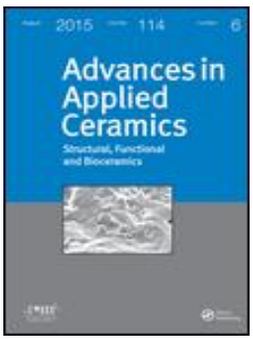

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Advances in Applied Ceramics

Structural, Functional and Bioceramics

ISSN: 1743-6753 (Print) 1743-6761 (Online) Journal homepage: http://www.tandfonline.com/loi/yaac20

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To cite this article: Jie Zhang, Wenjie Hu, Tijana Stijacic, Kwok-Hung Chung, Tingkai Li & Zhijian Shen (2018): Bonding of novel self-glazed zirconia dental ceramics, Advances in Applied Ceramics, DOI: 10.1080/17436753.2018.1463935

To link to this article: https://doi.org/10.1080/17436753.2018.1463935

Published online: 26 Apr 2018.

ADVANCES IN APPLIED CERAMICS, 2018
https://doi.org/10.1080/17436753.2018.1463935

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Bonding of novel self-glazed zirconia dental ceramics

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ABSTRACT

Bonding behaviours of a novel self-glazed zirconia dental material were investigated. The effect of a preformed porous nanoceramic bonding surface and the different cleansing methods on saliva-contaminated bonding surfaces was assessed in this *in vitro* study. Cleaning procedures commonly used in dental offices were tested. All specimens demonstrated adhesive fracture patterns except for airborne particle abrasion group, which resulted in mixed-type fracture pattern and the highest bonding force values. No statistically significant differences in bonding force values were found between self-glazed zirconia with and without a preformed porous nanoceramic bonding surface when bonded with the self-adhesive resin cement (RelyX™ Unicem 2). Scanning electron micrographs revealed no interaction between the bonding surface and the resin cement after priming. Mechanical retention is the predominant bonding mechanism between the bonding surface and the luting resin cement.

ARTICLE HISTORY
Received 7 December 2017
Accepted 6 April 2018

KEYWORDS
Zirconia; self-glazed zirconia; bonding; saliva contamination; dental ceramics

Introduction

Current advances in computer-aided design/computer-aided manufacturing (CAD/CAM) technology have facilitated and expanded the use of yttria-stabilised tetragonal zirconia polycrystalline (Y-TZP)-based ceramic materials in dentistry. Y-TZP ceramic restorations have been widely accepted due to their transformational toughening properties, which provide a combination of high flexural strength and fracture toughness [1–4]. Moreover, with the use of monolithic Y-TZP ceramic restorations becoming increasingly common in dentistry, desirable aesthetic properties have become critical to further integration of this material in daily practice. In order to obtain a higher optical transmittance and to increase translucency, Y-TZP dental ceramic tends to be manufactured with improved density homogeneity and with smaller, preferably nanograins [5]. Recently, a new member in Y-TZP ceramics family, named as self-glazed zirconia, with superior surface smoothness and human enamel-like optical appearance of its as-sintered surface, was developed by applying a precision additive three-dimensional gel deposition approach [6,7] and has been increasingly used for dental prostheses construction, Figure 1. The inherent formation of an enamel-like surface ensures no further need for glazing, thereby avoiding breakable interfaces and wear-out of glazed surface [8]. With vigorous laboratory tests, the reliability of this new zirconia ceramic material was proven as strong as traditional zirconia ceramic materials in crown and bridge constructions [9,10]. Aside from producing self-glazed zirconia with hybrid zirconia gel that yields a ceramic material with fundamentally improved density homogeneity and reduced grain size thus also improved optical transparency, the internal bonding surface is formed by milling green body to achieve an increased degree of marginal fit. This bonding surface can be further tailored to perform a porous nanoceramic bonding surface in green stage in order to enhance the micromechanical bonding effect with adhesive cements during clinical application [11–13].

Generally, zirconia-based restorations are cemented using conventional luting cements; bonding with resin-based cements to the internal bonding surface would be advantageous for many clinical applications [14,15]. However, dislodgement of zirconia-based restorations is still an issue in dental practices [15]. Wegner and Kern [16] demonstrated that a durable bond to zirconia-based restorations could be achieved by applying resin-based cements containing 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP) to an airborne particle abraded zirconia surface. Adhesion between resin-based cement and zirconia surface might be compromised in clinical situations

