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Effects of air-abrasion pressure on the resin bond strength to zirconia: a combined cyclic loading and thermocycling aging study

Al-Shehri, Eman Z; Al-Zain, Afnan O; Sabrah, Alaa H; Al-Angari, Sarah S; Al Dehailan, Laila; Eckert, George J; Özcan, Mutlu; Platt, Jeffrey A; Bottino, Marco C

Abstract: **OBJECTIVES** To determine the combined effect of fatigue cyclic loading and thermocycling (CLTC) on the shear bond strength (SBS) of a resin cement to zirconia surfaces that were previously air-abraded with aluminum oxide (Al₂O₃) particles at different pressures. **MATERIALS AND METHODS** Seventy-two cuboid zirconia specimens were prepared and randomly assigned to 3 groups according to the air-abrasion pressures (1, 2, and 2.8 bar), and each group was further divided into 2 groups depending on aging parameters (n = 12). Panavia F 2.0 was placed on pre-conditioned zirconia surfaces, and SBS testing was performed either after 24 hours or 10,000 fatigue cycles (cyclic loading) and 5,000 thermocycles. Non-contact profilometry was used to measure surface roughness. Failure modes were evaluated under optical and scanning electron microscopy. The data were analyzed using 2-way analysis of variance and 2 tests ($\alpha = 0.05$). **RESULTS** The 2.8 bar group showed significantly higher surface roughness compared to the 1 bar group ($p < 0.05$). The interaction between pressure and time/cycling was not significant on SBS, and pressure did not have a significant effect either. SBS was significantly higher ($p = 0.006$) for 24 hours storage compared to CLTC. The 2 bar-CLTC group presented significantly higher percentage of pre-test failure during fatigue compared to the other groups. Mixed-failure mode was more frequent than adhesive failure. **CONCLUSIONS** CLTC significantly decreased the SBS values regardless of the air-abrasion pressure used.

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Effects of air-abrasion pressure on the resin bond strength to zirconia: a combined cyclic loading and thermocycling aging study

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Running title: Air-abrasion pressure effects resin adhesion to zirconia

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Abstract

Objectives: To determine the combined effect of fatigue cyclic loading and thermocycling on the shear bond strength of a resin cement to zirconia surfaces that were previously air-abraded with aluminum oxide (Al_2O_3) particles at different pressures. **Materials and Methods:** Seventy-two cuboid zirconia specimens were prepared and randomly assigned to three groups according to the air-abrasion pressures (1, 2, and 2.8 bar) and each group was further divided into two groups depending on aging parameters ($n = 12$). Panavia F 2.0 was placed on pre-conditioned zirconia surfaces and shear bond strength (SBS) testing was performed either after 24h or 10,000 fatigue cycles (cyclic loading) and 5,000 thermocycles. Non-contact profilometry was used to measure surface roughness. Failure modes were evaluated under optical and scanning electron microscopy. The data were analyzed using two-way analysis of variance and chi-square tests ($\alpha = 0.05$). **Results:** The 2.8 bar group showed significantly higher surface roughness compared to the 1 bar group ($p < 0.05$). The interaction between pressure and time/cycling was not significant on SBS and pressure did not have a significant effect either. SBS was significantly higher ($p = 0.0064$) for 24 h storage compared to cyclic loading and thermocycling. The 2 bar-cyclic loading and thermocycling group presented significantly higher percentage of pre-test failure during fatigue compared to the other groups. Mixed-failure mode was more frequent than adhesive failure. **Conclusions:** Cyclic loading and thermocycling significantly decreased the SBS values regardless of the air-abrasion pressure used.

Key words: Air-abrasion; Bond strength; Fatigue; Panavia F 2.0; Resin cement; Thermocycling.

