©Operative Dentistry, 2014, **39-3,** E118-E127

Effects of Different Surface Treatments on Bond Strength Between Resin Cements and Zirconia Ceramics

A Erdem • GC Akar • A Erdem
T Kose

Clinical Relevance

The use of different surface treatments (airborne particle abradion or tribochemical silica coating/silane coupling system) on zirconia ceramic for different adhesive resin cements (dual or self-cure) may improve bond strength between resin cements and zirconia ceramic.

SUMMARY

This study compares the bond strength of resin cement and yttrium-stabilized tetragonal zirconia polycrystalline (Y-TZP) ceramic with different surface conditioning methods. Two hundred presintered Y-TZP ceramic specimens were prepared, sintered (4 × 4 × 4 mm), and randomly assigned to four equal groups as control (C, no conditioning); airborne particle abraded (APA, air abrasion with 11 μm Al_2O_3); tribochemical silica coating/silane coupling system (TSC, Rocatec, air abrasion with 110 μm Al_2O_3, 30 μm silica-coated Al_2O_3 and silane); and laser (L, Er:YAG laser irradiation treated at a power setting of 200 mJ). After specimen preparation, composite resin cylinders were prepared and cemented with resin cements (Clearfil Esthetic, Panavia F 2.0, Rely X-U100, Super Bond C&B, and Multilink Automix) on the ceramic surfaces and kept in an incubator at 37°C for 60 days. All specimens were tested for shear bond strength with a universal testing machine, and fractured surfaces were evaluated by environmental scanning electron microscopy. Statistical analysis was performed using Kruskal-Wallis and Mann-Whitney U-tests (α=0.05). The bond strengths for C and L groups were not significantly different according to adhesive resin cement. APA and TSC resulted in increased bond strength for Panavia F 2.0 and Rely X-U100 resin cements. Additionally, TSC presented higher bond

Ali Erdem, DDS, PhD, Department of Prosthodontics, School of Dentistry, University of Sifa, Bayrakli, Izmir, Turkey

*Gülcan Coşkun Akar, DDS, PhD, associate professor, Ege University, Prosthodontics, Ataturk Medical Technology Vocational Training School, University of Ege, Bornova, Izmir, Turkey

Adalet Erdem, DDS, PhD, professor, Department of Prosthodontics, School of Dentistry, University of Ege, Bornova, Izmir, Turkey

Timur Kose, PhD, Department of Biostatistics and Medical Informatics, School of Medicine, University of Ege, Bornova, Izmir, Turkey

*Corresponding author: Izmir, 35100 Turkey; e-mail: gulcan1976@yahoo.com

DOI: 10.2341/12-420-L

strength with Multilink Automix. Adhesive fracture between the ceramic and resin cement was the most common failure. Complete cohesive fracture at the ceramic or composite cylinders was not observed. Regardless of the adhesive resin cement used, laser treatment did not improve resin bond strength.

INTRODUCTION

Zirconia-based restorative materials have been used as core materials for single crowns and fixed dental prostheses due to their superior esthetics, biocompatibility, and high mechanical strength.[1] Early recommendations for luting zirconia restorations included the use of conventional cements, such as zinc phosphate or resin-modified glass ionomer cements, although adhesive cementation provides high retention, improves marginal adaptation, prevents microleakage, provides fracture resistance of the restored tooth and the restoration, and improves longevity of ceramic restorations.[1,2] In addition, resin cements offer the advantage of sealing minor internal surface flaws created by acid etching or airborne particle abrasion, significantly strengthening ceramic materials.[3] To achieve exceptional bonding durability between luting agents and a sintered zirconia surface, the surface area of the bonding surface must be increased and an active surface produced.[4-6] Airborne particle abrasion (APA), tribochemical silica coating (TSC), acid etching, plasma spraying, low-fusing porcelain layers, application of a glaze layer, silane application, heat-induced maturation-selective etching techniques, fusion sputtering, and lasers have been evaluated on yttrium partially stabilized tetragonal zirconia polycrystalline (Y-TZP) surface-conditioning methods.[7-14]

Unlike glass ceramics, etching is not possible for Y-TZP due to the high crystalline content and glass-free structure.[5,15-17] The zirconia surface has been shown to be minimally affected by conventional roughening techniques, although the strength may be enhanced.[18-21] APA is a suitable method to increase surface area to produce micromechanical interlocks of the bonding interface.[4,22] Tribochemical silicoating by Rocatec™ has been introduced as an alternative to APA.[23] In this technique, the ceramic surface is first air abraded with Al_2O_3 particles to remove contaminants and provide microroughness. Then the surfaces are airborne-particle abraded with aluminum trioxide particles modified with silica. The blasting pressure results in the embedding of the silica-coated alumina particles on the ceramic surface, rendering the silica-modified surface chemically reactive to the resin through silane coupling agents.[24] Bonding between a ceramic surface and resin luting agent may be facilitated by a silane coupling agent. The alkoxy groups (RO_3Si-) of the silane molecule react with water to form silanol groups (SiOH). The silanol groups further react with hydroxyl (OH) groups on a ceramic surface with available Si and O to form siloxane (-Si-O-Si-O-) covalent bonds. The monomeric end of the silane molecules reacts with the methacrylate groups of the resin luting agent. Thus, a strong network of siloxane covalent bonds is formed between the ceramic surface and methacrylate resin.[16] This chemical reaction is not applicable to zirconia-based ceramics because it lacks a silica phase.[4] A high initial tensile bond strength of resin to zirconia-based ceramics was achieved with silicoating followed by silanization.[4,7,20,24-26] Different laser systems (Er:YAG, CO_2, and Nd:YAG) have been used to alter and improve bond strength of resin cements to a zirconia surface.[12,13,27-29] Most reports, however, have demonstrated a significant reduction in bond strength after simulated artificial aging.[4,7,8,24]

The aim of this study was to compare bond strengths after three different surface treatments to zirconia and the use of five different adhesive resin cements. The null hypotheses of this study were that different types of surface treatment would not change the bond strength between the zirconia and adhesive resin cement and that the different types of adhesive resin cement would not affect the bond strength of zirconia.