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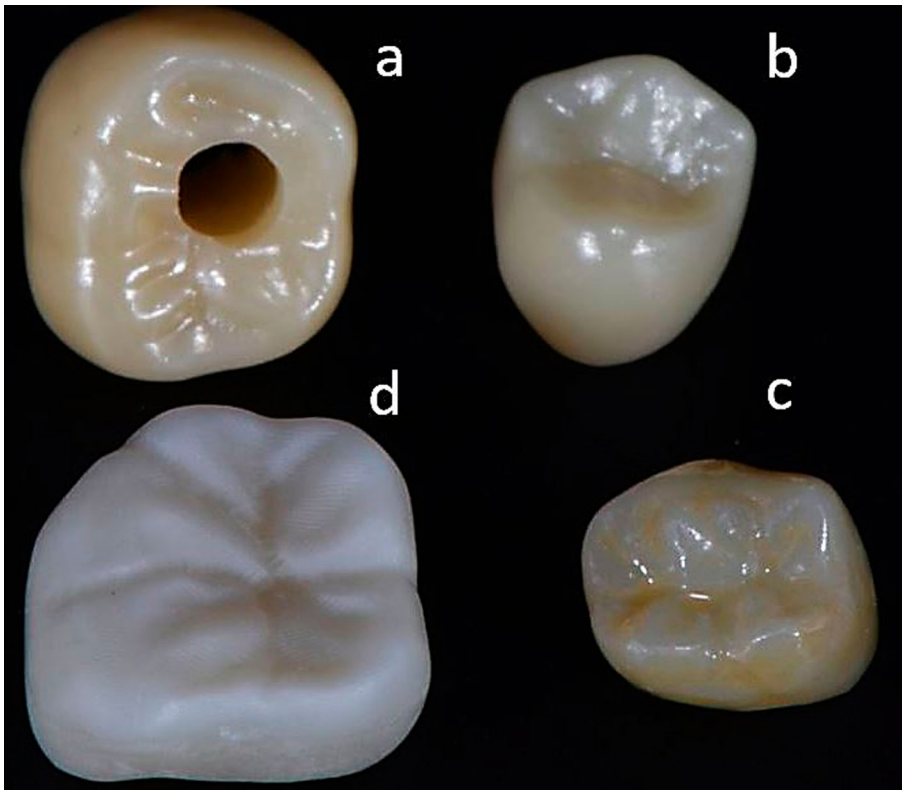

Figure 1. Representative zirconia crowns. (a) and (b) are self-glazed zirconia crowns; (c) is a conventional zirconia crown after glazing; and (d) is a conventional zirconia crown before glazing.

because the zirconia internal bonding surface might often be contaminated by saliva during the try-in procedure [17,18]. Some previous studies demonstrated that saliva contamination significantly affected the strength and durability of resin-based cement bonded onto zirconia surface and that airborne particle abrasion was the most useful cleaning method [17–19]. In order to facilitate cementation of all ceramic crowns and bridges, a commercial cleaning detergent (Ivoclean, Ivoclar Vivadent, Schaan, Liechtenstein) has been introduced to the dental profession with the purpose of decontaminating the zirconia extra-orally. The manufacturer claims that a simple application of the solution, followed by water rinsing and air-drying, effectively cleans the saliva-contaminated bonding surfaces of various dental restorations including zirconia ceramics. However, the cleaning efficacy of such cleaning procedures on saliva-contaminated zirconia still has no consensus in terms of reinforcing the resin cement–zirconia bonding.

In this *in vitro* study, we evaluated the cleaning efficacy of five commonly used cleaning procedures in an attempt to enhance resin cement to zirconia bonding of self-glazed zirconia surfaces following simulation of try-in with saliva exposure. In addition, the effect of a preformed porous nanoceramic bonding surface on the bonding strength of resin-based cements was assessed. The null hypothesis was that no statistically significant difference exists in shear bond strength between decontaminated self-glazed zirconia surfaces and adhesive resin cements, and the self-glazed zirconia with a preformed porous nanoceramic bonding surface and adhesive resin cements.

Material and methods

Preparation of the self-glazed zirconia specimens

Zirconia disc specimens (6.5 mm diameter and 4.0 mm thickness) were fabricated using hybrid zirconia gel and CAD/CAM technology. The self-glazed zirconia specimens were manufactured by a precision additive three-dimensional gel deposition approach. A hierarchically rough porous nanoceramic surface opposite to the smooth self-glazed surface was performed by green-milling and surface hybrid gelation afterwards. All samples were pressure-less sintered at 1450°C for 90 min in air to achieve a relative density approximately 99.9%. Thereafter, the samples were furnace cooled down to room temperature [6,7]. The disc specimens were randomly assigned into five surface decontamination groups ($n = 6$).

Preparation of the self-glazed zirconia specimens with a preformed porous nanoceramic bonding surface

The as-milled self-glazed zirconia specimens fabricated by the aforementioned protocol were subjected to

surface hybrid gelation over the intaglio surface in order to create micro-retention elements [11–13].

An identical number of lithium disilicate glass-ceramic disc specimens (IPS e.max Press, Ivoclar Vivadent AG) were fabricated by injecting autopolymerising acrylic resin into a silicone mould with a 6.5 mm diameter to a 4.0 mm depth and used as controls to compare with the self-glazed zirconia specimens with and without a preformed porous nanoceramic bonding surface. The patterns were sprued and invested in a pressing ring according to the manufacturer's instructions. Following the lost-wax technique, glass-ceramic ingots were heat-pressed into the mould cavity. After bench cooled, the discs were devested with glass beads at 0.2 MPa pressure, and the reactive layer was removed with a ceramic cleaning solution. The sprue was removed with a diamond disc, and the glass-ceramic discs were finished and polished according to the manufacturer's instructions. The lithium disilicate glass-ceramic discs received chemical etching with 4.5% hydrofluoric acid gel (IPS Ceramic Etching Gel, Ivoclar Vivadent AG) for 20 s and were ultrasonically cleaned and air-dried before saliva immersion treatment.