Introduction

Yttrium oxide-stabilized tetragonal zirconia polycrystal (Y-TZP) is frequently used in dentistry due to its outstanding mechanical properties, biocompatibility, and aesthetic performance.¹⁻⁴ These superior properties made zirconium dioxide ceramics a popular high-strength ceramic with a large variety of clinical applications.¹⁻⁵ However, its chemical inertness challenges establishment of a strong, durable bond with other materials.⁵⁻⁸ The composition and physical properties of zirconia ceramics differ substantially from silica-based ceramics and require alternative bonding techniques to achieve strong and durable bonding of resin.^{7,8}

The clinical success of resin bonding procedures for ceramic restorations depends on the quality and durability of the bond between ceramic and resin cements. The quality of the bond depends on several factors, such as the bonding mechanisms that are controlled by the surface treatment, which promotes micromechanical and/or chemical bonding to ceramics.⁸ Mechanical retention of adhesives to zirconia ceramics can be achieved by air-abrasion or tribochemical silica coating before using chemical bonding agents as organosilanes or ceramic primers. The aforementioned chemical agents promotes better interaction with the ceramic surface by increasing the surface energy, and in turn, the wettability of the cement.⁹⁻¹⁴ Dentin adhesives containing an organophosphate ester monomer, such as 10-methacryloyloxydecyl dihydrogen phosphate (MDP), 4-methacryloxyethyl trimellitate anhydride (4-META), 6-methacryloyloxyhexyl phosphonoacetate (6-MHPA), or 6-methacryl-oxyhexyl 2-thiouracil-5-carboxylate (MTU-6), were shown to activate zirconia surfaces.¹⁴ Consequently, cements containing these monomers have led to higher bond strength when used with zirconia, and even higher bond strength when combined with air-abraded zirconia.¹⁴⁻¹⁷ However, there is no

consensus in the literature regarding the effective air-abrasion procedure to improve the resin cement adhesion to the zirconia ceramics.¹⁸⁻²¹

Thermocycling has been widely used to simulate thermal stresses commonly occurring in the oral environment based on differences in the coefficient of thermal expansion of materials.²² However, thermocycling alone does not precisely mimic oral conditions. Adding fatigue cyclic loading may provide a better assessment of the clinical performance of adhesive systems.²³ Recent meta-analysis studies reported on different protocols that involved subjecting test specimens to either thermal stresses or mechanical fatigue in an occlusal direction or perpendicular to the adhesive interface, but not a combination of these methods in a shear direction.^{15,18} One study evaluated the effect of fatigue cycling (i.e., 26 N at 2 Hz for 27,500 cycles) on the shear bond strength of a resin/porcelain system.²⁴ Considering the failure mode that occurs in the oral cavity, the proposed *in vitro* testing method may give a more clinically relevant evaluation of bond strength between zirconia and adhesive cements.

To the best of our knowledge, the effects of different air-abrasion pressures on surface roughness and adhesion performance to zirconia ceramic after combining thermal aging and mechanical fatigue cyclic loading in a shear direction have not been investigated. Therefore, the overall goal of this study was to determine the combined effect of fatigue cyclic loading and thermocycling on the shear bond strength of a resin cement to Y-TZP zirconia surfaces prepared at different air-abrasion pressures. The null hypotheses tested were: (1) increasing air-abrasion pressure using aluminum oxide particles would not affect Y-TZP zirconia ceramic surface roughness, (2) the combined effect of fatigue cyclic loading and thermocycling (CLTC) would not affect the shear bond strength (SBS) of resin cement to Y-TZP zirconia surfaces prepared at different air-abrasion pressures.