MATERIALS AND METHODS

Preparation of Core Ceramics

Two hundred ($5 \times 5 \times 5$ mm) cubes were milled from presintered 3% Y-TZP (Zircon Ice Zirconia®, Zirkon-Zahn, Bruneck, Italy [ZrO_3; specifications, Y_2O_3 % 4-6, Al_2O_3 % <1, SiO_2 % max. 0.02, Fe_2O_3 % max. 0.01, Na_2O % max. 0.04]) using an electrical high-precision saw (IsoMet 1000 Precision Saw, Buehler Ltd, Lake Bluff, IL, USA) under water irrigation with a diamond wafering blade (6-inch Diamond Waferin Blade, Series 15 LC Diamond No 11-4776, Buehler). The cubes were sintered at 1500°C for 7 hours in a high-temperature sintering furnace for zirconia (Keramikofen 1500, ZirkonZahn). The dimensions of the cubes were $4 \times 4 \times 4$ mm following 20% volumetric shrinkage[30] associated with the sintering.

All specimens were ultrasonically cleaned (Professional Ultrasonic Cleaner CD-4800, Codyson, ShenZ-

hen, China) successively in 75% ethanol, 25% deionized water, 50% ethanol, 50% deionized water, 25% ethanol, and 75% deionized water for 15 minutes to remove factors that inhibit adhesion. The specimens were dried in the natural atmosphere and placed in a sterile cell culture dish (Costar 96 Well Cell Culture Cluster, Corning Inc, Corning, NY, USA).

Ceramic Surface Treatments

The cubes were assigned to one of the following four groups (n=50):

1) Control group (C): Underwent no mechanical surface treatment following sintering.
2) APA: The cubes were abraded using 110-μm alumina (Al_2O_3) particles (Sheraaluminiumoxid 110 MY, Shera Werkstoff Technologie, Hanover, Germany) at 2.8-bar pressure for 13 seconds from a distance of 10 mm perpendicular to the treatment surface of the cubes.
3) TSC: The bonding surface of each specimen was air abraded with 110-μm Al_2O_3 particles (Rocatec Pre, 3M ESPE, St Paul, MN, USA) at 2.8-bar pressure from a distance of 10 mm perpendicular to the treatment surface of the cubes for 10 seconds. Then 30-μm silicatized Al_2O_3 particles (Rocatec Plus) were applied at 2.8-bar pressure for 10 seconds at a distance of 10 mm in accordance with the manufacturer's recommendation. Silane coupling agent was applied to the silica-modified surface with a disposable brush, which was then dried with air.
4) Laser (L): Because ceramic surfaces reflect the laser beam, surfaces of the specimens were coated with graphite (pencil) to increase the laser energy absorption. The surfaces were then treated with erbium:yttrium-aluminum-garnet (ER:YAG) laser (AT Fidelis Plus III, Fotona, Ljubljana, Slovenia) (2 W, 10-Hz frequency, and 200-mJ energy, 2940 nm) from a distance of 10 mm for 10 seconds.

Environmental Scanning Electron Microscopy

After surface treatments, zirconia surfaces were examined at 500×, 1000×, 2000×, and 5000× magnification under an environmental scanning electron microscope (ESEM) (Quanta Feg 250, FEI, Oregon, USA).

Preparation of Composites Discs

Composite resin (Filtek™ P60, 3M ESPE) was prepared by injecting the resin composite into a glass pipette (Ø 3 mm) and was light polymerized using an LED polymerization light (Bluephase, Ivoclar Vivadent, Schaan, Liechtenstein) at 1350 mW/cm^2 for 40 seconds from two different directions for a total of 80 seconds

Two hundred (Ø 3 × 3 mm) composite cylinders were prepared and ultrasonically cleaned (Professional Ultrasonic Cleaner CD-4800, Codyson) by the same method and stored in a closed box to prevent contamination.

Cementation

Five different adhesive resin cement systems—Panavia F 2.0 (Kuraray Co. Ltd, Tokyo, Japan), RelyX U 100 (3M ESPE), Clearfil Esthetic (Kuraray Medical Co, Osaka, Japan), Super Bond C&B (Sun Medical, Shiga, Japan), and Multilink Automix (Ivoclar-Vivadent)—were used to lute the composite resin discs to the zirconia discs according to the manufacturers' instructions. Properties of all used materials are listed in Table 1. Each cement was mixed and applied on the surface of the composite resin disc and seated on the zirconia disc. The polymerization was completed under 700 g of constant load for 60 seconds using a custom-made jig modified from a parallelometer. Simultaneously, the excess cement was wiped off, and the specimen was light polymerized (except Superbond C&B) with the same unit at four different locations for 60 seconds each. Oxygen-blocking gel (Oxyguard II, Kuraray) was applied for 3 minutes, then washed with air/water spray for Panavia 2.0 cement. The polymerization unit was tested for power output after every 10 specimens.

Wet Storage

All bonded specimens were placed inside the compartments of special cell boxes in an incubator (Incubator IF110 Plus, Memmert, ,Büchenbach, Germany) at 100% humidity and 37°C (±2°C) for 60 days before being stressed with the notched shear bond test method.