Saliva collection and immersion procedure

Unstimulated saliva was collected using the spitting method from a healthy, non-smoking, male volunteer, in accordance with the Institutional Review Board at the University of Washington. Half an hour before collection, the participant was instructed to rinse mouth thoroughly with deionised water and then drink 200 mL of warm (37°C) drinking water. The participant was seated comfortably with eyes open, head held tilted slightly downward for unstimulated saliva collection. Using the spitting method, saliva was allowed to accumulate in the floor of the mouth and the subject spat out into a graduated test tube at 60-s intervals. The collection period of 5 min was deemed adequate [20].

Self-glazed zirconia specimens with and without a preformed porous nanoceramic bonding surface were immersed in saliva for 1 min at 37°C. After saliva immersion, the bonding surfaces of specimens were treated according to tested cleaning procedures, which are generally available in dental offices, Figure 2. Non-saliva-contaminated discs were used as controls.

Surface decontamination of saliva-treated specimens

Group H2O: Saliva-contaminated specimens were rinsed with water from the three-way syringe for 15 s, and then air-dried.

Group OCL: Saliva-contaminated specimens were swabbed with 6% NaOCl (sodium hypochlorite solution, Vista Dental Products) for 15 s using cotton tip, rinsed with water from the three-way syringe for 15 s, and then air-dried.

Group PHA: Saliva-contaminated specimens were cleaned with 37% phosphoric acid etching gel (Scotchbond Universal Etchant, 3M Oral Care) for 15 s, rinsed with water from the three-way syringe for 15 s, and then air-dried.

Group ABR: Saliva-contaminated specimens were abraded with 50 μm Al_2O_3 at 0.25 MPa pressure at a distance of 10 mm with rotating movement for 15 s, then rinsed with water from the three-way syringe for 15 s and air-dried.

Group IVC: Saliva-contaminated specimens were cleaned with a commercially available cleaning paste (Ivoclean, Ivoclar Vivadent) for 20 s, rinsed with water from the three-way syringe for 15 s, and then air-dried.

Group CTL: Self-glazed zirconia disc specimens without a preformed porous nanoceramic bonding surface

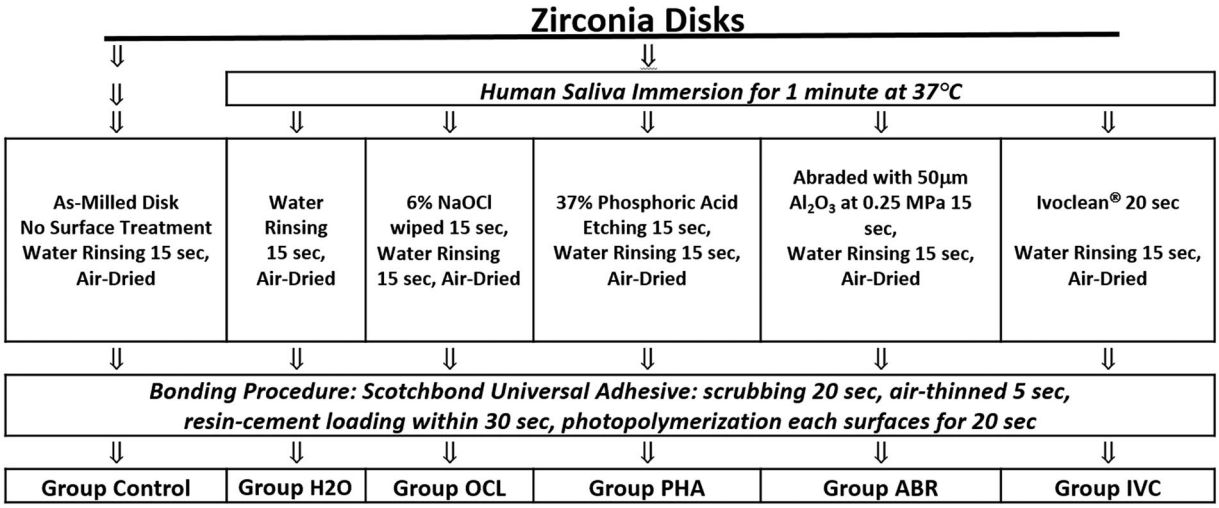

Figure 2. Study design for the self-glazed zirconia groups.

were rinsed with water from the three-way syringe for 15 s, and then air-dried. There was no saliva immersion.

Group CZR: No saliva immersion. Self-glaze zirconia disc specimens with a preformed porous nanoceramic bonding surface were rinsed with water from the three-way syringe for 15 s, and then air-dried.

Group SZR: Saliva-contaminated self-glazed zirconia disc specimens with a preformed porous nanoceramic bonding surface were rinsed with water from the three-way syringe for 15 s, and then air-dried.