Materials and Methods

Y-TZP surface treatment

Seventy-two cuboid samples (10 × 10 × 2 mm) were sectioned before sintering from a disk-shaped block of Y-TZP zirconia (Ivoclar Vivadent Inc., Amherst, NY, USA) using Isomet 1000 (Buehler, Lake Bluff, IL, USA). Specimens were dried in an oven (Cerampress QEX porcelain and processing furnace, Dentsply Neytech, York, PA, USA) at 270°C for 1 h, sintered using the Lindberg Furnace (Blue M, Ashville, NC, USA) for 4½ hours, and cooled down overnight.² Ceramic specimens were embedded in acrylic resin using a plastic mold to aid specimen handling during the experiments (Figure 1A). Specimens were finished and polished using silicon carbide papers from 240- to 1200-grit under running water. The embedded Y-TZP specimens were randomly assigned to three air-abrasion pressure groups ($n = 24$): 1 bar (1b), 2 bar (2b), and 2.8 bar (2.8b). The 2.8b group served as the control.²⁵⁻²⁹ Each Y-TZP zirconia specimen was air-abraded using airborne-particle abrasion with 50 µm Al₂O₃ for 30 seconds (SandStorm Expert, Vaniman Manufacturing Co., Fallbrook, CA, USA) at a 10 mm distance. The surfaces were rinsed with distilled (DI) water for 20 seconds and air-dried for 5 seconds.¹⁴

Surface roughness measurement

Two representative specimens from each group were scanned prior to the resin cement button preparation via a non-contact 3 dimensional optical profilometer (Proscan 2000, Scantron Industrial Products Ltd., Taunton, UK). Using the S5/03 chromatic sensor, five scans/specimen (1 × 1 mm²) were performed to determine surface roughness (step size of 0.01 × 0.01).² All scanning was completed at a frequency of 300 Hz with full sensor speed (100%). The scans were performed and compared to a non-air-abraded group (control) serving as a reference for the roughness measurements.

Resin cement button preparation

Each conditioned Y-TZP specimen was placed on an Ultradent jig (Ultradent Products Inc., South Jordan, UT, USA) coupled with a plastic mold that has a cylindrical opening in the middle (2.38 mm in diameter and 3.5 mm in height) to build the resin cement button on Y-TZP (Figures 1B and 1C). Panavia F 2.0 (PF) resin cement (Kuraray Noritake Dental Inc., Okayama, Japan) was bonded to the zirconia samples according to the manufacturer's instructions (Table 1). Briefly, equal amounts of paste A and paste B were mixed on a pad for 20 seconds with a plastic spatula. In order to avoid air bubble entrapment, a syringe and an applicator were used to apply the resin cement into the plastic mold. The specimens were photo-polymerized for 20 seconds using an LED system (DEMI LED, Kerr, Orange, CA, USA). Light irradiance was monitored using a Managing Accurate Light Curing system (MARC, BlueLight Analytics Inc., Halifax, NS, Canada). Light irradiance was approximately 1,000 mW/cm². OXIGUARD II (Kuraray Noritake Dental Inc.) was then applied around the button and allowed to rest for 3 minutes before being rinsed with DI water. The dimensions of the resin cement buttons were 2.38 mm in diameter and 3.5 mm in height (Figure 1C).

Fatigue cyclic loading combined with thermocycling (CLTC)

The specimens prepared in each air-abrasion pressure group were subdivided into two groups yielding to six groups ($n = 12$). Each prepared specimen with resin cement button was either subjected to SBS testing after 24 hours (1b-24h, 2b-24h, and 2.8b-24h) or to combined fatigue cyclic loading and thermocycling (fatigue cyclic loading, CLTC) (1b-CLTC, 2b-CLTC and 2.8b-CLTC), and then tested for SBS. Each specimen was loaded on an Ultradent jig and the designated groups were subjected to fatigue cyclic loading and then tested for SBS. The fatigue cyclic loading and SBS testing was applied in a shear direction parallel to the adhesive

interface using an Ultradent semicircular testing fixture (Ultradent Products Inc.).²⁹ The semicircular fixture loading area was 2.4 mm in diameter and positioned flushed with the Y-TZP specimen surface contacting the cylindrical bonded resin cement at the zirconia and cement interface (Figure 2). The fatigue cyclic loading was subjected to a low load (10 N, approximately 2.25 MPa) to prevent loading damage at the zirconia-resin interface for 10,000 cycles and a frequency of 1.0 Hz using a mechanical cycling machine (ElectroPuls E3000, Instron, Norwood, MA, USA).²⁹ After completion of the fatigue cyclic loading, the same groups were thermocycled for 5,000 cycles between 6 - 48°C (30 seconds dwell time and 10 seconds transfer time). All groups were then stored in DI water and tested for SBS either after 24 hours or after CLTC.