Shear Bond Strength Test

The specimens were mounted in the jig for shear bond strength testing described in ISO/TR 11405 of a computer-controlled universal testing machine (Autograph AG-50kNG, Shimadzu, Kyoto, Japan) and were loaded in shear at a constant crosshead speed of 0.5 mm/min until failure. The maximum load to failure was recorded by the corresponding software in newtons (N), and the shear bond strength was calculated in MPa by dividing the failure load (N) by

Table 1: *List of Brand Names, Curing Types, Main Compositions, Batch Numbers, and Manufacturers of Resin Cements Investigated*

Material	Type	Characteristics	Batch No.	Zirconia Primer	Batch No.	Manufacturer
Multilink Automix	Dual curing/ self-etch	Liquid: DMA, HEMA, Ba-glass fillers, ytterbium fluoride, spheroid mixed oxide A primer: aqueous solution of initiator B primer: HEMA and phosphoric acid and acrylic acid monomers Metal/zirconia primer: phosphoric acid acrylate and methacrylate cross-linking agents in an organic solution	L12252	Monobond S + Heliobond	K41829 K30706	Ivoclar Vivadent
Panavia F 2.0	Dual curing	10-methacryloxydecyldihydrogen-phosphate (MDP) Paste A: BPEDMA, MDP, DMA Paste B: Al-Ba-B-Si glass/silica-containing composite	00303B 00052A	Porcelain Bond Activator + SE Bond Primer	00004C 00799A	Kuraray Medical Co.
Clearfil Esthetic	Dual curing	Paste A: Bis-GMA, TEG-DMA, methacrylate monomers, silanated glass filler, colloidal silica Paste B: Bis-GMA, TEGDMA, methacrylate monomers, glass filler, colloidal silica, benzoyl peroxide, pigments	0005BC	Ceramic Primer	00004E	Kuraray Medical Co.
Rely X U 100	Dual curing/ self-etch	Glass powder, dimethacrylate, silanated silica, Sodium p-toluene sulfinate, calcium hydroxide	327580	ESPE Sil 3-		
		methacryloxypropyltrimethoxysilane (MPS) in ethanol	192348	3M ESPE		
Super Bond C&B	Self-curing	Powder: PMMA, Liquid: MMA, 4-META Catalyst: TBB + Liquid: MMA, 4-META	ME12F MS1 (monomer)	Porcelain Liner M	TE1	Sun Medical

Abbreviations: DMA, dimethacrylate; HEMA, 2-hydroxyethylmethacrylate; BPEDMA, bisphenol-A polyethoxydimethacrylate; DMA, aliphatic dimethacrlyate; TEGDMA, triethylene glycol dimethacrylate; BisGMA, bisphenylglycidyl dimethacrylate; 4-META, 4-methacryloxyethyl trimellitate anhydride; MMA, methyl methacrylate; PMMA, poly(methyl methacrylate); TBB, tri-n-butyl borane catalyst.

the bonding area (mm^2). The same operator prepared and tested all specimens to eliminate interoperator variability.

Failure Mode

The mode of failure of each specimen was determined by inspecting the bonding surfaces of each specimen under optical magnification (10× handheld loupe, Carl Zeiss AG, Oberkochen, Germany). Failure mode was classified into three types: (1) adhesive failure (failure between the resin luting agent and the resin composite or between the resin luting agent and the ceramic), (2) cohesive failure within the resin luting agent, and (3) mixed mode of failure, including the first and second types ESEM images of representative specimens were captured.

Statistical Analysis

Statistical analyses were performed by using SPSS 18.0 System for Windows (SPSS, Chicago, IL, USA). As a result of the Shapiro-Wilk normality test for the nonparametric methods applied to data that do not meet the normal distribution, p-values less than 0.05 were considered to be statistically significant in all tests. Kruskal-Wallis followed by a Mann-Whitney U-tests were used to comparatively assess the data.

RESULTS

The data of shear bond strength in all subgroups are listed in Table 2. For Panavia F 2.0, Clearfil Esthetic, and Rely X U100 adhesive resin cements, shear bond strength values of control and laser groups were statistically different from TSC ($p<0.000$) and APA ($p<0.000$; C: $p<0.019$; L: $p<0.002$ for Clearfil Esthetic resin cement) groups. In addition, there was a significant difference between the two resin cement groups (Clearfil Esthetic and Rely X U100) and the two surface treatment methods (APA and TSC) ($p<0.000$). For adhesive resin cement Multilink Automix, TSC and

Table 2: *The Median and Min-Max MPD Values (MPa) of Surface Finishing Groups and Adhesive Cements (n=10)*[a]

Cements Surface Treatment	Panavia F 2.0		Clearfil Esthetic		RelyX U100		Multilink Automix		Super Bond C&B	
	Median	Min–Max	Median	Min–Max	Median	Min–Max	Median	Min–Max	Median	Min–Max
Control (C)	0.2^{A}	0.0–0.3	0.0^{A}	0.0–0.2	0.3^{A}	0.0–0.4	0.0^{A}	0.0–0.5	0.2^{A}	0.0–3.0
Airborne-particle-abraded (APA)	$12.2^{B,1}$	9.9–14.8	$1.8^{B,2}$	0.0–5.7	$12.7^{B,1}$	10.9–20.1	$0.3^{A,2}$	0.0–0.8	$0.4^{A,2}$	0.3–1.1
Tribochemical silica coating/silane coupling system (TSC)	$13.3^{B,1}$	7.1–19.8	$8.8^{C,2}$	5.1–13.0	$10.5^{C,2}$	6.8–13.2	$10.1^{B,1,2}$	7.5–13.0	$0.9^{A,3}$	0.2–1.5
Laser (L)	0.4^{A}	0.0–5.0	0.1^{A}	0.0–0.4	0.1^{A}	0.0–2.6	0.0^{A}	0.0–0.4	0.4^{A}	0.0–1.6

[a] *Different uppercase letters represent statistically significant differences within each row. Different numbers represent statistically significant differences within each column.*

other surface treatment groups were statistically different (C: $p<0.000$; APA: $p<0.000$; L: $p<0.000$). There was no difference in shear bond strength between Superbond CB resin cement and surface treatment methods ($p>0.05$). The bond strength for control and laser groups were not significantly different according to adhesive resin cement ($p>0.05$).