Group emx: Saliva-contaminated lithium disilicate glass-ceramic disc specimens were rinsed with water from the three-way syringe for 15 s, and then air-dried.

Bonding procedure

Two commercially available resin-based cements, a self-adhesive resin cement (RelyX™ Unicem 2, 3M Oral Care) and an adhesive resin cement (RelyX™ Ultimate, 3M Oral Care), were used. All bonding procedures were carried out immediately after decontamination procedures. After the specimens were subjected to assigned cleaning regimen, an adhesive system (Scotchbond Universal Adhesive, 3M Oral Care) was applied with a microbrush and scrubbed for 20 s, followed by air-thinning for 5 s before loading the adhesive resin cement material. For the self-adhesive resin cement groups, no aforementioned adhesive priming procedure was performed because the adhesive had been preloaded in the resin cement according to the manufacturer's instruction. Resin cement buttons (4.0 mm in height and 6.5 mm in diameter) were fabricated from the tested resin cement materials using a cylindrical nylon mould (Flange Bearings, 6.5 × 8.0 mm; Midwest Fastener), with the treated and cleaned zirconia specimen inserted at one end. The resin cements were loaded directly into the moulds and covered with a 0.2 mm thick cover glass (Fisher Scientific). To standardise the bonding procedure, the zirconia disc specimens bonded with resin cements were positioned inside the mould with a load of 50 N applied during photopolymerisation at room temperature. The top and bonding margins were photopolymerised for 20-s per quarter surface with a photopolymerising unit (Paradigm DeepCure light, 3M ESPE).

Shear bond test and failure analysis

After preparation, all test specimens were kept in a water bath for 24 h at 37°C before testing. Shear bond test was conducted using a universal testing machine (Model 5500R, Intron Corp) with a customised fixture at a crosshead speed of 1.5 mm min^{-1} [21]. The fractured interfacial zones on the zirconia specimens were examined using optical stereomicroscopy (SZH-10, Nikon Corp) at 10X magnification to determine the mode of failure. The modes of failure were identified as follows: adhesive – failure at the bonding interface; cohesive – failure within the resin cement button; and mixed – failure at interface and within the resin cement [22]. Representative debonded zirconia specimens were observed using scanning electron microscopy (JEOL JSM-7000F, JEOL Ltd) to confirm the pattern of failure. Shear bond strength values were statistically analysed using two-way ANOVA, and Tukey HSD test was used to control the familywise error rate ($\alpha = 0.05$).

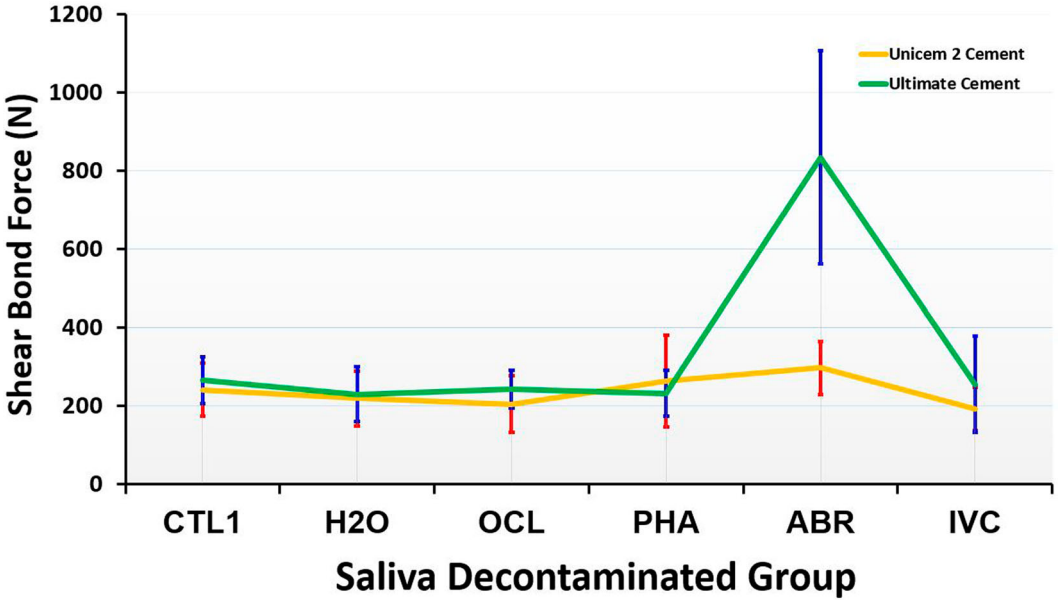

Figure 3. Shear bond force values (*N*) of all self-glazed zirconia groups according to saliva decontamination methods and cementation protocols. CTL1, control group; H_2O, water-rinsed group; OCL, 6% NaOCl-wiped group; PHA, phosphoric acid-etched group; ABR, airborne particle-abraded group; and IVC, Ivoclean-treated group.

Table 1. Results of shear bond force values of the preformed porous nanoceramic bonding surfaces.