Shear bond strength (SBS) test and failure mode analysis

Each specimen was mounted on the Ultradent jig as described earlier and subjected to debonding under shear force using a notched (semi-circular) edge at a crosshead speed of 1.0 mm/min using a universal testing machine (Electro Puls 3000, Instron, Boulder, MA, USA; Figure 2). The SBS was calculated using the following formula:

$$\text{SBS (MPa)} = \text{Load (N)} / \text{area (mm}^2\text{)}$$

Modes of failure were observed using an optical microscope (Measurescope UM-2, Nikon Corporation, Tokyo, Japan) at a magnification of $\times 40$ after SBS testing. The modes of failure were classified as follows: adhesive, failure between the Y-TZP ceramic surface and the resin cement; cohesive, failure within the resin cement; mixed, failures in which partly adhesive and partly cohesive ones were observed coincidentally in a fractured surface. Representative Y-TZP samples from the control and each air-abrasion treated specimen surface, before resin cement preparation, were prepared to qualitatively analyze the surface roughness under scanning

electron microscopy (SEM; JSM 6390 LV, JEOL Ltd., Tokyo, Japan). In addition, SEM images were obtained from fractured/debonded representative specimens. Specimens were sputter-coated with gold for 90 seconds (Desk II Cold Sputter, Denton Vacuum LLC, Moorestown, NJ, USA) prior to SEM imaging.

Statistical analyses

Comparisons between groups for SBS values were performed using two-way analysis of variance (ANOVA), followed by Tukey's post-hoc test. Specimens with pre-test failures were included in the analysis as 0 MPa; the lowest observed value was 1.8 MPa in group 1b-24h. Weibull characteristic strengths were compared using parametric Weibull-model survival analysis. The differences between the groups for type of failure were analyzed using Fisher's Exact tests. ANOVA was performed to compare the surface roughness between groups, with a fixed effect for the groups and a random effect to account for correlations among multiple roughness measurements within one specimen.

Results

Surface roughness measurement

The mean average surface roughness (Ra) is shown in Figure 3. The 2.8b group showed significantly higher Ra than that of the control group and 1b (Ra, $p = 0.006$, $p = 0.017$, respectively). No other statistically significant differences were found between other groups.

SBS test and failure mode analysis

Shear bond strength data indicated that the interaction effect between pressure and time/cycling was not significant ($p=0.22$, Table 2). Additionally, pressure did not have a significant effect on SBS. Mean shear bond strength ($p = 0.006$) and Weibull characteristic strength ($p = 0.012$) were significantly higher for the 24 hour storage groups compared to the

CLTC groups. Also, the 2b-CLTC group had significantly lower Weibull modulus than those of the other groups ($p < 0.05$).

The 2b-CLTC group presented significantly higher percentage of specimens failing during fatigue test than those of the 1b-24h ($p = 0.037$), 2b-24h ($p = 0.042$), 2.8b-24h ($p = 0.042$), and 2.8b-CLTC ($p = 0.042$) groups. None of the other groups showed significantly different failure modes from each other (Table 3). In general, the mixed failure mode was observed more than the adhesive one. Representative SEM images of specimens air-abraded at different pressures demonstrated a qualitative increase in surface roughness with increasing pressure (Figure 4). Figure 5 shows SEM images of a representative specimen with mixed failure.