The ESEM images (1000× and 5000×) showed morphological differences among Y-TZP specimens after surface treatment. Zirconia specimens with no treatment showed microstructures of grained Y-TZP ceramics (Figure 1A,B). The surface roughness of APA specimens was remarkably increased compared with the control surface (Figure 1C,D). ESEM images of TCS showed the smooth surface of zirconia ceramic caused by silanization (Figure 1E,F). Er:-YAG laser irradiation created a smooth surface, with cracks and loss of material, although the surface was not altered (Figure 1G,H).

Failure modes of the groups are presented in Figure 2 All shear bond strength specimens demonstrated adhesive failures at the resin/zirconia interface. Adhesive failure was mostly observed with an average of 83.5% between the ceramic and resin-luting agent. Fourteen percent of the specimens were associated with mixed failures (Figure 3). Resin cement residue was noted on the ceramic surface. Fractures always occurred at the ceramic/cement interface. Cohesive failure of the ceramic and composite was not observed.

DISCUSSION

Mechanical and chemical modifications of the prepared surface of fixed restorations are well-documented methods of establishing a reliable bond between the restoration and the adhesive cement. Adhesive resin cements are used not only to prevent the dislodgement of ceramic restorations but also to obtain a strong and long-lasting cementation between tooth and ceramic restoration.[18] Thus, it is very important to select the best combination of resin cement and surface treatment for durability. However, hydrofluoric acid etching is not sufficient to improve the bond strength between zirconia ceramics and resin cements, as zirconia lacks silica and is resistant to acid etching. With these considerations in mind, hydrofluoric acid etching was excluded from this study.

To promote micromechanical retention in zirconia ceramics, the APA method can be used instead of acid etching.[31] APA might create subcritical microcracks and phase transformation within the zirconia surface and thereby deteriorate the mechanical properties of zirconia.[32] Quaas and others[33] found that contaminating zirconia surfaces by air abrasion led to higher resin-ceramic bond strength than cleaning the contaminated ceramic surfaces with phosphoric acid or alcohol. The results of a study by Blatz and others[34] also confirm that air-particle abrasion of zirconia surfaces with Al_2O_3 increases bond strength values of self-adhesive luting cements to ceramic surfaces. Derand and Derand[35] reported that acid etching and airborne-particle abrasion had only minor influence on bond strengths. In the present study, the outcome of APA treatment was found to be comparable to that of the TSC treatment, as bond strength values were higher than the laser and control groups. Lüthy and others[36] found that the bond strength of the phosphate monomer (MDP)-containing composite resin P21 to the sandblasted zirconia surface was more than the bond strength of other cements tested (Ketac-Cem, Nexus, Rely X Unicem, Superbond C&B, and Panavia F). This might depend on the fact that the phosphate ester group of the MDP can directly bond to metal oxides.[37] Similarly, APA treatment results were similar with TSC in MDP-containing adhesive resin cements, and high bond strength values were obtained in the present study.

Theoretically, air-particle abrasion could improve bond strength by enhancing surface microroughness

