the CoJet system, the silica particles not only roughened the surface but also promoted chemical retention through the bonding between the silane and the silica-coated zirconia surface (21). Previous studies have reported that finer micro-retentive grooves are observed in the SEM images of CoJet groups than in the groups with Al_2O_3 sandblasting (22,23,24,25). Thus, the CoJet group exhibits higher SBS values than the group with Al_2O_3 sandblasting. In this study, similar to the literature, the SBS values of the CoJet system were observed to be statistically high in the G-CEM ONE (G3, G7) and G-CEM LinkForce (G2, G6) cement groups.

Specimens containing 10-MDP-containing systems are recommended for the long-term adhesive durability of adhesion between monolithic zirconia and resin cement (26). Researchers have speculated that 10-MDP does not hydrolyze because it reacts with the hydroxyl groups on the ceramic surface, provides chemical bonding with zirconia, and contains a long carbonyl chain (11). Specimens treated with self-adhesive resin cement containing 10-MDTP and 10-MDP (G2, G3, G6, G7) showed higher SBS values than those treated with other cement types (27,28). In this study, the SBS was found to be significantly higher in the adhesive cement systems containing 10-MDTP and 10-MDP.

Because shear strength are the most dominant forces during chewing and other jaw movements (29,30,31), the SBS test, which is the most commonly used test method, was used in our study to evaluate the metal-resin bonding efficiency in vitro.

The type of failure also supports the SBS values (32,33). Altan et al. evaluated the SBS values between a monolithic zirconia material obtained by computer-aided design/manufacturing and resin cement obtained after different surface treatments and reported that cohesive failure were observed in groups with low SBS values, whereas combined and adhesive failures were observed in groups with high SBS values (33). In accordance with the findings of Altan et al., cohesive failure occurred in GIC and resin-modified GIC in this study.

The zirconia and titanium-zirconia bonding ability. The bonding ability of titanium and 3Y-TZP is still under investigation, and several bonding protocols consisting of different surface treatments, primers, and luting agents have been reported to have a significant influence on bonding to the zirconia surface. A number of factors (temperature, pH, saliva chemistry, food or drink interaction, presence of microorganism) may interfere bond strength. Śmielak et al. (34) reported greater retentive shear bond strength for polycarboxylate cement and zinc-oxide-eugenol cement compared with Panavia F.2, when the monolithic zirconia disc cemented onto the Ti disc. Shear bond strength values were found to be greater in the groups that were sandblasted with aluminum oxide. Adhesive failures were observed in the Panavia F.2 cement groups.

The simulation of intraoral conditions is significantly challenging in a laboratory setting. Moreover, the negative C-factor effect of the cements and the shrinkage of the resin cement after polymerization, which occurs in clinical conditions, could not be imitated. The thermal cycle, pH changes, and dynamic fatigue load, which were not evaluated in this study, may affect the durability of the resin bond. To confirm the data obtained from this in vitro study, clinical follow-up studies are required in the presence of chewing forces and in the oral environment that can affect the long-term stability of the resin bond.

Considering the results obtained in this study, the first null hypothesis, that different types of cements do not affect the bonding to the titanium surface, was rejected. Tha statistical analysis revealed a significant difference between the study groups (p<.001). The second hypothesis was rejected because different surface treatment methods affected the bond strength. The CoJet system was found to be significantly more successful in the 10-MDTP – and 10-MDP-containing adhesive resin cement groups (p<.001).

In this study, the post-thermocycling bond strength was not evaluated, this might be considered as a limitation. This matter should be investigated further by comparing differences in initial and postthermocycling bond strength values. Another limitation was that the titanium discs used to provide fundamental information on cement adhesion did not accurately represent the clinical situation of cement flow and distribution between titanium and zirconia surfaces. In addition, only monolithic zirconia material was used, different results might have been provided with different types of materials.

5. CONCLUSION

Within the limitations of this in vitro study, the following conclusions were drawn:

1. Conventional GIC (GC Fuji I) and resin-modified GIC (GC FujiCEM Evolve) exhibited significantly lower SBS values with the titanium surface, whereas the use of self-adhesive resin cements such as G-CEM ONE and G-CEM LinkForce, following the application of 10-MDP and 10-MDTP primer, provided effective bonding to the titanium surface.

2. While cohesive failures occur in conventional and resin-modified GICs with low SBS values, mostly adhesive and combined failures are predominantly observed in groups with high bond strengths.

3. Both sandblasting and tribochemical silica coating methods, which are applied for the surface treatment of monolithic zirconia, gave satisfactory SBS values in the adhesive resin cement groups. As a result of this, G-CEM ONE and G-CEM LinkForce cements can be clinically preferred for cementation of titanium and monolithic zirconia surfaces.

4. Long-term clinical studies are required to prove the validity of the obtained findings, which is in line with the limitations of any in vitro study.

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