DISCUSSION

The main goal of the present study was to investigate whether fatigue cyclic loading combined with thermocycling (CLTC) could serve as an aging method to evaluate resin bond durability to zirconia. According to the present findings, the null hypothesis was rejected as the combined aging method significantly decreased resin bond strength to zirconia regardless of the air-abrasion pressure used to condition the ceramic surface.

In this study, the fatigue cyclic loading was performed on the bond interface of adhesive cement and zirconia in a shear direction. This methodology was devised in an attempt to allow the application of cyclic low loads to the bonded interface, which may better mimic the fatigue environment occurring in the oral cavity.^{24,29} The results of this study show that interfacial adhesion of MDP-containing cement – zirconia is indeed susceptible to mechanical degradation, although the optimal stress for use in the method needs further investigation.

In the present investigation, the zirconia surface roughness data obtained using non-contact profilometry (quantitative) and SEM images (qualitative) after air-abrasion confirm an

increase in surface irregularities with increasing air-abrasion pressure. Although the surface roughness for group 2b was not significantly different from those of 1b and 2.8b, there was a significant increase in surface roughness between 1b and 2.8b. These observations are in agreement with the results of a recent study in which the SEM images showed differences between 1b and 2.8b.²⁵

It is well established that the luting agent plays a critical role in the long-term success of resin-ceramic bonding. The resin cement used (PF) contained a phosphate monomer (10-MDP). The phosphate ester monomer in 10-MDP is suggested to enhance bond strength due to the chemical P-O-Zr bond formed between zirconia and MDP.^{8,11,14-21,30} Furthermore, the bond strength between the MDP-containing resin cement and zirconia is suggested to be enhanced when the zirconia surface is air-abraded with alumina particles, therefore, a two-fold bonding is produced, namely chemical bonding and micromechanical interlocking.³⁰⁻³² In this study, PF showed significantly lower SBS after the combined CLTC processes. The significantly higher SBS observed for 24 hour storage compared to CLTC suggests that CLTC had a significant effect on the strength of bonded cement to zirconia. Worth mentioning, only 2b-CLTC group showed a large number of specimens failing during fatigue-cyclic loading and before shear bond testing. This behavior resulted in the significantly lower Weibull modulus for 2b-CLTC.

According to Nemli *et al.*, cyclic fatigue can cause phase transformation of tetragonal crystals of Y-TZP to monoclinic crystal structures.³³ Therefore, one would expect that cyclic fatigue would lead to some degree of phase transformation. However, the low stress value used (~2.25 MPa) may not have resulted in significant transformation. Meanwhile, the association between the surface roughness and resin cement is particularly critical when using

low air-abrasion pressure as the topography created may not be deep enough to properly impregnate the resin cement in the micro-irregularities on the zirconia surface.¹⁹⁻²¹ However, this was not supported in this study, where Group 1b may have generated sufficient roughness and surface morphology for satisfactory bonding to zirconia compared to Group 2b.

The proposed testing methodology may give a more relevant evaluation of bond strength between zirconia and adhesive cements as it better represents the worst case in the oral cavity. Further investigations are needed to validate this testing method by addressing the effect of increasing the load and fatigue cycle number, testing different adhesive cements and surface treatments. Additional research of the phase transformation and surface flaw geometry that occurs during specimen preparation is also worth including in future studies.

Conclusions

Based on the results of this study, 2.8b air-abrasion resulted in higher surface roughness and increased the SBS of resin cement to zirconia. The combined CLTC significantly decreased the SBS of resin cement to zirconia regardless of the air-abrasion pressure used to condition the zirconia surfaces.

Conflict of Interest: No potential conflict of interest relevant to this article was reported.