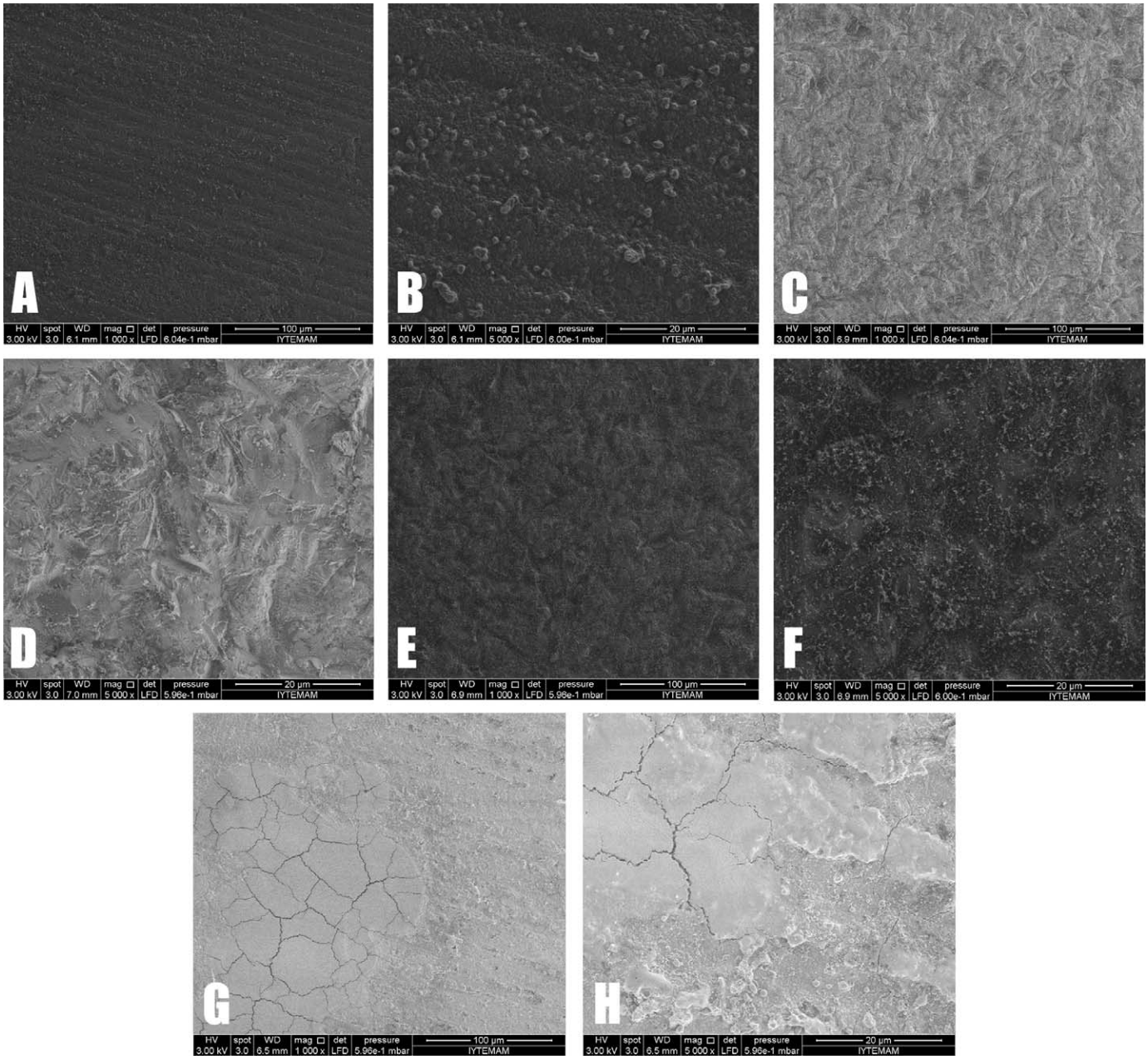

Figure 1. *Environmental scanning electron micrographs (1000× and 5000× original magnification) of zirconia ceramic specimen surfaces. (A and B): Control. (C and D): Airborne particle abraded. (E and F): Tribochemical silica coated. (G and H): Laser applied.*

and generating more hydroxyl groups on the ceramic surface to react with the silanol groups of the silane. The silane molecules alternatively chemically bond to the methacrylate groups of the luting resin.[16] It has also been suggested that the silane agent promotes resin bonding by increasing surface energy and thus wettability of the bonding substrate.[17] Xible and others[20] reported a strengthening effect of tribochemical airborne-particle abrasion using a larger particle size (Rocatec, 110 μm; 3M ESPE) at 2.8-bar pressure. Kern and Wegner[4] reported good initial bond strength of a dual-polymerizing bis-GMA resin cement to APA zirconia. Adding a silane, however, did not improve the bond strength. Blatz and others[8] reported that a coupling agent containing an adhesive phosphate monomer can achieve superior long-term shear bond strength to APA zirconia restorations when using a resin cement. The silicoated/silanated group achieved the highest bond strength among the groups.[15,38] This outcome

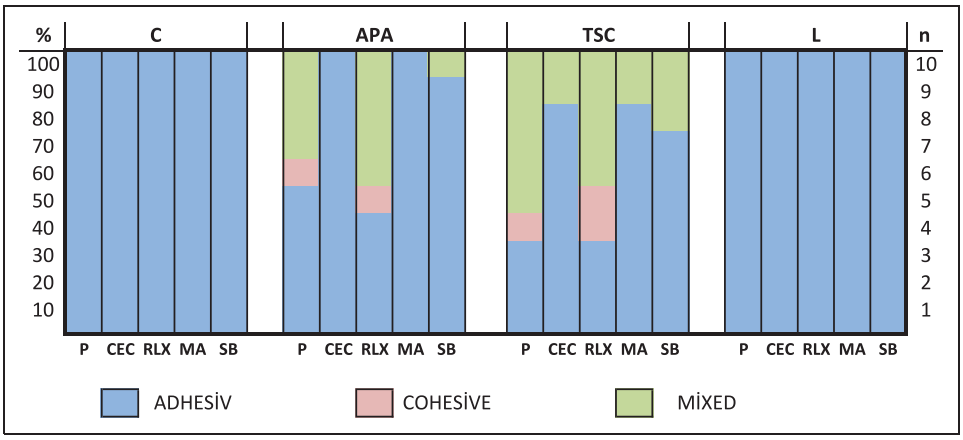

Figure 2. *Distribution of failure modes of experimental groups (C: Control; APA: Airborne particle abraded; TSC: Tribochemical silica coating/silane coupling system, L: Laser groups and P: Panavia F 2.0; CEC: Clearfil Esthetic; RLX: Rely X-U100; MA: Multilink Automix; SB: Super Bond C&B cement).*

is in line with the findings of Kim and others,[25] who demonstrated a high tensile bond strength of resin to zirconia after silicoating and silanization. In the present study, high bond strength values were obtained with the TSC method, and this finding is also in line with previous reports.