Groups	RelyX Unicem 2 Means (SD)	RelyX Ultimate Means (SD)
CZR	341.7 (162.1)[a]	642.6 (177.9)[A]
SZR	292.7 (94.6)[a]	628.9 (220.1)[A]
emx	638.0 (69.0)[A, B]	749.5 (227.6)[A, B]

Notes: Means and standard deviations (SD) in Newton ($n = 6$). Within the same column, means with the same letters are not statistically different ($P > .05$). Within the same row, means with the same letters are not statistically different ($P > .05$). CZR, self-glazed zirconia disc specimens with a preformed porous nanoceramic bonding surface rinsed with water from the three-way syringe for 15 s, and then air-dried; SZR, saliva-contaminated self-glazed zirconia disc specimens with a preformed porous nanoceramic bonding surface rinsed with water from the three-way syringe for 15 s, and then air-dried; emx, saliva-contaminated lithium disilicate glass-ceramic disc specimens rinsed with water from the three-way syringe for 15 s, and then air-dried.

Results

The mean ± SD bond force values of self-glazed zirconia specimens without a preformed porous nanoceramic bonding surface ranged from 192.6 ± 55.3 N (Group IVC, RelyX™ Unicem 2 cement) to 835.0 ± 271.4 N (Group ABR, RelyX™ Ultimate cement) and the data are shown in Figure 3. The shear bond force values of the self-glazed zirconia disc specimens with a preformed porous nanoceramic bonding surface ranged from 292.7 ± 94.6 N (Group SZR, RelyX™ Unicem 2 cement) to 642.6 ± 177.9 N (Group CZR, RelyX™ Ultimate cement), Table 1. Significantly lower bond force values were found in the self-glazed zirconia disc specimens with a preformed porous nanoceramic bonding surface cemented with self-adhesive resin cement (RelyX™ Unicem 2) ($P < .05$) as compared with those cemented with adhesive resin cement (RelyX™ Ultimate). Statistical analyses revealed that the saliva contamination was not a significant factor ($P > .05$), while combination of airborne particle abrasion and adhesive application had a significant influence on the shear bond force ($P < .01$) of self-glazed zirconia.

Representative self-glazed zirconia specimens are presented in Figure 4. The representative surface morphologies of specimens after different decontamination protocols are shown in Figure 5. For the self-glazed zirconia tested groups, optical examination of the fractured surfaces revealed that all failures were adhesive in nature, occurring at the resin cement–zirconia interface, except for the airborne particle abrasion group (Group ABR) which exhibited failure of the mixed type. Scanning electron micrographs are presented in Figure 6.

Representative original and fractured specimens of the self-glazed zirconia disc specimens with a preformed porous nanoceramic bonding surface are shown in Figure 7. All failures were of mixed-type patterns.

Discussion

This study was designed to assess the bonding forces of adhesive resin cements to a novel zirconia material with a specific manufacturing protocol involving precision additive three-dimensional gel deposition approach. Within the limitations of this *in vitro* study, the null hypothesis was rejected. The two resin cement systems used were significantly different from each other when bonded to airborne particle abrasion surfaces. Airborne particle abrasion surface treatment gave better resin cement bond strength than other decontamination methods when adhesive resin cement was used, Figure 3. Saliva-contaminated zirconia surfaces cleaned with other decontamination procedures produced lower micromechanical retention than airborne particle abrasion treatment. The finding is in agreement with the results of previous studies [23–25]. However, an improvement was noted when a preformed porous nanoceramic bonding surface was introduced to the self-glazed zirconia, giving comparable results to airborne particle abrasion treatment.

The novel self-glazed zirconia ceramic material tested in this study manufactured with nano-scaled hybrid zirconia gel provided a smoother and glazed-like surface. Regarding the crystal structure of this novel zirconia ceramic material, Shen and his colleagues investigated the XRD patterns on the as-sintered and fracture surfaces after flexural strength test reported that the t-ZrO_2 was proved to be the dominating phase on both surfaces [9]. The comparison in the XRD patterns suggested that, during fracture, the

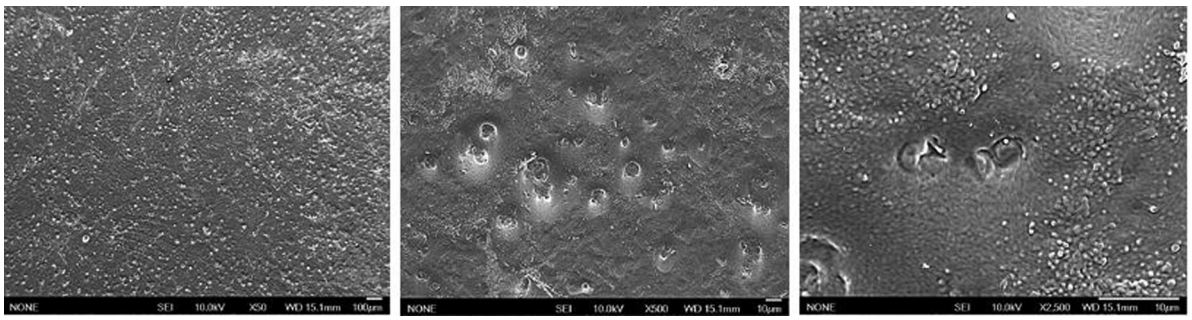

Figure 4. Representative self-glazed zirconia surfaces before saliva contamination and cleansing treatment.