Author Contributions

Conceptualization: Bottino MC. Data curation: Sabrah AH, Al-Zain A, Bottino MC. Formal analysis: Eckert GJ. Funding acquisition: Bottino MC. Investigation: Al-Shehri E, Al-Zain A, Sabrah AH, Al-Angari S, Al Dehailan L. Methodology: Bottino MC. Project administration: Bottino MC, Al-Zain A. Resources: Bottino MC. Software: not applicable. Supervision: Bottino MC. Validation: Bottino MC, Platt JA. Visualization: Al-Zain A, Sabrah AH. Writing - original

draft: Al-Shehri E, Al-Zain A, Sabrah AH, Al-Angari S, Al Dehailan L. Writing - review & editing:
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References

1. Lazar DR, Bottino MC, Özcan M, Valandro LF, Amaral R, Ussui V, Bressiani AH. Y-TZP ceramic processing from coprecipitated powders: a comparative study with three commercial dental ceramics. *Dent Mater* 2008;24:1676-85.
2. Luangruangrong P, Cook NB, Sabrah AH, Hara AT, Bottino MC. Influence of full-contour zirconia surface roughness on wear of glass-ceramics. *J Prosthodont* 2014;23:198-205.
3. Denry I, Kelly JR. Emerging ceramic-based materials for dentistry. *J Dent Res* 2014;93:1235-42.
4. Lawson NC, Burgess JO. Dental ceramics: a current review. *Compend Contin Educ Dent* 2014;35:161-6.
5. Wang H, Aboushelib MN, Feilzer AJ. Strength influencing variables on CAD/CAM zirconia frameworks. *Dent Mater J* 2008;24:633-638.
6. Lohbauer U, Zipperle M, Rischka K, Petschelt A, Müller FA. Hydroxylation of dental zirconia surfaces: characterization and bonding potential. *J Biomed Mater Res B Appl Biomater* 2008;87:461-467.
7. Wegner SM, Kern M. Long-term resin bond strength to zirconia ceramic. *J Adhes Dent.* 2000;2:139-147.
8. Blatz MB, Sadan A, Kern M. Resin-ceramic bonding: a review of the literature. *J Prosthet Dent* 2003;89:268-274.
9. Hummel M, Kern M. Durability of the resin bond strength to the alumina ceramic Procera. *Dent Mater J* 2004;20:498-508.
10. Friederich R, Kern M. Resin bond strength to densely sintered alumina ceramic. *Int J Prosthodont* 2002;15:333-338.

11. Kern M. Bonding to oxide ceramics—laboratory versus clinical outcome. *Dent Mater* 2015;31:8-14.
12. Thompson JY, Stoner BR, Piascik JR, Smith R. Adhesion/cementation to zirconia and other non-silicate ceramics: where are we now? *Dent Mater* 2011;27:71-82.
13. Seo DG. Zirconia surface treatment for successful bonding. *Restor Dent Endod* 2014;39:333.
14. Bottino MA, Bergoli C, Lima EG, Marocho SM, Souza RO, Valandro LF. Bonding of Y-TZP to dentin: effects of Y-TZP surface conditioning, resin cement type, and aging. *Oper Dent* 2014;39:291-300.
15. Inokoshi M, De Munck J, Minakuchi S, Van Meerbeek B. Meta-analysis of bonding effectiveness to zirconia ceramics. *J Dent Res* 2014; 93:329-934.
16. De Souza G, Hennig D, Aggarwal A, Tam LE. The use of MDP-based materials for bonding to zirconia. *J Prosthet Dent* 2014;112:895-902.
17. Blatz MB, Sadan A, Martin J, Lang B. In vitro evaluation of shear bond strengths of resin to densely-sintered high-purity zirconium-oxide ceramic after long-term storage and thermal cycling. *J Prosthet Dent* 2004;91:356-362.
18. Özcan M, Bernasconi M. Adhesion to zirconia used for dental restorations: a systematic review and meta-analysis. *J Adhes Dent* 2015;17:7-26.
19. Özcan M, Melo RM, Souza RO, Machado JP, Valandro LF, Bottino MA. Effect of air-particle abrasion protocols on the biaxial flexural strength, surface characteristics and phase transformation of zirconia after cyclic loading. *J Mech Behav Biomed Mater* 2013;20:19-28.
20. Kern M, Barloi A, Yang B. Surface conditioning influences zirconia ceramic bonding. *J Dent Res.* 2009; 88(9):817-822.