Akyil and others[13] reported that air abrasion with 110 μm Al_2O_3 and silica coating with CoJet-Sand were the most effective surface treatment methods with Clearfil Esthetic resin cement. The shear bond strength between a zirconium oxide ceramic and composite resin using an MDP-containing resin luting agent (Panavia F) was increased with chair-side TSC.[17] Passos and others[39] reported that groups conditioned using silica coating and silanization showed higher bond strengths in both dry and aged conditions. Two microtensile bond studies[40,41] on zirconium oxide ceramics using composite resin block as substrate showed that tribochemical silica coating with 50-μm Al_2O_3 particles modified by silica was ineffective in improving the bond strength of three luting agents, including an MDP-containing resin luting agent (Clearfil Esthetic Cement, Kuraray). In the present study, TSC treatment increased bond strength values of MDP-free resin adhesive cements more than MDP-containing resin.

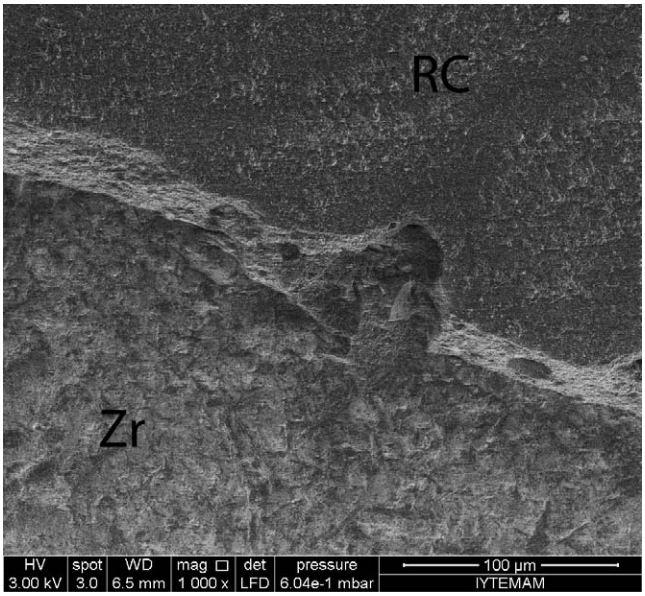

Figure 3. *SEM photograph (1000× original magnification) of APA treated/Rely X U100 adhesive cement failed surface (RC, resin cement; Zr, zirconia).*

In this study, a lower power setting of 200 mJ was selected for the Er:YAG laser with meticulous water cooling to prevent internal tensions related to heating/cooling-dependent local temperature changes. Bond strength results indicated that adhesion was not improved by laser treatment. Er:YAG laser–treated and –untreated surfaces presented similar results. These results agree with those of Cavalcanti and others,[27] who evaluated the influence of surface treatments and metal primers on the bond strength of resin cements to a Y-TZP ceramic with microshear bond test. In addition, our findings are in line with those of Foxton and others,[12] who aimed to evaluate the durability of the bond of conventional dual-cured resin cements to Procera Al_2O_3 and zirconium oxide ceramics after surface treatment with air abrasion and erbium laser. Akyıl and others[13] found that the Er:YAG laser irradiation (200 mJ/pulse, 10 Hz, 10 seconds) increased the bond strength compared to that of untreated surface and created a rough surface similar to that of air abrasion with SEM evaluations. In this study, ESEM evaluations showed that the irradiated surface exhibited a

bubbled blister-like appearance and unusual microcracks. This appearance may have been due to the development of a heat-damaged layer caused by the application of Er:YAG laser on the Y-TZP surface. This heat-damaged layer, containing bubbles, may be poorly attached to the infralayer of the substrate and may account for specimen rupture when a low force is applied during the shear bond test.

Long-term water storage and thermal cycling are commonly used to simulate aging of resin bond interfaces.[8] In the literature, there is no consensus on a relevant regimen for artificial aging.[42] Kern and Wegner[4] evaluated resin bond strength to zirconia after water storage for 150 days and 2 years, combined with thermal cycling. They indicated that artificial aging with thermal cycling decreased the tensile bond strength of resin to silicoated (Rocatec, 3M ESPE) zirconia by almost one-third of the initial bond. Strong degradation at the Y-TZP ceramic/resin cement interface has been noted when the specimens are submitted to aging with stored in distilled water at 37°C for 7 days.[43] Ozcan and others[44] found no adhesion (0 MPa, debonding during aging) between Y-TZP ceramic air abraded with Al_2O_3 and different resin cements (Panavia F2.0, Multilink, Superbond C&B, and Quadrant Posterior Dense) after thermocycling (6000×, 5-55°C). Ozcan and others,[45] after subjecting the specimens to water storage at 37°C for 3 months in the dark, compared bond strength of two resin cements to Y-TZP ceramic using 3-methacryloxypropyltrimethoxysilane (MPS) or MPS/4-methacryloyloxyethy trimellitate anhydride (4-META) silanes. They did not conduct thermocycling, although the effect of thermocycling is controversial.[4,6] Blatz and others[8] noted a decrease in the tensile bond strength values between Y-TZP and two resin cements (adhesive application + RelyX ARC and adhesive application + Panavia F) after 24 hours of storage in water. Passos and others[39] compared dry conditions and 90 days of water storage at 37°C and thermocycling (12,000×, 5-55°C) bonding to Y-TZP with resin cements and showed that bond strength was reduced dramatically after aging. Oyagüe and others[40] evaluated the hydrolytic stability (6 months of water storage at 37°C) of different dual-cure resin cements when luted to zirconia ceramic. They found that the bond strength of Clearfil Esthetic Cement significantly decreased after water storage. The studies showed similar results, as thermocycling and wet storage decreased the bond strength between zirconia and resin cements regardless of the aging process. In this study, wet storage was done by holding the incubator at 37°C for 60 days, and some of the cement bond strength values obtained were zero in the laser and control groups.