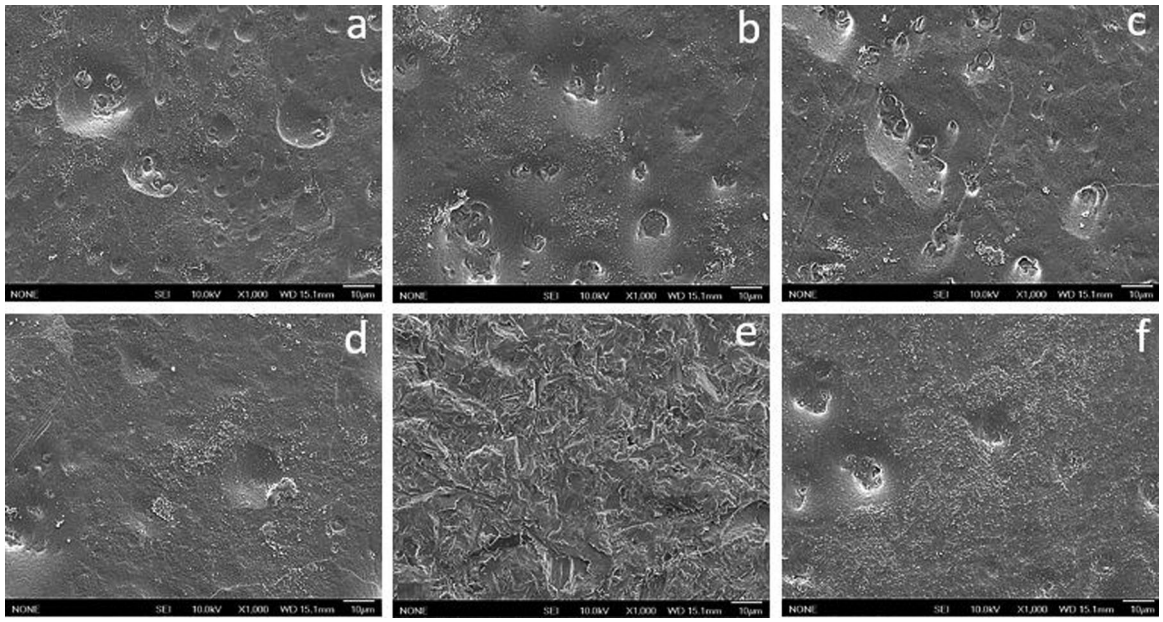

Figure 5. Representative self-glazed zirconia surfaces after treatments according to saliva decontamination methods. (a) Group control; (b) Group H2O; (c) Group OCL; (d) Group PHA; (e) Group ABR; (f) Group IVC. H_2O, water-rinsed group; OCL, 6% NaOCl-wiped group; PHA, phosphoric acid-etched group; ABR, airborne particle-abraded group; and IVC, Ivoclean-treated group. Magnification of ×1000.

contribution of phase transformation of t- to m-ZrO_2 to toughening was surprisingly negligible. In the current study, the decontamination methods tested after saliva contamination treatment were mostly chemical methods, with the exception of the airborne particle abrasion. However, the differences in shear bond strength between decontaminated surfaces and resin cements were not statistically significant for various chemical methods tested and values were comparable to the self-glazed zirconia disc specimens without a preformed porous nanoceramic bonding surface as control surface. Neither negative effect of 37% phosphoric acid etching nor positive effect of clean paste (Ivoclean) on shear bond forces was determined in the present study. This is in contrast with the results of previous studies, which reported improvement when Ivoclean paste was used [23–25], and a decrease in bond strength values after phosphoric acid etching [26].

Good wetting of resin cement is crucial for obtaining adhesion of restorations to tooth surface. In