21. Aurélio IL, Marchionatti AM, Montagner AF, May LG, Soares FZ. Does air particle abrasion affect the flexural strength and phase transformation of Y-TZP? A systematic review and meta-analysis. *Dent Mater* 2016;32:827-845.
22. Barkmeier W, Erickson R, Latta M. Fatigue limits of enamel bonds with moist and dry techniques. *Dent Mater J* 2009;25:1527-1531.
23. Wegner SM, Gerdes W, Kern M. Effect of different artificial aging conditions on ceramic-composite bond strength. *Int J Prosthodont* 2002;15:267-272.
24. Williamson RT, Mitchell RJ, Breeding LC. The effect of fatigue on the shear bond strength of resin bonded to porcelain. *J Prosthodont* 1993;2:115-119.
25. Re D, Augusti D, Augusti G, Giovannetti A. Early bond strength to low-pressure sandblasted zirconia: evaluation of a self-adhesive cement. *Eur J Esthet Dent* 2012;7:164-175.
26. Zhang Y, Lawn BR, Malament KA, Van Thompson P, Rekow ED. Damage accumulation and fatigue life of particle-abraded ceramics. *Int J Prosthodont*. 2006;19:442-448.
27. Zhang Y, Lawn BR, Rekow ED, Thompson VP. Effect of sandblasting on the long-term performance of dental ceramics. *J Biomed Mater Res B Appl Biomater* 2004;71:381-386.
28. Özcan M, Kerkdijk S, Valandro LF. Comparison of resin cement adhesion to Y-TZP ceramic following manufacturers' instructions of the cements only. *Clin Oral Investig* 2008;12:279-282.
29. Atsushi N, Yoshida T, Bottino MC, Platt JA. Influence of zirconia surface treatment on veneering porcelain shear bond strength after cyclic loading. *J Prosthet Dent* 2014;112:1392-1398.

30. Kern M, Wegner SM. Bonding to zirconia ceramic: adhesion methods and their durability. *Dent Mater* 1998;14:64-71.
31. Chang JC, Powers JM, Hart D. Bond strength of composite to alloy treated with bonding systems. *J Prosthodont* 1993;2:110-114.
32. Chen L, Suh BI, Brown D, Chen X. Bonding of primed zirconia ceramics: evidence of chemical bonding and improved bond strengths. *Am J Dent* 2012;25:103-108.
33. Nemli SK, Yilmaz H, Aydin C, Bal BT, Tıraş T. Effect of fatigue on fracture toughness and phase transformation of Y-TZP ceramics by X-ray diffraction and Raman spectroscopy. *J Biomed Mater Res B Appl Biomater* 2011;100B:416-424.

Captions to Figures

Figure 1. Zirconia specimen with resin cement adhered. a) Zirconia specimen embedded in acrylic resin; b) Placement of the specimen on the Ultradent jig coupled with the semicircular plastic mold (*); c) Zirconia specimen after resin cement button fabrication.

Figure 2. Fatigue cyclic loading and shear bond strength test apparatus. Fatigue cyclic loading was applied in a shear direction parallel to the adhesive interface using an Ultradent loading jig with a semicircular loading surface (2.4 mm in diameter) in close proximity to the zirconia-resin button interface and subjected to 10 N load for 10,000 cycles with a frequency of 1.0 Hz. a) Frontal-view of the testing apparatus; b) A close up for the testing setup.

Figure 3. Mean surface roughness and standard deviations of different groups after air-abrasion. Control group represents the zirconia surface before air abrasion treatment. Different letters represent significant differences among the air-abrasion pressures tested.