CONCLUSIONS

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

1. Bond strengths of resin cements in untreated control and laser-applied specimens were low. APA and TSC methods can improve the bond to the resin cement.
2. Phosphate monomer–containing adhesive resin cements (Panavia 2.0, Clearfil Esthetic, and Rely X U100) in combination with air-particle abrasion or tribochemical silane application of ceramic surface produced a higher bond strength.
3. The tribochemical silica coating/silane coupling system increased bond strength values of phosphate monomer–free adhesive resin cement (Multilink Automix).
4. Regardless of the dual and self-cure resin cement used, laser treatment did not improve resin bond strength.

Conflict of Interest

The authors of this article certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

(Accepted 3 August 2013)

REFERENCES

1. Koutayas SO, Vagkopoulou T, Pelekanos S, Koidis P, & Strub JR (2009) Zirconia in dentistry: Part 2. Evidence-based clinical breakthrough *European Journal of Esthetic Dentistry* **4(4)** 348-380.
2. Albert FE, & El-Mowafy OM (2004) Marginal adaptation and microleakage of Procera AllCeram crowns with four cements *International Journal of Prosthodontics* **17(5)** 529-535.
3. Blatz MB (2002) Long-term clinical success of all-ceramic posterior restorations *Quintessence International* **33(6)** 415-426.
4. Kern M, & Wegner SM (1998) Bonding to zirconia ceramic: adhesion methods and their durability *Dental Materials* **14(1)** 64-71.
5. Wolfart M, Lehmann F, Wolfart S, & Kern M (2007) Durability of the resin bond strength to zirconia ceramic after using different surface conditioning methods *Dental Materials* **23(1)** 45-50.
6. Wegner SM, & Kern M (2000) Long-term resin bond strength to zirconia ceramic *Journal of Adhesive Dentistry* **2(2)** 139-147.

7. Blatz MB, Chiche G, Holst S, & Sadan A (2007) Influence of surface treatment and simulated aging on bond strengths of luting agents to zirconia *Quintessence International* **38(9)** 745-753.
8. Blatz MB, Sadan A, Martin J, & Lang B (2004) In vitro evaluation of shear bond strengths of resin to densely-sintered high-purity zirconium-oxide ceramic after long-term storage and thermal cycling *Journal of Prosthetic Dentistry* **91(4)** 356-362.
9. Conrad HJ, Seong WJ, & Pesun IJ (2007) Current ceramic materials and systems with clinical recommendations: A systematic review *Journal of Prosthetic Dentistry* **98(5)** 389-404.
10. Yang B, Barloi A, & Kern M (2010) Influence of air-abrasion on zirconia ceramic bonding using an adhesive composite resin *Dental Materials* **26(1)** 44-50.
11. Kitayama S, Nikaido T, Maruoka R, Zhu L, Ikeda M, Watanabe A, Foxton RM, Miura H, & Tagami J (2009) Effect of an internal coating technique on tensile bond strengths of resin cements to zirconia ceramics *Dental Materials Journal* **28(4)** 446-453.
12. Foxton R, Cavalcanti AV, Nakajima M, Pilecki P, Sherriff M, Melo L, & Watson TF (2011) Durability of resin cement bond to alumina oxide and zirconia ceramics after air abrasion and laser treatment *Journal of Prosthodontics* 2011 **20(2)** 84-92.
13. Akyıl MS, Uzun IH, and Bayindir F (2010) Bond strength of resin cement to yttrium-stabilized tetragonal zirconia ceramic treated with air abrasion, silica coating, and laser irradiation *Photomedicine and Laser Surgery* **28(6)** 801-808.
14. Aboushelib MN (2012) Fusion sputtering for bonding to zirconia-based materials *Journal of Adhesive Dentistry* **14(4)** 323-328.
15. Qeblawi DM, Muñoz CA, Brewer JD, & Monaco EA Jr (2010) The effect of zirconia surface treatment on flexural strength and shear bond strength to a resin cement *Journal of Prosthetic Dentistry* **103(4)** 210-220.
16. Amaral R, Ozcan M, Bottino MA, &Valandro LF, (2006) Microtensile bond strength of a resin cement to glass infiltrated zirconia-reinforced ceramic: The effect of surface conditioning *Dental Materials* **22(3)** 283-290.
17. Atsu SS, Kilicarslan MA, Kucukesmen HC, & Aka PS (2006) Effect of zirconium-oxide ceramic surface treatments on the bond strength to adhesive resin *Journal of Prosthetic Dentistry* **95(6)** 430-436.
18. Borges GA, Sophr AM, de Goes MF, Sobrinho LC, & Chan DC (2003) Effect of etching and airborne particle abrasion on the microstructure of different dental ceramics *Journal of Prosthetic Dentistry* **89(5)** 479-488.
19. Kosmac T, Oblak C, Jevnikar P, Funduk N, & Marion L (2000) Strength and reliability of surface treated Y-TZP dental ceramics *Journal of Biomedical Materials Research* **53(4)** 304-313.
20. Xible AA, de Jesus Tavarez RR, de Araujo Cdos R, & Bonachela WC (2006) Effect of silica coating and silanization on flexural and composite-resin bond strengths of zirconia posts: An in vitro study *Journal of Prosthetic Dentistry* **95(3)** 224-229.
21. Kosmac T, Oblak C, Jevnikar P, Funduk N, & Marion L (1999) The effect of surface grinding and sandblasting on flexural strength and reliability of Y-TZP zirconia ceramic *Dental Materials* **15(6)** 426-433.
22. Kumbuloglu O, Lassila LV, User A, & Vallittu PK (2006) Bonding of resin composite luting cements to zirconium oxide by two air-particle abrasion methods *Operative Dentistry* **31(2)** 248-255.
23. Kern M, & Thompson VP (1994) Sandblasting and silica coating of a glass infiltrated alumina ceramic: Volume loss, morphology, and changes in the surface composition *Journal of Prosthetic Dentistry* **71(5)** 453-461.
24. Ozcan M, & Vallittu PK (2003) Effect of surface conditioning methods on the bond strength of luting cement to ceramics *Dental Materials* **19(8)** 725-731.
25. Kim BK, Bae HE, Shim JS, & Lee KW (2005) The influence of ceramic surface treatments on the tensile bond strength of composite resin to all-ceramic coping materials *Journal of Prosthetic Dentistry* **94(4)** 357-362.
26. Blatz MB, Sadan, A & Blatz U (2003) The effect of silica coating on the resin bond to the intaglio surface of Procera AllCeram restorations *Quintessence International* **34(7)** 542-547.
27. Cavalcanti AV, Foxton RM, Watson TF, Oliveria MT, Giannini M, & Marchi GM (2009) Bond strength of resin cements to a zirconia ceramic with different surface treatments *Operative Dentistry* **34(3)** 280-287.
28. Ural C, Külünk T, Külünk S, & Kurt M (2010) The effect of laser treatment on bonding between zirconia ceramic surface and resin cement *Acta Odontologica Scandinavica* **68(6)** 354-359.
29. Spohr AM, Borges GA, Junior LH, Mota EG, & Oshima HM (2008) Surface modification of In-Ceram Zirconia ceramic by Nd:YAG laser, Rocatec system, or aluminum oxide sandblasting and its bond strength to a resin cement *Photomedicine and Laser Surgery* **26(3)** 203-208.
30. Heydecke G, Butz F, Binder JR, & Strub JR (2007) Material characteristics of a novel shrinkage-free $ZrSiO_4$ ceramic for the fabrication of posterior crowns *Dental Materials* **23(7)** 785-791.
31. Tsukakoshi M, Shinya A, Gomi H, Lassila LV, Vallittu PK, & Shinya A (2008) Effects of dental adhesive cement and surface treatment on bond strength and leakage of zirconium oxide ceramics *Dental Materials Journal* **27(2)** 159-171.
32. Zhang Y, Lawn BR, Rekow ED, & Thompson VP (2004) Effect of sandblasting on the long-term performance of dental ceramics *Journal of Biomedical Materials Research Part B Applied Biomaterials* **71(2)** 381-386.
33. Quaas AC, Yang B, & Kern M (2007) Panavia F 2.0 bonding to contaminated zirconia ceramic after different cleaning procedures *Dental Materials* **23(4)** 506-512.
34. Blatz MB, Phark JH, Ozer F, Mante FK, Saleh N, Bergler M, & Sadan A (2010) In vitro comparative bond strength of contemporary self-adhesive resin cements to zirconium