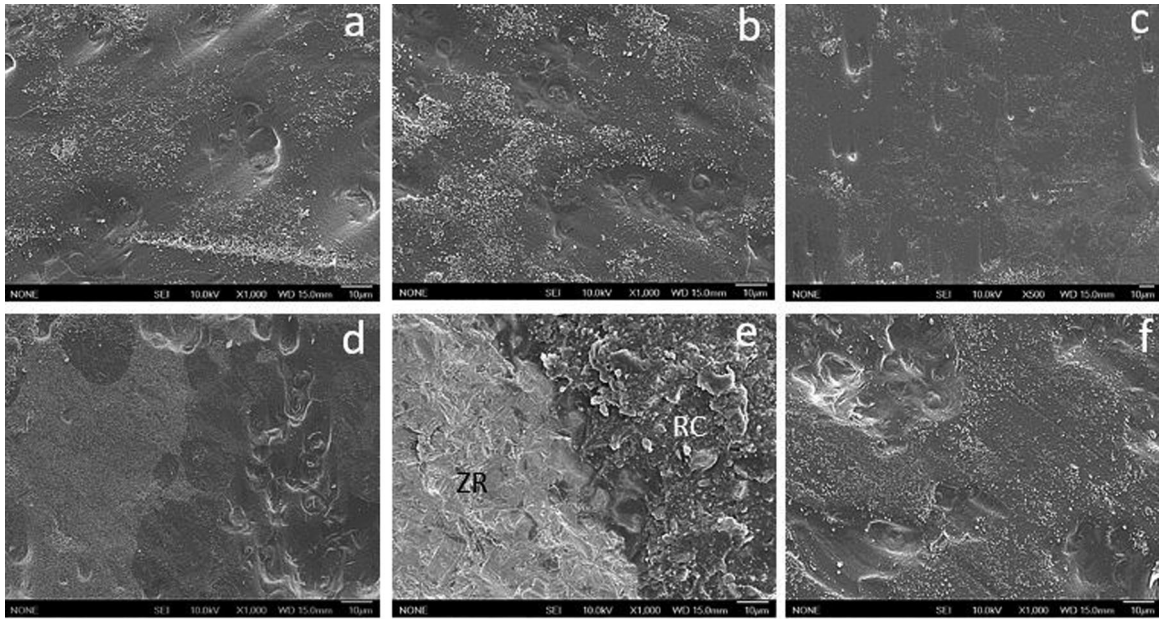

Figure 6. Representative self-glazed zirconia fracture surfaces after decontamination treatment and shear bond test. (a) Group control; (b) Group H2O; (c) Group OCL; (d) Group PHA; (e) Group ABR, RC: residual adhesive resin cement and ZR: airborne particle-abraded zirconia surface; (f) Group IVC. H_2O, water-rinsed group; OCL, 6% NaOCl-wiped group; PHA, phosphoric acid-etched group; ABR, airborne particle-abraded group; and IVC, Ivoclean-treated group. Magnification of ×1000.

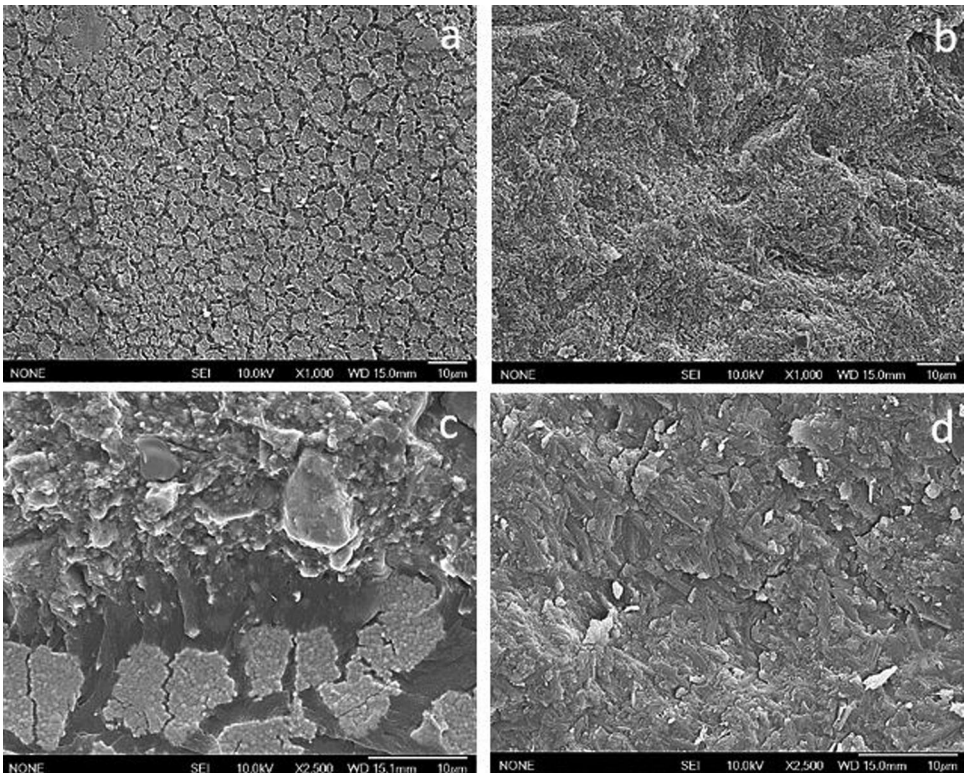

Figure 7. Representative saliva-contaminated self-glazed zirconia with a preformed porous nanoceramic bonding surface (a) and etched glass-ceramic surface (b) after water rinse and air-dried, magnification of ×1000. Mixed type fracture of the self-glazed zirconia with a preformed porous nanoceramic bonding surface (c) and of lithium disilicate glass-ceramic surface (d) after adhesive resin cement bonding and shear bond test, magnification of ×2500.