Figure 4. Scanning electron microscopic images ($\times 2,000$) of zirconia surface for control and after different air-abrasion pressures. a) control group (no air-abrasion); b) 1 bar; c) 2 bar; d) 2.8 bar.

Figure 5. Scanning electron microscopic images of zirconia surface denoting mixed mode of failure after debonding at magnification a) $\times 30$; b) $\times 300$.

List of Tables

Table 1. Material, composition, and application procedure for Panavia F 2.0

Material	Manufacturer	Lot No.	Composition	Application
Panavia F 2.0	Kuraray	061288		
<u>Components:</u> Paste A		00571 A	10-MDP, hydrophobic aromatic and aliphatic photoinitiator, dibenzoyl peroxide dimethacrylate, hydrophilic dimethacrylate, silanized silica	Dispense equal amounts of pates A and B for 20 sec. Apply paste. In this study, paste was applied using a syringe and applicator.
Paste B		00284 A	hydrophobic aromatic and aliphatic dimethacrylate, sodium aromatic sulphinate, N,N-diethanol-p-toluidine, functionalized sodium fluoride, and silanized barium glass	Light cure for 20 sec. (LED light)
OXYGUARD II		00676 A		Apply around the margins. Wait for 3 min. Rinse with distilled water.

Abbreviations. MDP: 10-methacryloyloxydecyl dihydrogen phosphate.

Table 2. Mean and standard deviation (SD) of the shear bond strength (SBS, in MPa)

Group	SBS Mean \pm SD	Weibull Characteristic Strength (95% CI)	Weibull Modulus (95% CI)
1b-24h	9.2 \pm 3.4 ^a	10.2 (8.3-12.2) ^a	3.1 (1.7-4.6) ^a
2b-24h	10.5 \pm 3.0 ^a	11.6 (9.9-13.4) ^a	4.0 (2.3-5.7) ^a
2.8b-24h	10.7 \pm 5.9 ^a	12.1 (8.5-15.7) ^a	2.0 (1.2-2.9) ^a
1b-CLTC	8.7 \pm 4.2 ^b	9.4 (6.4-12.5) ^b	1.8 (0.9-2.7) ^a
2b-CLTC	5.8 \pm 5.3 ^b	4.3 (0.0-8.6) ^b	0.6 (0.3-0.9) ^b
2.8b-CLTC	7.6 \pm 1.9 ^b	8.3 (7.2-9.4) ^b	4.5 (2.5-6.4) ^a

Mean SBS was significantly higher ($p = 0.006$) along with Weibull characteristic strength ($p = 0.012$) for 24 hour storage compared to CLTC. 2b-CLTC had significantly lower Weibull modulus than the other groups ($p < 0.05$). Superscript lowercase letters represent significant differences within the same column.

Abbreviations: CI, confidence interval; 1b, 1 bar; 2b, 2 bar; 2.8b, 2.8 bar; 24h, 24 hours; CLTC, fatigue cyclic loading and thermocycling;

Table 3. Failure mode of the samples

Group	Adhesive failure		Mixed failure		Failed During Cyclic Loading	
	n	%	n	%	n	%
1b-24h ^b	1	8	11	92	0	0
2b-24h ^b	3	25	9	75	0	0
2.8b-24h ^b	4	33	8	67	0	0
1b-CLTC ^{a,b}	0	0	11	92	1	8
2b-CLTC ^a	1	8	6	50	5	42
2.8b-CLTC ^b	4	33	8	67	0	0

Adhesive failure, failure at the interface between Y-TZP zirconia and resin cement; Cohesive failure, failure within the resin cement; Mixed failure, failure including both adhesive and cohesive failure.

Superscript lowercase letters represent significant differences among the groups.

Abbreviations: 1b, 1 bar; 2b, 2 bar; 2.8b, 2.8 bar; CLTC, cyclic loading and thermocycling.