oxide ceramic with and without air-particle abrasion *Clinical Oral Investigations* **14(2)** 187-192.

35. Derand P, & Derand T (2000) Bond strength of luting cements to zirconium oxide ceramics *International Journal of Prosthodontics* **13(2)** 131-135.
36. Lüthy H, Loeffel O, & Hammerle CH (2006) Effect of thermocycling on bond strength of luting cements to zirconia ceramic *Dental Materials* **22(2)**195-200.
37. Wada T (1996) *Development of a new adhesive material and its properties* Proceedings of the International Symposium on Adhesive Prosthodontics Academy of Dental Materials, Amsterdam 9-18.
38. Valandro LF, Özcan M, Amaral R, Vanderlei A, & Bottino M (2008) Effect of testing methods on the bond strength of resin to zirconia-alumina ceramic: Microtensile versus shear test *Dental Materials Journal* **27(6)** 849-855
39. Passos SP, May LG, Barca DC, Ozcan M, Bottino MA, & Valandro LF (2010) Adhesive quality of self-adhesive and conventional adhesive resin cement to Y-TZP ceramic before and after aging conditions *Operative Dentistry* **35(6)** 689-696.
40. de Oyagüe RC, Monticelli F, Toledano M, Osorio E, Ferrari M, & Osorio R (2009) Influence of surface treatments and resin cement selection on bonding to densely-sintered zirconium-oxide ceramic *Dental Materials* **25(2)** 172-179.
41. Oyagüe RC, Monticelli F, Toledano M, Osorio E, Ferrari M, & Osorio R (2009) Effect of water aging on microtensile bond strength of dual-cured resin cements to pre-treated sintered zirconium-oxide ceramics *Dental Materials* **25(3)** 392-399.
42. Magne P, & Cascione D (2006) Influence of post-etching cleaning and connecting porcelain on the microtensile bond strength of composite resin to feldspathic porcelain *Journal of Prosthetic Dentistry* **96(5)** 354-361.
43. Valandro LF, Leite FP, Scotti R, Bottino MA, & Niesser MP (2004) Effect of ceramic surface treatment on the microtensile bond strength between a resin cement and an alumina-based ceramic *Journal of Adhesive Dentistry* **6(4)** 327-332.
44. Özcan M, Kerkdijk S, & Valandro LF (2008) Comparison of resin cement bond to Y-TZP ceramic following manufacturers' instructions of the cements only *Clinical Oral Investigations* **12(3)** 279-282.
45. Ozcan M, Cura C, & Valandro LF (2011) Early bond strength of two resin cements to Y-TZP ceramic using MPS or MPS/4-META silanes *Odontology* **99(1)** 62-64.