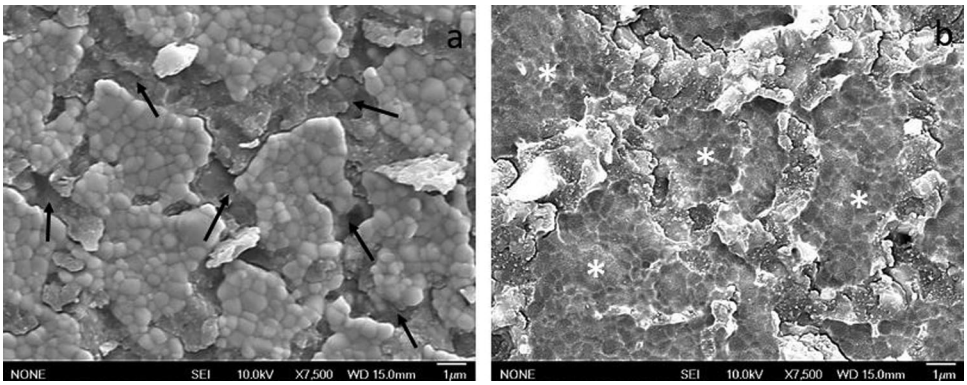

Figure 8. Representative self-glazed zirconia with a preformed porous nanoceramic bonding surface (a) zirconia surface with incomplete wetting of self-adhesive resin cement within the interstices (arrows); (b) fracture surface of self-adhesive resin cement with duplicating of zirconia cluster surfaces (asterisks), magnification of ×7500.

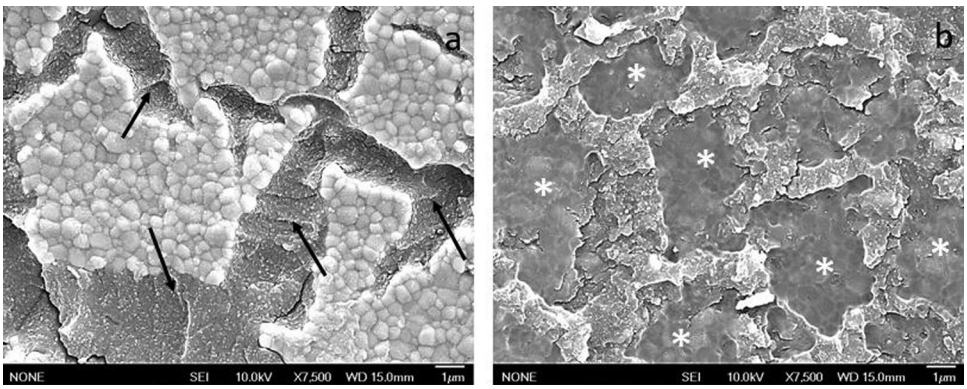

Figure 9. Representative self-glazed zirconia with a preformed porous nanoceramic bonding surface (a) zirconia surface with complete wetting of adhesive and resin cement within the interstices (arrows) resulted in brittle fracture patterns; (b) fracture surface of adhesive resin cement with duplicating of zirconia cluster surfaces (asterisks), magnification of ×7500.

general, both micromechanical retention and chemical adhesion are required to achieve a good and durable bonding. However, the current dental zirconia (Y-TZP) ceramic materials are nearly fully dense and comprised almost no glassy phase [2]. Analysis of the fractured zones showed a predominance of adhesive failure for chemical cleaning groups, which may be related to low interaction between chemical bonding groups and zirconia. The fracture interfaces revealed incomplete wetting of self-adhesive resin cement within the interstices of the self-glazed zirconia disc specimens with a preformed porous nanoceramic bonding surface, Figure 8. For the adhesive resin cement, adhesive was applied and reacted before loading of adhesive resin cement. Owing to the priming of adhesive agent before loading of adhesive resin cement, the wetting of adhesive resin cement showed completely occupying the interstices with brittle fracture patterns, Figure 9. The results of duplicating the preformed porous nanoceramic bonding surfaces indicated that there is a limited chemical interaction between zirconia surface and adhesive resin cement. If there are no micromechanical elements being generated by methods such as airborne particle abrasion or surface topography modification by, for example, hybrid gelation, adhesive resin cement will act only as a luting agent for the final cementation of zirconia restorations.

Conclusion

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

1. Significantly higher shear bond force values were achieved when the airborne particle abrasion was used to clean saliva-contaminated self-glazed zirconia surface.
2. Neither negative effect of 37% phosphoric acid etching, nor positive effect of cleaning paste (Ivoclean) on shear bond forces was determined by the present study.
3. The self-glazed zirconia with a preformed porous nanoceramic bonding surface obtained comparable shear bond force to etchable lithium disilicate glass-ceramics when using an adhesive resin cement.
4. Limited interaction was revealed between zirconia surface and resin cement tested as observed via optical stereomicroscopy.

Acknowledgements

Errantech is acknowledged for the preparation of all the zirconia samples used for this study.

Disclosure statement

No potential conflict of interest was reported by the authors.

Funding

This work was supported by Department of Restorative Dentistry, University of Washington, Spencer Funds, (grant 65-4909 task 821).

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